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Cu (II) And Ag (I) Complexes of Schiff Base Derived from 2-Hydroxy-5-nitrobenzaldehyde with o- Aminobenzenethiol Synthesis, Characterization And Anti-Corrosion Behaviour Study

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Abstract

A Schiff base ligand was synthesized through the reflux reaction between 2-hydroxy-5-nitrobenzaldehyde and o-aminobenzenethiol in a 1:1 molar ratio, resulting in the formation of a novel compound. New copper (II) and silver (I) complexes from this ligand were synthesized also by the reflux reaction between the synthesized ligand and Cu(II) and Ag(I) salts in 1:1 molar ratio (metal: ligand). The coloured synthesised complexes were characterized by ¹HNMR, ¹³CNMR, mass spectra, FTIR, UV-Visible and molar conductivity techniques. In this work the synthesized ligand acts as a tridentate ligand coordinate with the metal ions through its imine, hydroxyl and the thione groups. The structure of both complexes is estimated from the mass spectra which suggest the octahedral and tetrahedral chemical structures of copper (II) and silver (I) complexes respectively. The anticorrosive properties of the synthesized compounds were investigated using an electrochemical corrosion cell, with experiments conducted at two different temperatures (298 K and 308 K) and two varying concentrations (0.01 M and 0.05 M) for each compound. Both complexes show a higher anti-corrosion activity (92%) in comparison with the ligand (90%).

Keywords: o- Aminobenzenethiol, 2-Hydroxy-5-nitrobenzaldehyde, Cu (II) and Ag (I) Complexes, Anti-Corrosion Study

معقدات النحاس الثنائي والفضة الاحادي مع قاعدة شيف المشتقة من 2- هيدروكسي-5- نيتروبنزالديهايد و اورثو- امينوبنزوثايول , تحضير وتشخيص ودراسة سلوكها كمضادات للتآكل

سعاد طه سعد

قسم الكيمياء، كلية العلوم للبنات، جامعة بابل، بابل، العراق

الخلاصة

تم تحضير قاعدة شيف من خلال تفاعل التصعيد بين 2- هيدروكسي-5- نيتروبنزالديهايد و اورثو- امينوبنزوثايول بنسبة 1:1 حيث تم الحصول على هذا المركب .ثم تم تحضير معقد النحاس الثنائي والفضة الاحادي الجديدين من هذا الليكاند ايضا بتفاعل التصعيد بين الليكاند المحضر واملاح النحاس الثنائي والفضة الاحادي ايضا بنسبة 1:1 من الليكاند: الفلز. تم تشخيص المركبات الملونة المحضرة باستخدام تقنيات طيف الرنين النووي المغناطيسي و مطياف الكتلة و مطيافية الاشعة تحت الحمراء و مطيافية الاشعة المرئية-فوق البنفسجية والتوصيلية المولارية. في هذا البحث يسلك الليكاند المحضر سلوك ثلاثي السن حيث يتناسق مع

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الايونات الفلزية من خلال مجاميع الازوميثين والهيدروكسل والثايون. وكان الشكل المقترح بالاعتماد على نتائج مطياف الكتلة هو الثماني السطوح والرباعي السطوح لكل من معقدي النحاس الثنائي والفضة الاحادي على التوالي. تم دراسة الخواص المضادات للتآكل للمركبات المحضرة باستخدام خلية التآكل الكهروكيميائية حيث اجريت هذه التجربة عند الدرجات الحرارية (298 و 308) كلفن وباستخدام تركيزين مختلفين (0.01 و 0.05) مولاري لكل مركب. اظهرت المعقدات المحضرة فعالية مضادة للتآكل اعلى (92%) مقارنة مع الليكاند (90%).

1. INTRODUCTION

In coordination chemistry there are several kinds of molecules which act as ligands. These molecules range from simple to complex in structure. The more intricate ones can be synthesized using suitable reaction techniques [1]. An example of such molecules is Schiff bases. Schiff bases can be considered as important and interesting class of synthesized molecules due to their ease to synthesize, high stability and applications in different fields such as, in chemistry, medicine and in industry [2-5]. These compounds are widely used due to the presence of an azomethine ($-N=CH-$) or imine functional group in their chemical structure, which gives them certain characteristics [6]. Precisely the lone pair of electrons in nitrogen sp^2 hybridized orbital gives this group its importance [7, 8]. As mentioned, in chemistry, Schiff bases can form complexes with metal ions as they are considered as attractive ligands thanks to their ability to form a stable chelate ring with a wide range of metal ions [9, 10]. This ring is formed by the coordination of the metal with the nitrogen atom of the azomethine group and often with the support of electron rich neighbour atom [11-12]. In medicine also they have played an important role for cancer, inflammatory, fever and virus treatments [13-16]. While in industry they are capable to act as dyes, shows activity as a suppressible of corrosion, catalysts in photo-catalysed reactions and in polymerization reactions [4,7, 17, 18].

In this paper a ligand derived from o- aminobenzenethiol with one of salicylaldehyde derivatives was synthesized by the condensation reaction between the two mentioned components. Two new complexes of the ligand with Cu(II) and Ag(I) were synthesized via reflux reactions between the ligand and the respective metal salts. The complexes were characterized using various methods and evaluated as corrosion inhibitors.

2. EXPERIMENTAL SECTION

2.1 Materials

2-hydroxy-5-nitrobenzaldehyde and o- aminobenzenethiol were supplied from Shanghai Macklin biochemical. Metal salts were purchased from Merck and Fluka. Methanol was purchased from Biosolvechimie SARL, DCM, acetonitrile and DMSO were supplied from CHEM-SUPPLY-PTY.LTD, BDH and CDH respectively.

2.2 Instrumentation

The infrared spectra were measured using Fourier transform infrared spectrophotometer (FT-IR – 8400S) ($4000-400\text{ cm}^{-1}$) from Shimadzu using KBr disk. The electronic transitions were measured by UV-Visible spectrophotometer (Peak Instruments C-7200) from Shimadzu. Bruker spectrometer instrument operating at (400Hz) was used to measure ^1H -NMR and ^{13}C -NMR spectra. Mass spectra was measured using mass device from Agilent Technologies (5975C). Conductivity was measured by WTW SERIES, cond 722. Melting points were measured using melting point / SMP30 Stuart apparatus.

2.3 Synthesis of ligand (S)

This ligand was synthesized according to the literature with a very slight modification [19] 2-hydroxy-5-nitrobenzaldehyde (1g, 5.89mmol) was dissolved in ethanol (25ml) and mixed with *o*-aminobenzenethiol (0.75g, 5.89 mmol) and two drops of HCl were added. The reaction was left to reflux for seven hours. A yellow precipitate of (S) was crashed after cooling. The precipitate was filtrated and washed with ethanol.

2.4 Synthesis of copper (II) and Ag(I) complexes with (S) ligand

Copper (II) and silver(I) complexes were synthesized by refluxing (0.4g, 1.46mmol) of the previously mentioned ligand with $\text{CuCl}_2 \cdot 2\text{H}_2\text{O}$ and AgNO_3 in 1:1 (ligand: metal) molar ratio. The ligand was firstly dissolved in DCM/ MeOH mixture (25/5 ml) and mixed with metal salts. Green and brown precipitates of copper (II) and silver(I) complexes respectively were formed after four and eight hours where they then filtered and washed with DCM.

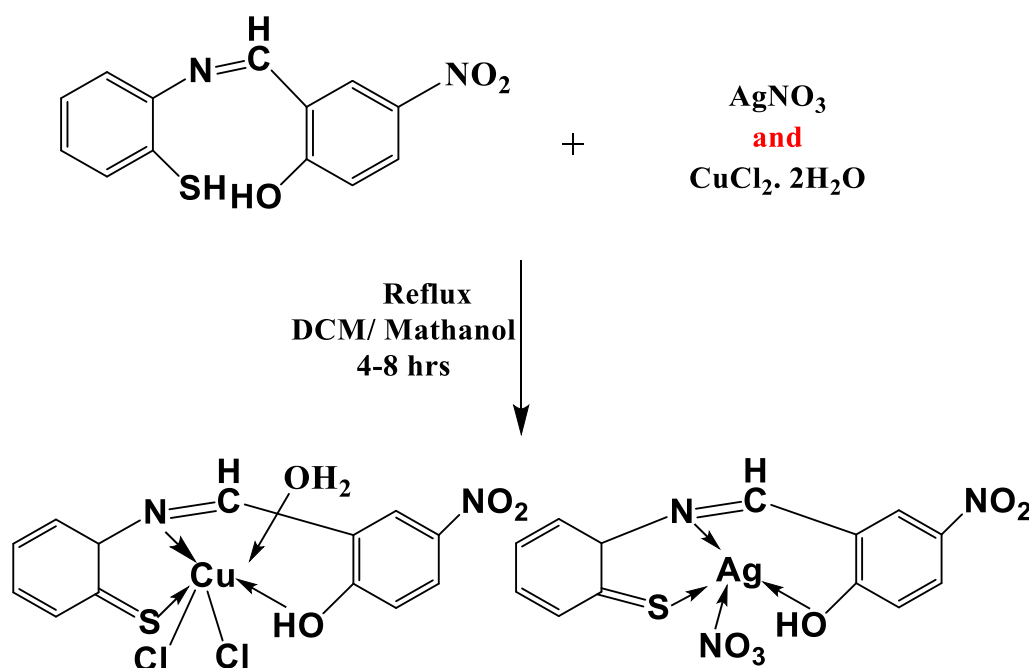
2.5 Anti- corrosion study of (S) and metal complexes

This experiment followed procedures previously described in the literature [20-22]. All tests were conducted at the University of Baghdad- College of Science at 298K and 308K using a circulation of cold and hot water bath and two concentrations of the tested compounds were used (0.01 M and 0.05M). The tested samples were inserted in the corrosion cell in which the diameter of the exposed surface to the solution was (16.55cm^2).

3. RESULTS AND DISCUSSION

3.1 Ligand and metal complexes synthesis

The synthesis of the Schiff base was carried out using the common procedure to synthesize such compounds. This procedure relies on the reflux reaction between the aldehyde (2-hydroxy-5-nitrobenzaldehyde) with the amine (*o*-aminobenzenethiol) in 1:1 molar ratio and hydrochloric acid was added as a catalyst. The yellow ligand (S) were produced. This ligand was then reacted with $\text{CuCl}_2 \cdot 2\text{H}_2\text{O}$ and AgNO_3 also in 1:1 L: M molar ratio for four hours and eight hours respectively using DCM/ MeOH as a reaction solvent as given in Scheme 1. Table 1 shows some merits of the synthesized compounds.



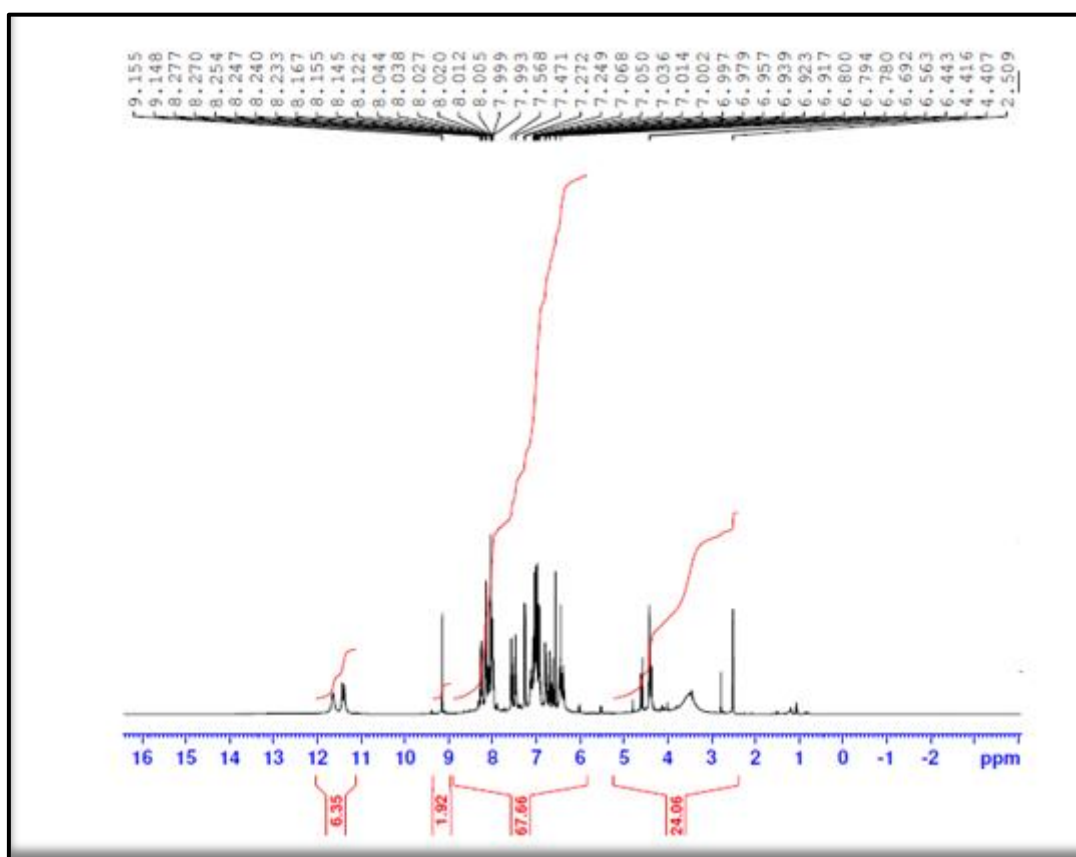
Scheme 1: The synthesized ligand (S) and its complexes

Table 1: Properties of the ligands and its metal complexes

Compound	Color	m.p °C	Yield %
Ligand (S)	Yellow	187-189	55
[Cu(S)Cl ₂ H ₂ O]	Green	165-167	72
[Ag(S)NO ₃]	Brown	125-127	70

3.2 Ligand ¹H-NMR spectrum

The spectrum of the ligand (Figure 1) displays three prominent signals corresponding to the three main functional groups in the ligand: the -OH and -SH groups, and the azomethine and aromatic proton signals. In detail, the signal in the range 3-4 ppm can be referred to the thiol (-SH group) [23]. While the aromatic protons appear in the range 7-8 ppm [24,25]. The signal at 9 ppm refers to azomethine group proton [4]. Finally, single in the range 11 and 12 ppm refers to the hydroxyl group [26].

**Figure 1:** The ¹H-NMR spectrum of Ligand

3.3 Ligand ¹³C-NMR spectrum

In Figure 2, the carbon atoms of greatest significance in the ligand's chemical structure are those in the azomethine group and those in the aromatic rings, as they contribute significantly to its structure and properties. The azomethine group (CH=N) carbon atom appears proximately at 160 ppm [27]. Other aromatic carbon atoms appear in the range 120-160 ppm [28].

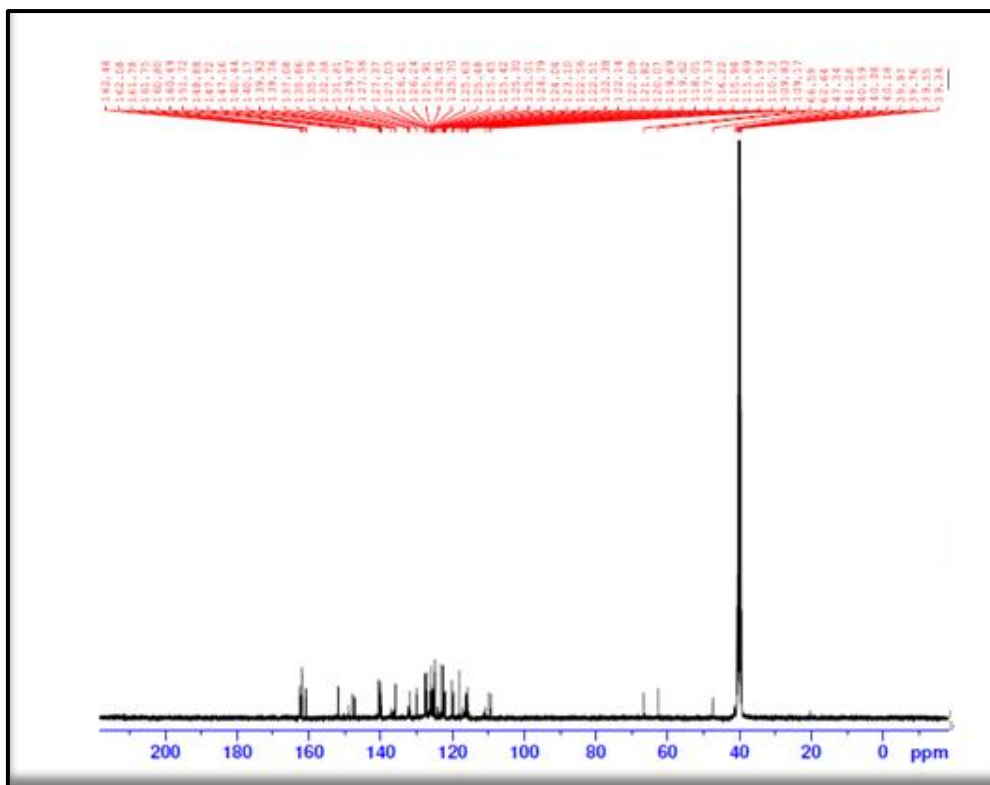


Figure 2: The ^{13}C -NMR spectrum of Ligand

3.4 Complexes mass spectra

The mass spectrum of Cu(II) complex (Figure 3) shows peak at 426 (m/z) which referred to the formula weight of $(\text{M}-2\text{H}^+)$, while Ag(I) complex shows peak at 444 (m/z) which indicates the fragment $(\text{M}-\text{OH}+2\text{H}^+)$ as shown in Figure 4.

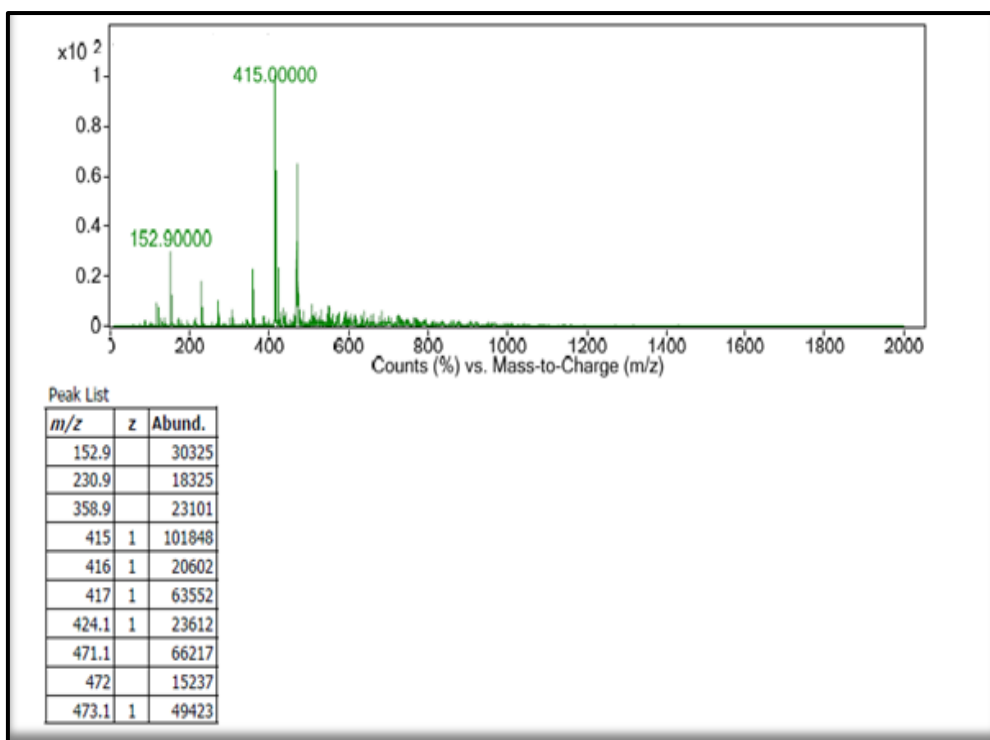


Figure 3: The mass spectrum of Cu(II) complex

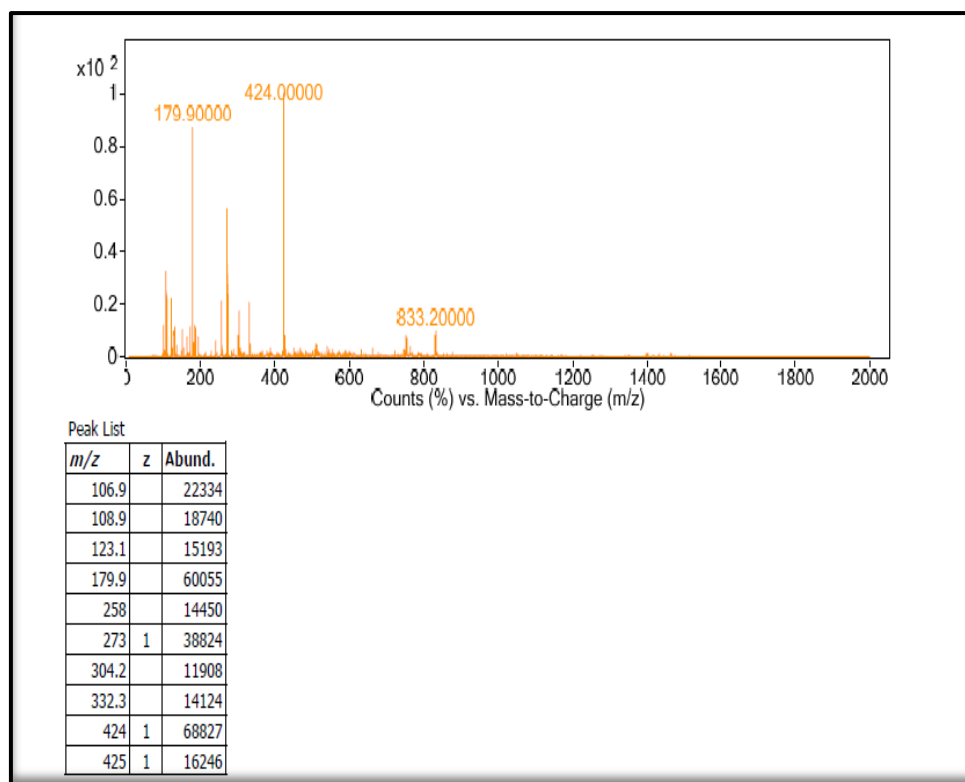


Figure 4: The mass spectrum of Ag(I) complex

3.5 FT-IR spectroscopy

In the FT-IR spectrum of the Schiff base ligand (Figure 5), the newly formed imine group can be identified by its characteristic peak at 1627 cm^{-1} [29]. The appearance of this band is combined by the disappearance of both carbonyl group of the aldehyde at 1664 cm^{-1} and the amine group of o- aminobenzenethiol at 3356 cm^{-1} and 3446 cm^{-1} . The $\nu(\text{S-H})$ vibration frequency would appear at 2528 cm^{-1} where it vanished in the ligand FT-IR spectra which means its conversion to the thione group ($\text{C}=\text{S}$) which appears at 1278 cm^{-1} [30]. In addition, the $\nu(\text{O-H})$ frequency of the hydroxyl group appears at 3066 cm^{-1} as a single band.

Upon coordination of the metal ions with the ligand, the positions of the mentioned bands shifted, indicating the formation of complexes, as evidenced by the spectral changes depicted in Figure 5. For example, the azomethine group is shifted to 1604 cm^{-1} and 1622 cm^{-1} in Cu(II) and Ag(I) complexes respectively as well as the thione group to 1274 cm^{-1} in both complexes [31]. In addition, the protonated hydroxyl group is also shifted to 3163 cm^{-1} and 3074 cm^{-1} in Cu(II) and Ag(I) complexes respectively and that suggests the bond between the two ions and this group.

Other bands in the range $400\text{--}600\text{ cm}^{-1}$ refer to the M-O, M-N and M-S bonds [32-33]. In Cu(II) complex a broad band in the region $3200\text{--}2600\text{ cm}^{-1}$ indicates the presence of water molecules [34]. Table 2 shows the significant bands in the FT-IR spectra of the ligand and its complexes.

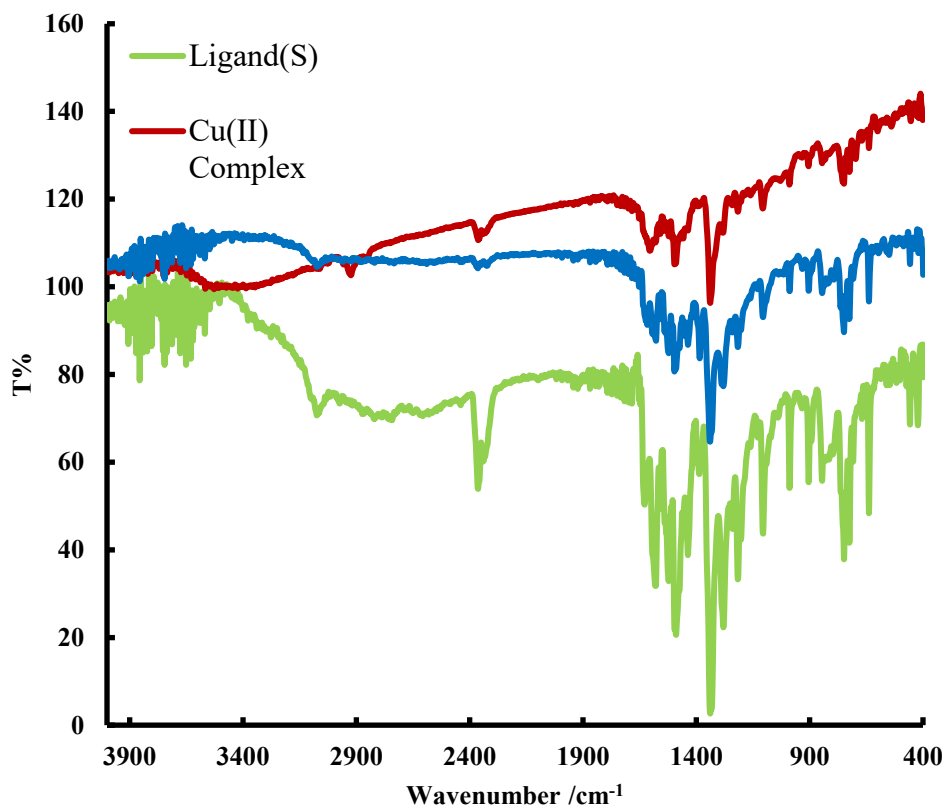


Figure 5: The FT-IR spectra of both Ligand: Cu(II) and Ag(I) complexes

Table 2: FT-IR data of Schiff base ligand and its complexes

Compound	$\nu(\text{H}_2\text{O})$	$\nu(\text{OH})$	$\nu(\text{C}=\text{N})$	$\nu(\text{C}=\text{S})$	$\nu(\text{M}-\text{N})$	$\nu(\text{M}-\text{O})$	$\nu(\text{M}-\text{S})$
Ligand (S)	-----	3066	1627	1278	-----	-----	-----
[Cu(S)Cl ₂ H ₂ O]	3600-3200	3163	1604	1274	580	493	414
[Ag(S)NO ₃]	-----	3074	1622	1274	601	480	410

3.6 Electronic spectra

The electronic transitions as shown in Figure 6 of ligand and its complexes were studied in acetonitrile as a solvent. The ligand spectrum shows three peaks at 299, 328 and 412 nm which refer to $\pi \rightarrow \pi^*$, $n \rightarrow \pi^*$ respectively [35,36]. These peaks were blue shifted and red shifted in Cu(II) and in Ag(I) complexes respectively as given in Table 3. This shift indicates the complexes formation. In addition, the most important shift is that related to the ligand peak at 412 nm which can be seen at 455 and 468 nm in Cu(II) and in Ag(I) complexes respectively. This red shift is related to the MLCT in both complexes [37].

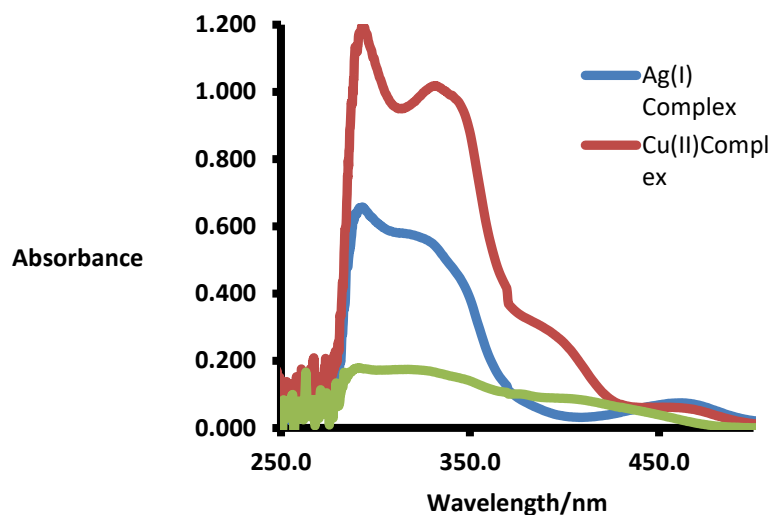


Figure 6: UV. Visible spectra of S, Cu (II) and Ag(I) complexes

Table 3: Suggested electronic transitions for the ligand and metal complexes

Compound	λ max(nm)	Transitions Types
Ligand (S)	299, 328, 412	$\pi \rightarrow \pi^*$, $n \rightarrow \pi^*$
[Cu(S)Cl ₂ H ₂ O]	295, 338, 401, 455	MLCT
[Ag(S)NO ₃]	285, 333, 468	MLCT

3.7 Molar conductivity measurements

The molar conductivity for both complexes was detected in DMSO. Based on the results as shown in Table 4 the non-electrolytic nature of both complexes was concluded. As this stimulated from the molar conductivity of non-electrolytic compounds in DMSO which appear in the range (15-27 $\text{ohm}^{-1} \cdot \text{cm}^2 \cdot \text{mol}^{-1}$) [38, 39]. In other words, the results suggest that the two chloride ions in the copper (II) complex and the nitrate ion in the silver (I) complex are likely to be incorporated within the coordination sphere of their respective complexes.

Table 4: Molar conductivity data in DMSO for the two complexes

Compound	Molar Conductivity $\text{Ohm}^{-1} \text{ cm}^2 \text{ mol}^{-1}$
[Cu(S)Cl ₂ H ₂ O]	12.1
[Ag(S)NO ₃]	13.1

3.8 Anti-corrosion study

3.8.1 Polarization curves

The corrosion was studied using a carbon steel with components as mentioned before in literatures [40]. and the corrosion experiments were carried out using the synthesized compounds with two concentrations and also two temperatures were used. The calculated parameters are given in Table 5 and are concluded from the polarization curves (Figures 7-10). These parameters include the corrosion current densities ($i_{\text{corr}})_0$ and (i_{corr}) (μA), which increased when the temperature increased and also when the concentration decreased. The corrosion potential (E_{corr}) (V) was obtained by the extrapolation of the cathodic and anodic

Tafel constants (V/decade) with and without the presence of the prepared compounds as inhibitors respectively in hydrochloric acid (0.1M) solution. The two Tafel slopes (β_a) and (β_c) (mV/Dec) were also calculated. Furthermore, the protection efficiency IE% was calculated from the following equation [41]:

$$\%IE = \frac{(i_{corr})_0 - (i_{corr})}{(i_{corr})_0} * 100 \quad (1)$$

According to the obtained results all the synthesized compounds give a good anti-corrosion behaviour or protection efficiency as shown in Table 7. However, this inhibition effect is disparate. For instance, the ligand exhibits enhanced anti-corrosion performance at the higher concentration (0.05 M) and lower temperature (298 K) compared to its lower concentration and higher temperature. While both complexes show a higher anti-corrosion activity in comparison with the ligand. Ag(I) complex exhibits a higher anti-corrosion activity at the highest concentration and at the two studied temperatures. While Cu(II) complex exhibits a similar anti-corrosion activity at the higher concentration and at the lowest temperature (298 K)

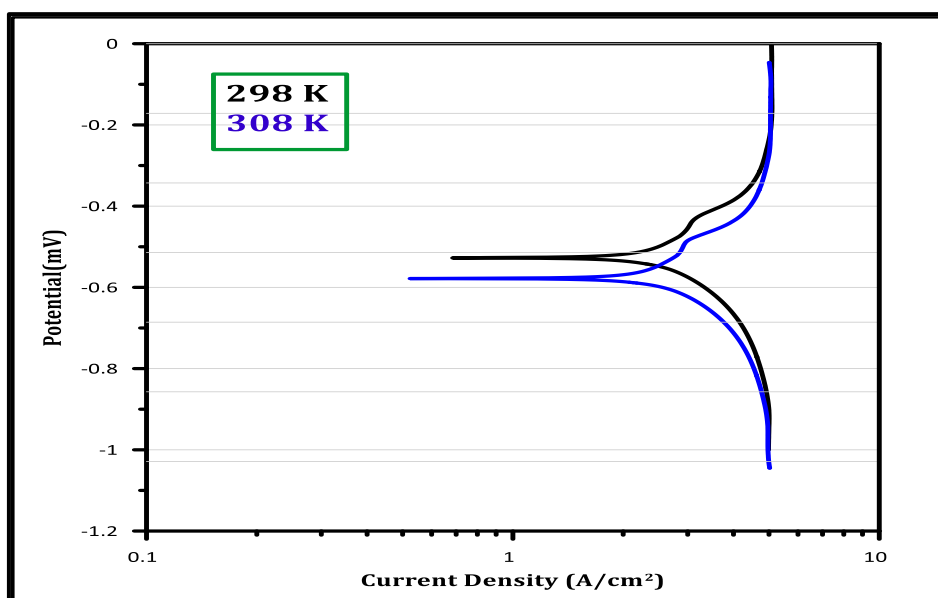
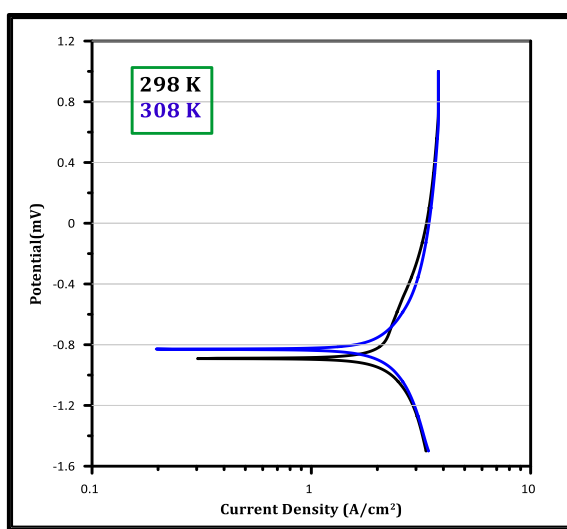
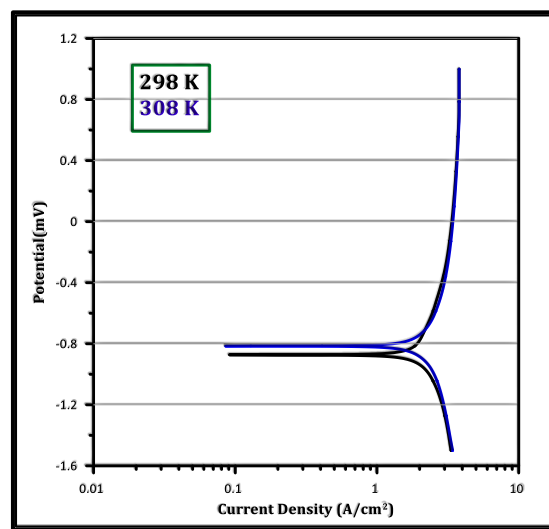


Figure 7: Polarization curve for corrosion of blank (HCl solution) at 298K and 308K



(A)



(B)

Figure 8: Polarization curves for corrosion of blank solution and (A): (0.01M) and (B): (0.05 M) of ligand at 298K and 308K

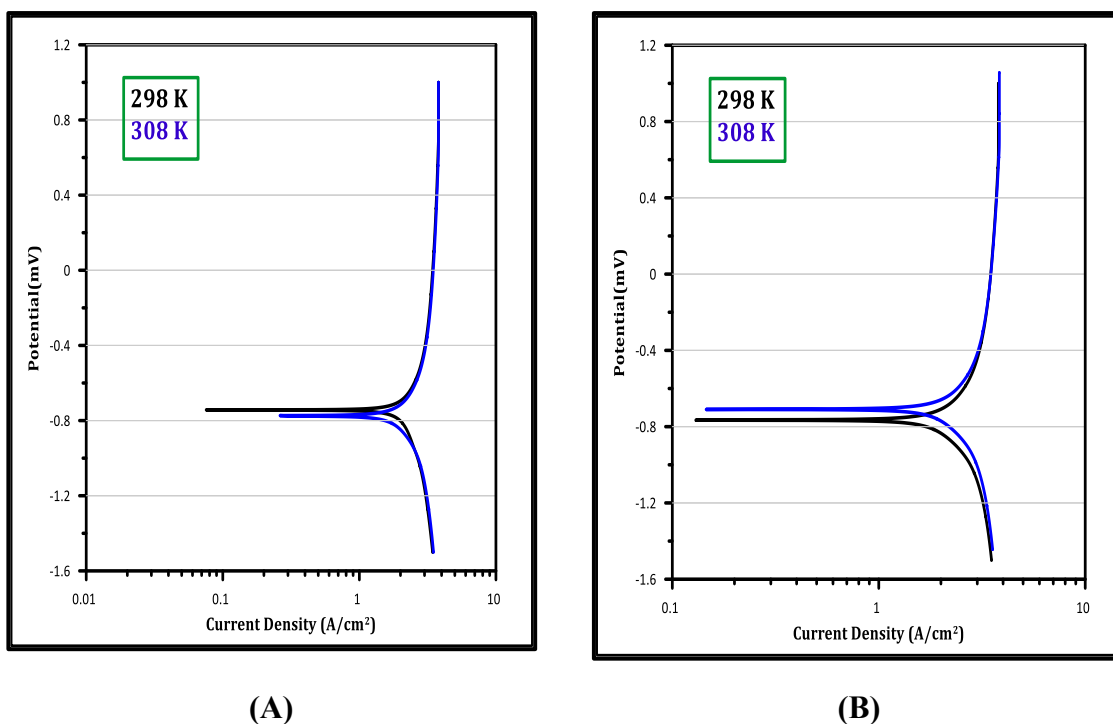


Figure 9: Polarization curves for corrosion of blank solution and (A): (0.01M) Cu(II) complex and (B): (0.05 M) at 298K and 308K

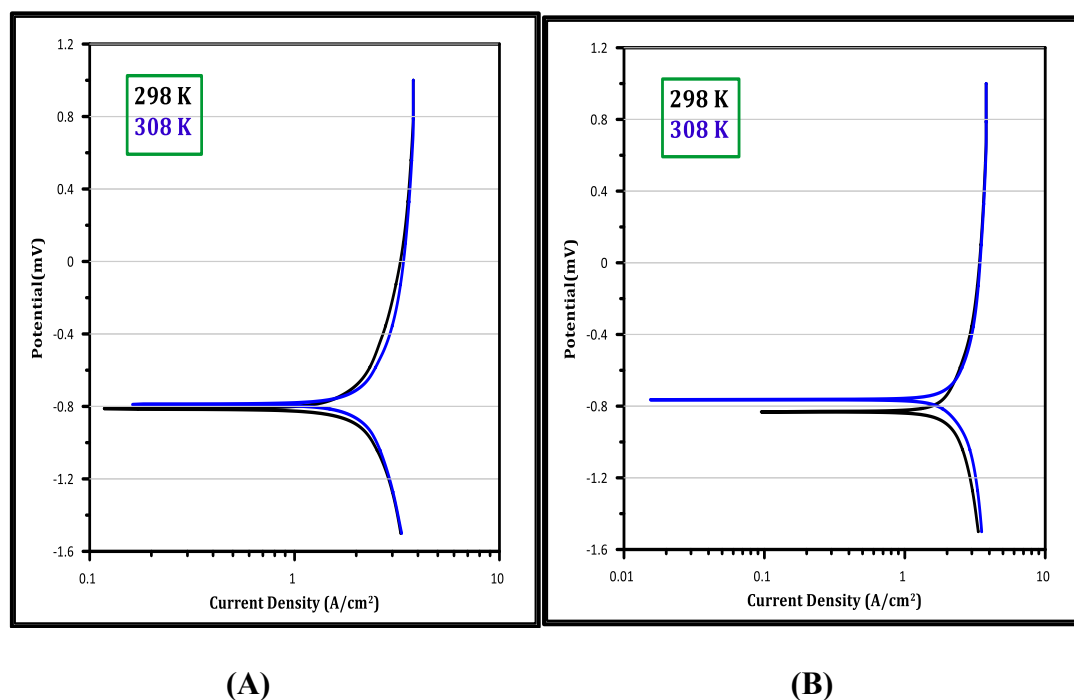


Figure 10: Polarization curves for corrosion of blank solution and (A): (0.01M) Ag(I) complex and (B): (0.05 M) at 298K and 308K

Table 5: Corrosion parameters for the blank (HCl solution) and all compounds in the blank

Comp.	Temp. (K)	E corr.	I corr.	I corr./ r	Resis	Anodic (β_a)	Cathodic (β_c)	Corr. rate	IE%
Blank	298	-0.527	392.1	7.842E-4	65.32	0.112	0.125	3.849	-
	308	-0.521	412.4	8.248E-4	64.69	0.112	0.136	4.048	-
S (0.01)	298	-0.871	47.20	9.440E-5	1994	0.589	0.343	0.463	88
	308	-0.802	56.63	1.133E-4	1606	0.407	0.432	0.556	86
S(0.05)	298	-0.852	39.93	7.986E-5	2127	0.468	0.336	0.392	90
	308	-0.800	47.40	9.480E-5	1613	0.342	0.363	0.465	89
Ag(I) (0.01)	298	-0.829	35.30	7.060E-5	2075	0.395	0.294	0.347	91
	308	-0.770	45.11	9.021E-5	1427	0.315	0.280	0.443	89
Ag(I) (0.05)	298	-0.801	29.60	5.921E-5	2570	0.397	0.314	0.291	92
	308	-0.777	34.70	6.940E-5	1991	0.318	0.319	0.341	92
Cu(II) (0.01)	298	-0.732	40.76	8.151E-5	1437	0.217	0.356	0.400	90
	308	-0.767	44.05	8.810E-5	1443	0.270	0.320	0.432	89
Cu(II) (0.05)	298	-0.756	32.89	6.579E-5	1469	0.196	0.258	0.323	92
	308	-0.769	35.14	7.028E-5	1451	0.229	0.241	0.345	91

Conclusion

The Schiff base ligand derived from 2-hydroxy-5-nitrobenzaldehyde and *o*-aminobenzenethiol was synthesized in addition to its two complexes with copper and silver ions. The ligand was synthesized by the condensation reaction between 2-hydroxy-5-nitrobenzaldehyde and *o*-aminobenzenethiol. While Cu(II) and Ag(I) complexes were synthesized by the reflux reaction of both metal salts with the ligand. In both complexes the ligand acts as a tridentate ligand coordinates with the metals through the imine, hydroxyl and the thione groups. The coordination with the metals was confirmed by the available characterization techniques. The octahedral and the tetrahedral structures were suggested to copper(II) and silver(I) respectively. The corrosion inhibition of the synthesized compounds was tested in acidic medium at two temperatures (298 K and 308 K) and two concentrations (0.01 and 0.05 M) of the synthesized compounds. The results show the high corrosion inhibition of both complexes (92%) at the highest concentration (0.05 M) and at the two different temperatures in comparison with the ligand which shows a lower corrosion inhibition effect with a protection efficiency (90%).

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