

Synthesis, Physicochemical, Thermal and Fluorescence Studies of Cobalt(II) Complex with Tridentate (ONS) Schiff Base Ligand Derived From 2-Aminophenol and 2-Acetylthiophene

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Abstract

A tridentate Schiff base ligand HL(ATS), N-[2-acetylthiophene]-salicylideneimine was derived from the 1 : 1 condensation of 2-acetyl thiophene and 2-aminophenol was prepared and characterized by elemental analysis, FT-IR, Mass, ¹H-NMR, UV-VIS, thermogravimetric analysis and fluorescence spectra. Cobalt(II) complex was obtained by the reaction of the Schiff base ligand (HL) with cobalt(II) hexahydrate in 1 : 1 molar ratio. The cobalt (II) complex have been characterized by elemental analysis, FT-IR, ¹H-NMR, Ms, UV-VIS, magnetic moment, molar conductance, thermogravimetric analysis (TGA) and fluorescence spectra. Elemental analysis (CHNS) data proved 1 : 1 (M : L) stoichiometry for the reported cobalt (II) complex. IR spectra showed that cobalt (II) complex was coordinated in metal ions via the imine N, O and S atoms. Magnetic and UV-VIS spectra, showed that the geometrical structure of cobalt(II) complex is an octahedral. The molar conductance data revealed that the cobalt(II) complex is non-electrolyte. Thermogravimetric analysis (TGA) revealed the high thermal stabilities.

Keywords: 2-acetyl thiophene and 2-aminophenol, cobalt(II) complex,

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1. Introduction

Schiff bases represent one of the most widely utilized classes of ligands in metal coordination chemistry and the chemistry of Schiff bases is an area of increasing interest [Atkins, A et al., 1985 and Raman, N et al., 2009]. Schiff bases are capable of forming coordinate bonds with many metal ions via azomethine or phenolic groups, and so they have been used for synthesis of metal complexes due to their easy formation and strong metal binding ability [Cozzi, P.G, 2004 and Holm, R.H et al., 2011]. Schiff bases have wide applications in analytical chemistry and catalysis [Dhahagani, K et al., 2014; Small, B.L and Brooker, M, 1998]. The interest in Schiff bases compounds as analytical reagents is increasing since they enable simple and inexpensive determinations of different organic and inorganic substances [Estrela dos Santos, E.R et al., 2003]. Moreover, it was reported that the Schiff bases containing ONS donors have an important role in biochemistry [Saxena, A et al., 1981]. Particular attentions has been devoted to the metal Schiff base complexes. Schiff bases have been widely used as ligands because of the high stability of coordination compounds. There is a continuing interest in metal complexes of Schiff bases because of the presence of hard nitrogen or oxygen and soft sulfur donor atoms in the back bone of these ligands. Schiff base ligand have numerous applications such as fluorescence. Many of tridentate Schiff base ligand showed enhanced fluorescence. Moreover, luminescent compounds of Schiff base ligand and its transition metal complexes are attracting much current research interest because of their application including emitting materials for organic light , light materials for photofluorescent sensors for organic or inorganic analytes. In the present work, we described the cobalt (II) Schiff base complex produced from the reaction of cobalt (II) chloride

hexa hydrate with Schiff base ligand HL(ATS) N-[2-acetylthiophene]salicylideneimine.

2. Material and Methods

2.1. Materials and physical measurements

All chemicals were reagent grade quality purchased from commercial sources and used as received. Elemental analysis (CHNS) was performed on a Perkin Elmer 2400 elemental analyzer. FT-IR measurements (KBr pellets) were carried out on a Shimadzu 8000 FT-IR spectrometer at the central laboratory, Ain Shams University, Egypt .

¹H-NMR measurements were performed on a Varian-mercury 300 MHz spectrometer at the Microanalytical Center, Cairo University, Giza, Egypt. Samples were dissolved in DMSO with TMS as internal reference. Mass spectroscopy measurements of the solid ligands and complexes were carried out on a JEOL JMS-AX500 spectrometer at the National Research Center, Giza, Egypt, magnetic measurements of the complexes were measured by the Gouy method at room temperature using a magnetic susceptibility balance, Sherwood Scientific, Cambridge Science Park, Cambridge England. Thermogravimetric analysis(TG and DTG) were carried out under N₂ atmosphere with a heating rate of 10° C/min. Using a Shimadzu DT-50 thermal analyzer at the Microanalytical Center, Cairo University, Giza, Egypt. All conductivity measurements were performed in DMF (1X10⁻³M) at 25° C, by using WAP, GMP 50 conductivitymeter. Ultraviolet spectra were recorded using a Shimadzu UV 1800 spectrophotometer in the range (200-800 nm) at the Micro analytical Center, Cairo University, Giza, Egypt. The photo luminescent properties of all complexes were studied using a Jenway 6270 fluorimeter ,Ain Shams University. All melting points were measured using an

ordinary MEL-TEMP II Laboratory device (U.S.A), melting point apparatus.

2.2- Synthesis

2.2.1. Synthesis of the tridentate Schiff base ligand N-[2-acetylthiophene]salicylideneimine Schiff base HL (ATS).

The ligand was synthesized by slow addition of 2-acetylthiophene (1.09 ml, 10 mmole) in 50 ml ethanol to 2-aminophenol (1.09 gm, 10 mmole) in 50 ml ethanol. The reaction mixture was heated to reflux for 4 hrs. The dark brown product obtained after cooling was filtered off and washed with different amounts of 1,2-dichloromethane then diethylether, fine crystals were obtained by recrystallization from hot ethanol . The yield was (1.645gm, 70%), m.p. (157-159°C), Anal. Calc. for [C₁₂H₁₁NOS] (M.Wt: 217), C, 66.36; H, 5.07 ; N, 6.45; S, 14.75%, Found: C; 66.40; H; 4.87; N; 6.60; S; 14.60%; IR (KBr, v/cm⁻¹); 3375 v(OH), 1602v(C=N), 1467v(C=C), 743v(C-S) and 1141v(C-O). ¹H-NMR (300 MHz, DMSO-d⁶, δ (ppm): 8.879 (1H, s, -OH), 6.358-7.722 (phenolic hydrogen and acetyl thiophene sulfur protons, 3.172 (3H, s,CH₃).

2.2.2. Synthesis of the cobalt Schiff base complex.

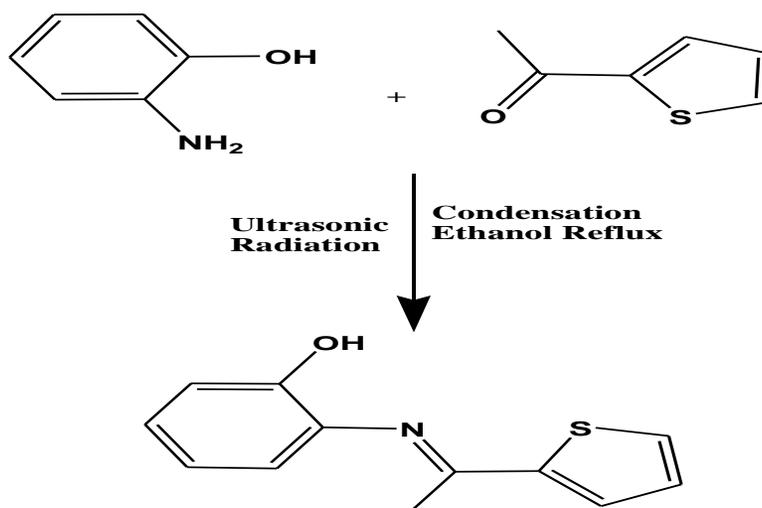
A red solution of CoCl₂.6H₂O (0.455 gm, 1mmole) in ethanol (50ml) was added gradually with constant stirring to a solution of ligand HL(ATS) (0.217gm, 1mmole) in ethanol (50ml). The solution was refluxed for 2hrs. A red precipitate was formed and washed with small amounts of ethanol then diethylether. The yield was (0.571 gm, 85%), m.p. (315-316°C), Anal. Calc. for [C₁₂H₂₃NO₇SCl₂Co] (2): (M.Wt: 453.9), C, 31.73 ; H, 5.07 ;N, 3.08; S, 7.05%. Found: C, 31.64; H, 5.23;

N, 3.06; S, 7.03%; IR (KBr, ν/cm^{-1}): 3383 ν (OH), 1591 ν (C=N), 755 ν (C-S), 1175 ν (C-O), 584 ν (Co-N), 490 ν (Co-O), 427 ν (Co-S) and 412 ν (Co-Cl).

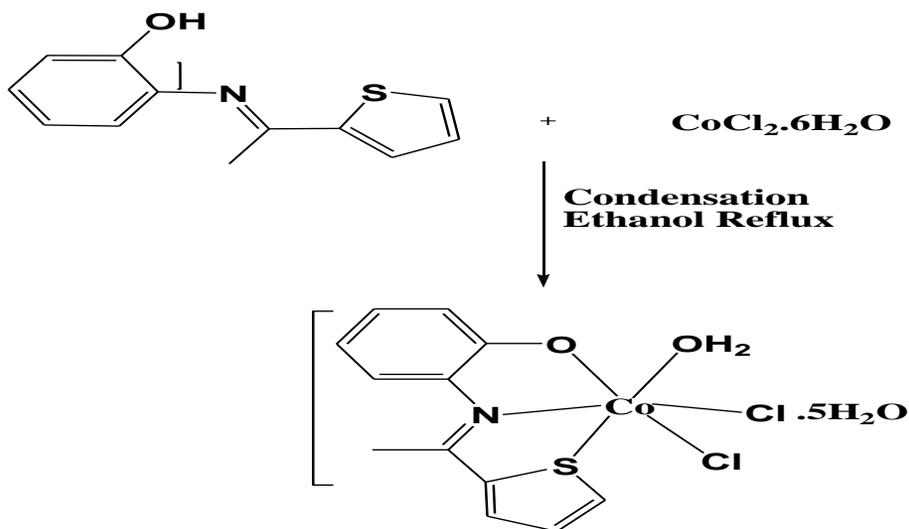
3. Results and Discussion.

3.1. Synthesis and characterization of compounds

The tridentate Schiff base ligand HL(ATS) was synthesized by the condensation of 2-aminophenol with 2-acetyl thiophene in EtOH in a (1 : 1) M ratio under heating on an ultrasonic radiation and the dark brown crystals of the ligand were obtained Scheme (1). The Schiff base ligand was reacted with cobalt (II) complex in (1 : 1) M ratio in ethanol Scheme (2). The ligand obtained was soluble in 1,2-dichloromethane, chloroform, acetonitrile (CH_3CN), methanol and partially soluble in acetone, dimethylsulfoxide (DMSO). The cobalt (II) complex was insoluble in acetone and methanol but soluble in 1,2-dichloromethane, dimethylformamide (DMF) and dimethylsulfoxide (DMSO).



Scheme 1. Schematic representation for the formation of Schiff base ligands N-[2-acetylthiophene]salicylideneimine HL(ATS).



Scheme 2. Schematic representation for the formation of cobalt(II) Schiff base complex.

3.2. Spectroscopy

3.2.1. IR spectra

In the IR spectra Figure (1), the IR spectra of the cobalt complexes are compared with the free ligand in order to determine the coordination sites that may be involved in chelation. Upon comparison, it was determined that the $\nu(\text{OH})$ stretching vibration which is found in the free ligand at 3375 cm^{-1} disappeared in the spectra of cobalt (II) complex, indicating the participation of OH group in chelate formation. Cobalt complex showed a broad band in the region 3383 cm^{-1} assigned to $\nu(\text{OH})$ of the coordinated or hydrated water molecules associated with the complex which were confirmed by elemental and thermal analysis. But the participation of the OH group in the complex were evidence from the shift in position of the $\delta(\text{OH})$ in plane deformation vibration from 1267

cm^{-1} in the spectrum of the free ligand and found at 1278 cm^{-1} in the spectra of the complex. Additionally the IR spectrum of the ligand HL (ATS) exhibits medium band at 1141 cm^{-1} which may be referred to $\nu(\text{C-O})$ (phenolic) and shifted toward higher frequency in the cobalt (II) complex. This shift confirms the participation of the phenolic oxygen in coordination to the metal cation. The IR spectra of the free ligand showed a strong band at 1602 cm^{-1} which is characteristic of the azomethine group. Coordination of the Schiff bases is expected to reduce the electron density and so the azomethine nitrogen $\nu(\text{C=N})$ shows a decrease in the stretching frequency for the cobalt (II) complex [Pouralimardan, O et al., 2007]. The strong to medium bands due to $\nu(\text{C-S})$ stretching vibration of acetylthiophene ring appeared at 743 cm^{-1} in the free Schiff base ligand [Nakamoto, K., 1978]. This band was shifted to 755 cm^{-1} in Co(II) complex. These shifts refer to the coordination through acetyl thiophene ring sulfur. Conclusive evidence of the binding is also shown by observation that new bands in the spectra of cobalt (II) complex appear in the region ($584, 490, 427, 412 \text{ cm}^{-1}$) characteristic to $\nu_{\text{M-N}}$, $\nu_{\text{M-O}}$, $\nu_{\text{M-S}}$ and $\nu_{\text{M-Cl}}$.

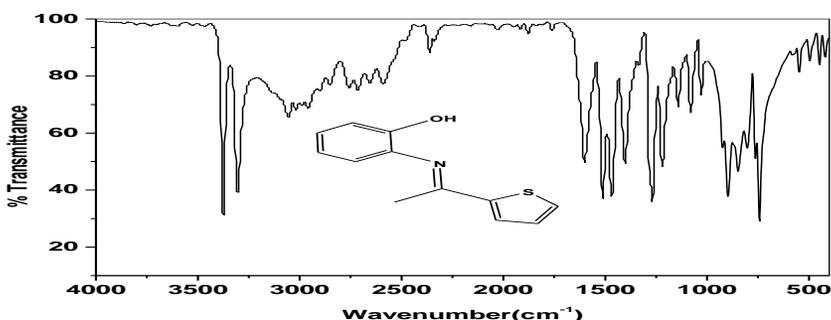


Figure (1): Infrared spectrum of the Schiff base ligand, HL(ATS).

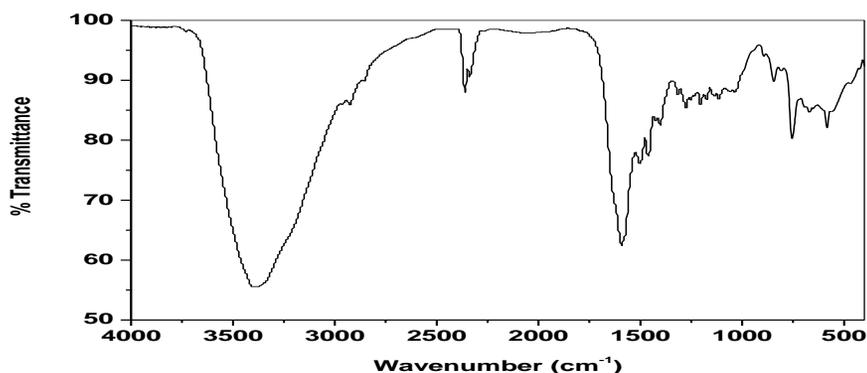


Figure (2): Infrared spectrum of $[\text{Co}(\text{HL})(\text{Cl})_2(\text{H}_2\text{O})]\cdot 5\text{H}_2\text{O}$.

3.2.2. $^1\text{H-NMR}$

The structural assignments were further supported by their $^1\text{H-NMR}$ spectra. $^1\text{H-NMR}$ spectra of the compounds were obtained in DMSO-d^6 at room temperature using TMS as the internal standard. In the $^1\text{H-NMR}$ spectra, the integral intensities of each signals were found to agree with the number of different types of protons present in the Schiff base ligand. In the Schiff base ligand, Figure (3), the protons peaks of CH_3 were appeared in 3.172 ppm. The multiplets of the aromatic protons in Schiff base ligand HL(ATS) appeared in the range (6.358 -7.722 ppm) with different multiplicity and coupling constants, the protons peak of OH were appeared in 8.879 ppm.

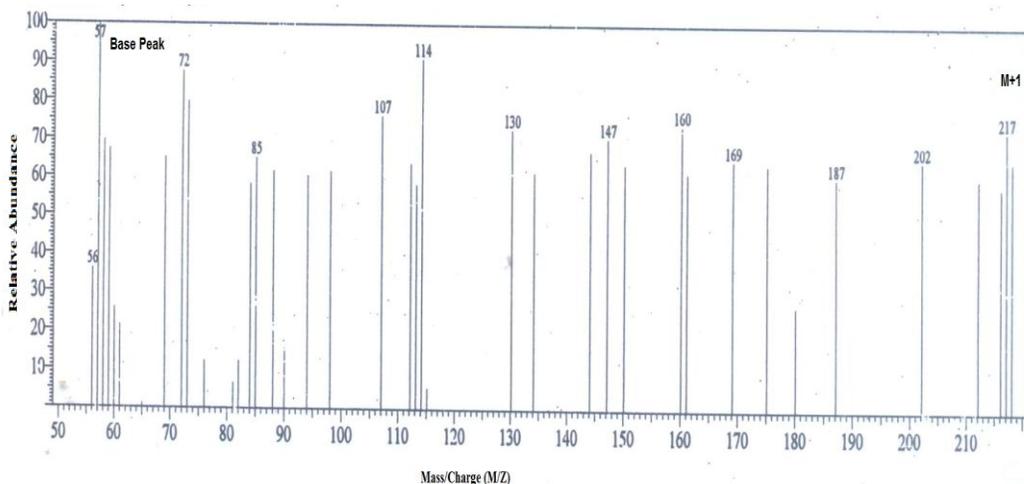
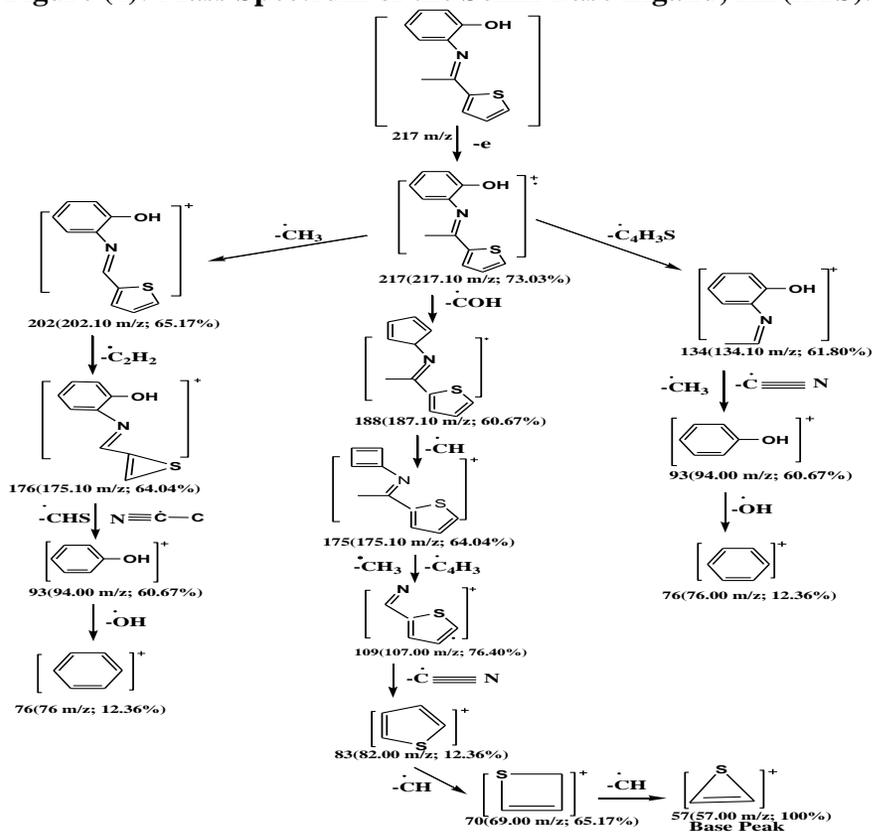


Figure (4): Mass Spectrum of the Schiff Base Ligand, HL(ATS).



Scheme (3): Mass fragmentation pattern of the Schiff base ligand HL(ATS).

Note: Calc. [Found; Intensity].

3.2.4. Electronic spectra, magnetic and molar conductance.

The electronic spectra of the Schiff base ligand HL(ATS) and its cobalt(II) complex were investigated in DMF solution. The Schiff base ligand and its cobalt (II) complex exhibit two absorption peaks at a lower energy region (>200 nm). The stronger and higher energy peak, Table (2) was attributed to the π - π^* transition of the azomethine chromophore, the phenyl and thiophene ring, while the weaker and less energetic peak is assigned to the n - π^* transition involving the promotion of the lone pair electron of azomethine nitrogen atom to the antibonding π orbital associated with the azomethine group [Bosnich, B., 1968]. The electronic spectrum of the cobalt (II) complex gives three bands, two of them at 736, 597nm [Neelkantana, M.A et al., 2008]. These are assigned to ${}^4T_{1g}(F) \rightarrow {}^4T_{2g}(F)$ and ${}^4T_{1g}(F) \rightarrow {}^4A_{2g}(F)$ transition, respectively suggesting that there is an octahedral geometry around Co(II) ion. The third band at 436 nm refers to charge transfer band [Lever, A.B.P., 1984].

The magnetic measurements obtained at room temperature for cobalt (II) complex is 5.82 B.M, i.e. close to the spin only value expected for cobalt (II) in octahedral environment. The molar conductance measurements of cobalt (II) complex was dissolved in DMF at 10^{-3} M, and the molar conductance of cobalt(II) complex in DMF is $14.75 (\Omega^{-1}\text{mol}^{-1}\text{cm}^{-1})$ is non ionic in nature and non electrolytes [Usha, A.K and Chandra, S., 1992].

Table (2): Electronic spectral data (nm), magnetic moment and molar conductance of Schiff base ligand HL(ATS) and its cobalt (II) complex.

Compound	π - π^*	n - π^*	Charge transfer	d-d transition	Magnetic moment	Molar conductance $\Omega^{-1}\text{mol}^{-1}\text{cm}^{-1}$
HL (ATS)	266	416
[Co(HL)(Cl ₂)(H ₂ O) ₂].5H ₂ O	277	412	436	597, 736	5.82	14.75

3.2.5. Thermal studies

The thermal stability of the cobalt (II) complex was investigated using TGA. The thermogravimetric analysis (TGA) was carried out at a heating rate of 10 °C/min in a nitrogen atmosphere over a temperature range (25- 1000°C). The thermogravimetric analysis of [Co(HL)(Cl₂)(H₂O)].5H₂O involves three decomposition steps, Table (3), Figure (5). The first step from (29-102°C) showed removal of the four lattice water with an estimated mass loss of 14.923 (Calc., 15.863%). The second step from (103-309°C) showed removal of Cl₂ with an estimated mass loss of 19.139 (Calc., 18.591%). In the last step from (311-1000°C) corresponds to the loss of 2H₂O, CN, OH, 3H₂, SH and CH₃ with an estimated mass loss of 42.379 (Calc., 42.779%). The total mass losses of the decomposition steps is found to be 76.441 (Calc., 77.233%).

Table (3): Thermoanalytical results (TGA, DTG) of Co(II) complex.

Complex	Steps	Temperature range (°C)	DTG Peak	TG Weight Loss %		Assignment
				Calc	Found	
[Co(HL)(Cl ₂)(H ₂ O)].5H ₂ O	1 st	29-102	63.5	15.863	14.923	Loss of four lattice water
	2 nd	103-309	219	18.591	19.139	Loss of Cl ₂
	3 rd	311-1000	467	42.779	42.379	Loss of 2H ₂ O, CN, OH, 3H ₂ , SH, CH ₃

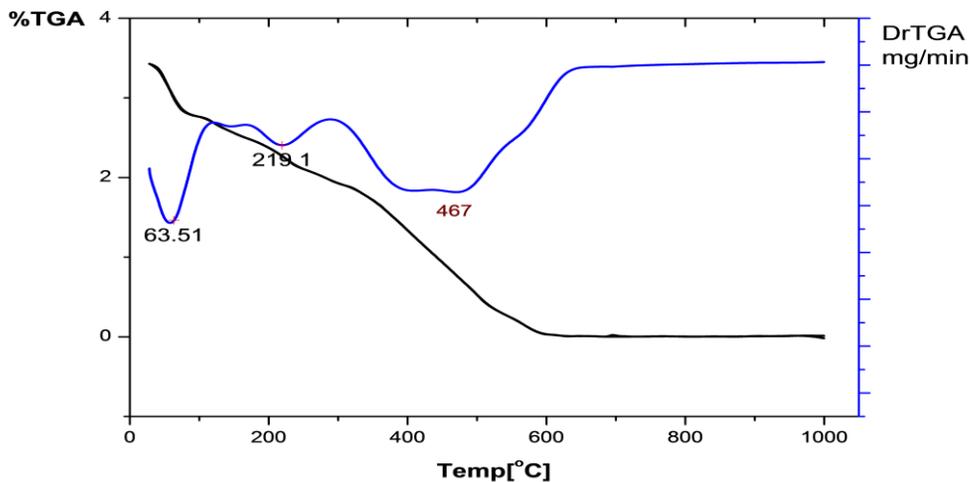


Figure (5): TGA/DTG curves of $[\text{Co}(\text{HL})(\text{Cl})_2(\text{H}_2\text{O})].5\text{H}_2\text{O}$.

3.2.6. Kinetic data

The kinetic and thermodynamic activation parameters of decomposition processes of complexes namely activation energy (ΔE^*), enthalpy of activation (ΔH^*), entropy (ΔS^*) and Gibbs free energy change of the decomposition (ΔG^*) were evaluated graphically by employing the Coats-Redfern relation [Coats and Redfern, 1964].

$$\log \left[\log \left\{ \frac{W_f}{(W_f - W)} \right\} / T^2 \right] = \log \left[\frac{AR}{q\theta E^*} \left(1 - \frac{2RT}{E^*} \right) \right] - \frac{E^*}{2.303RT} \quad (1)$$

Where E^* , R , A and θ are the heat of activation, the universal gas constant, pre-exponential factor and rate of heating respectively, W_f is the mass loss at the completion of the decomposition reaction, W is the mass loss up to temperature T . Since $1 - 2RT/E^* \sim 1$, the plot of the left hand side of equation (1) against $1/T$ would give a straight line. The other kinetic parameters; the entropy of activation (ΔS^*), enthalpy of activation (ΔH^*) and the free energy change of activation (ΔG^*) were calculated using the following equations:

$$\Delta S^* = 2.303 \left(\log \frac{Ah}{KT} \right) R \quad (2)$$

$$\Delta H^* = E^* - RT \quad (3)$$

$$\Delta G^* = H^* - T\Delta S^* \quad (4)$$

Where, (k) and (h) are the Boltzman and planck constants, respectively. The kinetic parameters are listed in Table (4). From the obtained results, it is apparent that ΔG^* values of the complexes acquire highly positive magnitudes. The high value of the energy of activation of the complexes revealed the high stability of the investigated complexes due to their covalent character [Taakeyama & Quinn, 1994]. The negative values of ΔS^* for the degradation process indicates that more ordered activated complex than the reactants and the decomposition reaction is slow [Frost & Pearson, 1961]. The positive values of ΔH^* mean that the decomposition process are endothermic.

Table (4): The kinetic and thermodynamic data of the thermal decompositions of cobalt complex.

Complex	Stage	Mid Temperature (°K)	ΔE^* (KJ mol ⁻¹)	ΔA (S ⁻¹)	ΔS^* (KJ/mol K)	ΔH^* (KJ/ mol)	ΔG^* (KJ mol)
<i>[Co(HL)Cl₂(H₂O)]·5H₂O</i>	1 st	302-375	213.55	4.38X10 ⁻¹	-180.59	208.8	311.5
	2 nd	376-582	237.29	2.84X10 ⁻²	-234.04	218.3	751.7
	3 rd	584-1273	384.11	1.20X10 ⁻²	-268.58	372.5	962.1

3.2.7. Fluorescence spectral studies

The emission spectra of the Schiff base ligand HL(ATS) and its cobalt (II) complex were investigated at room temperature in DMSO solution Figure (6). The schiff base ligand was characterized by emission

band around 504 nm upon photo excitation at 432nm. The emission observed in the Schiff base ligand was assigned to the π - π^* intraligand fluorescence. The fluorescence of the ligand was probably quenched by the occurrence of a photoinducted electron transfer (PET) process due to the presence of a lone pairs of electrons of the donor atoms in the ligand (N, O and S atoms). On the other hand, the cobalt(II) complex was characterized by the emission band at 560 nm with a lower emission than the Schiff base ligand [Majumder, A et al., 2006].

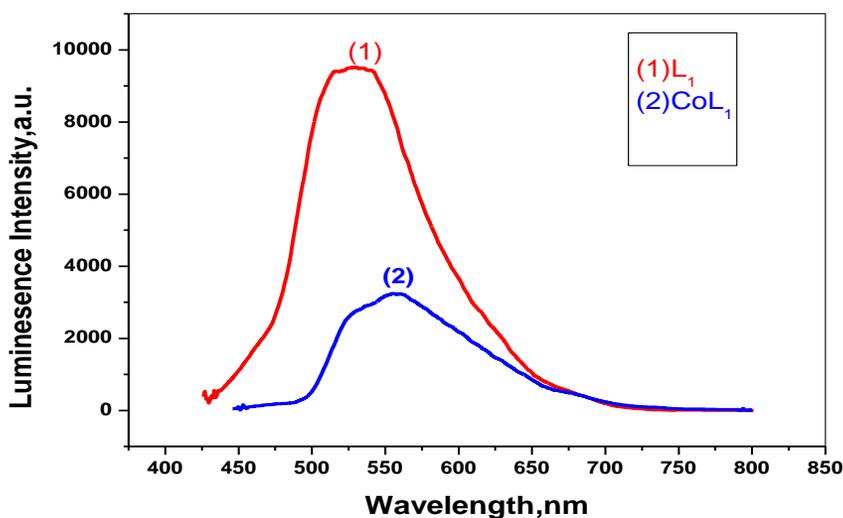


Figure (6): Emission spectra of : (1): Ligand (HL); (2) [Co(HL)Cl₂(H₂O)]. 5H₂O.

3.2.7.1. Effect of solvent and pH

The influence of the solvent on Schiff base ligand HL(ATS) and its cobalt (II) complex was studied. The results showed that the optimal solvent for Schiff base ligand and its cobalt (II) complex obtained is DMF as shown in Figure (7-8). The influence of pH on Schiff base ligand HL(ATS) and its cobalt complex were investigated in both acidic and alkaline media by the addition of dilute HCl and NH₄OH. The optimum

signal of Schiff base ligand and its cobalt(II) complex [Liu, Q.X., 2009] were recorded in strongly alkaline conditions. The highest intensity of the Schiff base ligand and its cobalt (II) complex were obtained at pH= 9.4 Figure (9.10).

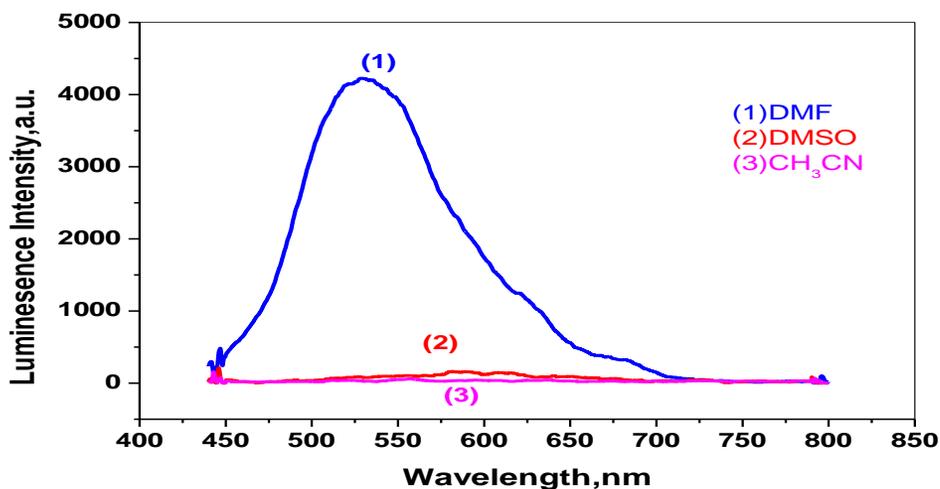


Figure (7): Effect of solvent on Schiff base ligand HL(ATS).

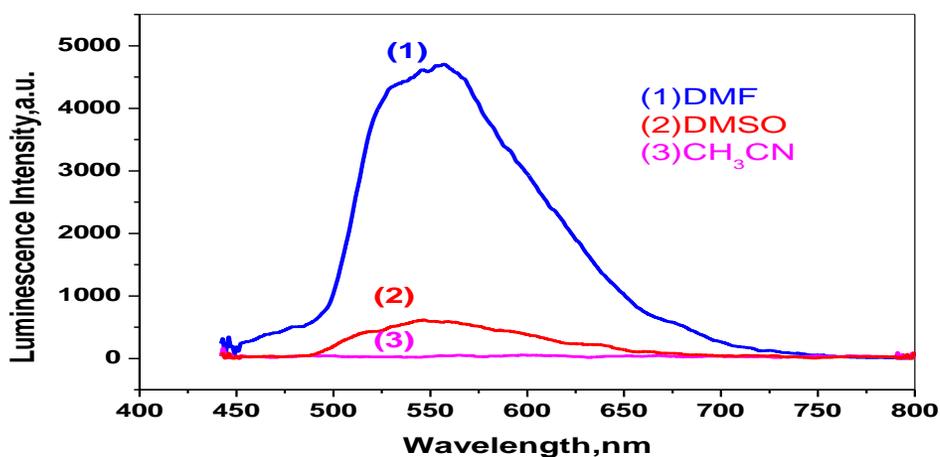


Figure (8): Effect of solvent on [Co(HL)(Cl)₂(H₂O)]. 5H₂O.

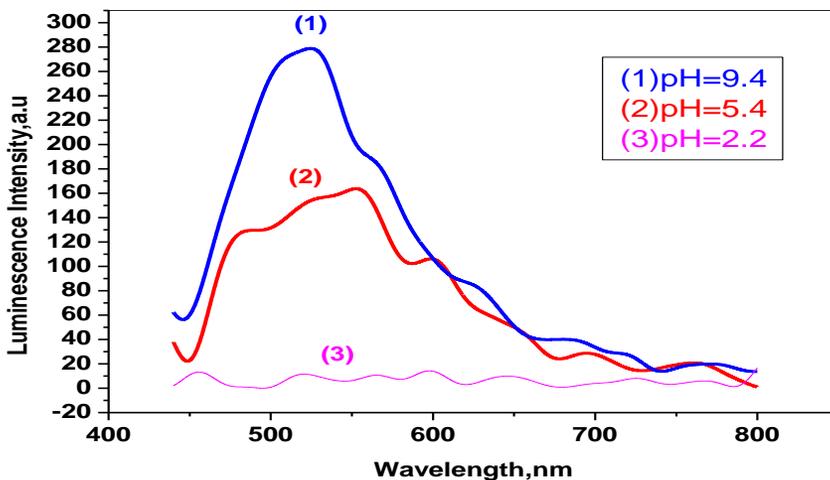


Figure (9): Effect of pH on Schiff base ligand HL(ATs).

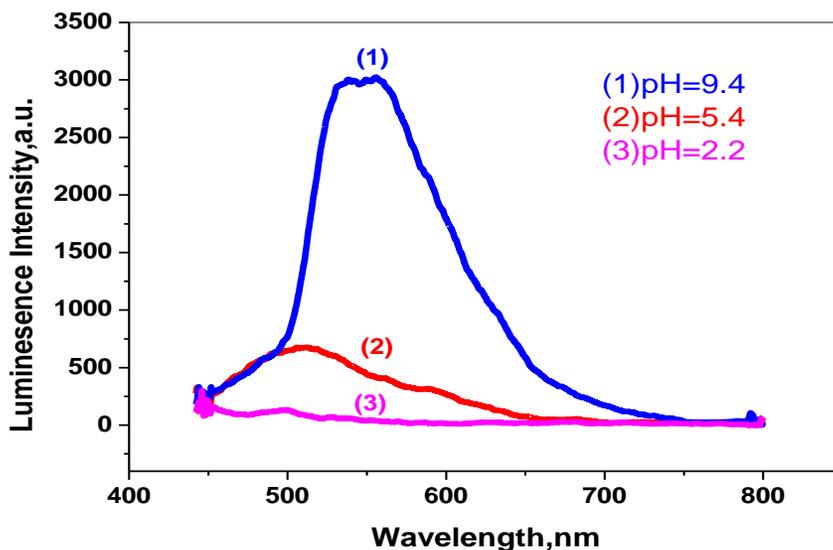


Figure (10): Effect of pH on $[\text{Co}(\text{HL})(\text{Cl})_2(\text{H}_2\text{O})] \cdot 5\text{H}_2\text{O}$.

4. Conclusion

In this work, the tridentate Schiff base ligand N-[2-acetylthiophene]salicylideneimine HL(ATs) and its cobalt(II) complex were synthesized and characterized using spectroscopic methods. IR

spectra of the Schiff base ligand HL(ATS) showed that tridentate ligand was coordinating through ONS donors atoms of phenolic oxygen, azomethine nitrogen and acetyl thiophene sulfur in cobalt (II) complex.. From magnetic and UV-Vis spectra, it is found that the geometrical structure of cobalt (II) complex is an octahedral. Thermogravimetric analysis revealed that the cobalt (II) complex was studied and the activation thermodynamic parameters such as, activation energy, enthalpy, entropy and Gibbs free energy change of decomposition of cobalt(II) complex decomposition indicated that the their stability was determined by Coate-Redfern method. Fluorescence studies indicated that the Schiff base ligand HL(ATS) and its cobalt (II) complex can serve as potential photoactive materials, as indicated from their characteristic fluorescence properties.

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الملخص باللغة العربية

تحضير و دراسه الخواص الطبيعيه ،الكيميائيه ،الحراريه و الضوئيه لمتراكب الكوبلت الثنائي مع قاعده شيف ثلاثيه المخلب المشتقه من 2-امينو فينول مع 2-اسيتيل ثيوفين

مناره احمد ايوب ، ايمان حامد عبد الناصر ، منى عبد العزيز، مريم جرجس رزق سعيد
قسم الكيمياء -نبات-عين شمس.

1-دراسه الخواص الطبيعيه و الكيميائيه بهدف التعرف على تركيب الليجند العضوي المستخدم و ذلك بتعين درجه الأنصهار و التحليل العنصرى و طيف إمتصاص الاشعه تحت الحمراء و طيف الكتله و ميكانيكيه التكسير و طيف الرنين النووى المغناطيسي و طيف امتصاص الاشعه فوق بنفسجيه و المرئيه و الخاصيه الضوئيه. و قد تم اثبات وجود مجموعه الازوميثين و الهيدروكسيل الكحوليه و الكبريت لحلقه ثيوفين و تعتبر هذه المركبات ثلاثيه المخلب.
2-تم دراسه تفاعل الليجند المحضر مع ايون الكوبلت الثنائي و تم الحصول على المتراكب الكوبلت الثنائي فى حاله الصلبه و تمت دراسته و اثبات التركيب الجزيئى و الفراغى له بواسطه التحليل العنصرى، مطياف الكتله، ميكانيكيه التكسير، طيف امتصاص الاشعه تحت الحمراء، فوق بنفسجيه و المرئيه، الرنين الالكترونى المغزلى و ايضا الخواص المغناطيسيه و الحراريه فقد تم التوصل الى ان الشكل الفراغى ثمانى الواجه حول ذره الكوبلت الثنائي و قد اظهرت النتائج انه امكن ترابط الليجند مع الفلز عن طريق زوج الكترولونات الحره الموجوده على ذره النيتروجين فى مجموعه الازوميثين و زوج الالكترولونات الحره الموجوده على ذره الاكسجين فى مجموعه الهيدروكسيل و زوج الالكترولونات الحره الموجوده على ذره الكبريت فى حلقه الاستيل ثيوفين.

3-تم دراسه الخواص الحراريه للمتراكب و تعيين كل من ΔE^* , ΔA^* , ΔS^* , ΔH^* , ΔG^* .

4-تم دراسه الخاصيه الضوئيه لكل من الليجند و المتراكب كما تم اثبات ان الليجند و المتراكب لديهم امكانيه التالىق كما تم دراسه تاثير الأس الهيدروجينى و المذيبات المختلفه لكل من الليجند و المتراكب و اثبتت النتائج ان مذيب ثنائى مثيل الفورماميد افضل مذيب وافضل أس هيدروجينى ظهر عند $pH=9.4$.

5- النتائج العمليه التى تم الحصول عليها تتفق تماما مع التفسيرات المرجعيه.