Synthesis and Antibacterial Activity of Isatin Schiff Base Derivative with 3-Aminoacetophenone and its Ni^{II}, Co^{II} Transition Metals Complexes

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Abstract-The (E)-3-(3-acetylphenylimino) indolin-2-one (Bidentate) ligand type [HL] has been prepared from Isatin and 3-aminoacetophenone in the presence of KOH. In general, the ligand contains oxygen (O) and nitrogen (N) donor atoms. The reaction of Isatin and 3-aminoacetophenon was carried out in ethanol by condensation reaction at 80°C with reflux for 4 h, to form [HL] ligand type. This ligand has been used to prepare Ni^{II} and Co^{II} complexes in the ratio of 1:1 metal-ligand. All compounds have been characterized by spectroscopic methods (Fourier transform infrared and ultravioletvisible), C.H.N, thin-layer chromatography, mass spectrum, X-ray diffraction, magnetic moment, conductivity measurements and milting point, the synthesized ligand and its metal complexes have been tested for their antibacterial activity against Staphylococcus aureus and Bacillus subtilis using agar disc diffusion method. The ligand and its complexes showed significant activities against S. aureus and B. subtilis. Our study revealed the formation of four coordinate square planar complexes around Ni^{II} and Co^{II} metal ions.

Index Terms—3-aminoacetophenone, Bidentate ligand and (N) donor atoms, Isatin, Schiff base.

I. INTRODUCTION

Schiff bases have played an important role in the development of coordination chemistry as they readily form stable complexes with most of the transition metals. The chemistry of Schiff base ligands species has been gaining considerable interest primarily because of their fascinating structural diversities (Asadi, et al., 2011; Monfared, et al., 2011; Chamayou, et al., 2011). The electrophilic carbon atoms of aldehydes and ketones can be targets of nucleophilic attack by amines. The end result of this reaction is a compound in which the C=O double bond is replaced by a C=N double bond. This type of compound is known as an imines, or Schiff base with the general formula R₁R₂C=NR₃,

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Corresponding author's e-mail: eman.ibraheem@koyauniversity.org Copyright © 2018 Eman I. Alsalihi, Aeed S. Al-Fahdawi. This is an open-access article distributed under the Creative Commons Attribution License. where R is an organic side chain. In this definition, Schiff base is synonymous with azomethine. Many Schiff base complexes show excellent catalytic activity in various reactions and in the presence of moisture (Abu-Dief Ahmed and Mohamed Ibrahim, 2015). The Schiff bases are widely used for industrial purposes and also exhibit a broad range of biological activities. This short review compiles examples of the most promising antimalarial, antibacterial, antifungal, and antiviral Schiff bases. An overview of synthetic methodologies used for the preparation of Schiff bases is also described (Da Silva Cleiton, et al., 2011). Isatin is a vellowish red crystalline compound soluble in hot water, used for the preparation of vat dyes. Formula: C₈H₅NO₂. Is found in many plants and Schiff bases of Isatin are investigated for their pharmaceutical properties (Jarrahpour and Khalili, 2005). This was prepared according to the literature method, Isatins can be made from the corresponding indole in good yield by a mixing of InCl, and IBX in an acetonitrile-water solution at 80°C (Yadav, et al., 2007). The electrophilic carbon atoms of Isatin carbonyl group can be targets of nucleophilic attack by amines (Jarrahpour and Khalili, 2005). 3-Aminoacetophenone is one of the most typical aromatic carbonyls which show photochemical properties3-aminoacetophenone with molecular formula C₀H₀NO, having melting point 96-98°C. Another additional interest that 3-aminoacetophenone derivatives possess some degree of local anesthetic activities (Nomenclature of Organic Chemistry, 2014). Acetophenone is used for the synthesis of many pharmaceuticals (Braish and Gadamasetti, 2007). Acetophenone is recovered as a by-product of the oxidation of ethylbenzene to ethylbenzene hydroperoxide (Siegel H., Eggersdorfer M., 2005). In this work, we report the synthesis characterization of Isatin compound with 3-aminoacetophenone and its complexes with selected transition elements such as Ni¹¹ and Co¹¹, the antibacterial activity of the prepared complexes has been tested against Gram-positive Staphylococcus aureus and Bacillus subtilis (Shaker, et al., 2013) (Fig. 1).

II. EXPERIMENTAL

Reagents were purchased from Fluka and Redial - Dehenge Chemical Co. and used without further putrefaction. Fourier transform infrared (FT-I.R) spectra were recorded in the range 4000–400/cm. Electronic spectra of the prepared compounds





Fig.1. The synthesis route of the complexes (Where $M = Ni^{11}$ and Co^{11})

were measured in the region 200–900 nm for 10⁻³ M solutions in methanol for the ligand and in distilled water for the complexes at 25°C using a Shimadzu 160 spectrophotometer with 1.000–0.001 cm matched quartz cell. Elemental microanalyses were performed on a (C.H.N) analyzer.

A. Syntheses of the ligand (E)-3-(3-acetylphenylimino) indolin-2-one [HL]

The ligand (E)-3-(3-acetylphenylimino)indolin-2-one type [HL] has been prepared from dissolving (0.3 g and 2.039 mmole) of Isatin in 25 ml ethanol, and mixed with a solution of 3-aminoacetophenone (0.275 g and 2.034 mmole) under refluxed for 4 h to gain (0.2 g) of a pale brown precipitate after filtration and drying at room temperature, to yield 66.6%, mp (63°C).

B. Syntheses of [Ni(L)Cl₂] complex

[Ni(L)Cl₂] complex has been prepared from 0.15 g, 0.567 mmole of [HL] dissolved in 25 mL methanol with a solution of 0.134 g, 0.663 mmole of NiCl₂.6H₂O. Moreover, refluxed for 2 h to obtain (0.11 g) pale yellow precipitate. Yield 73%, mp 134°C.

C. Syntheses of $[Co(L)Cl_2]$ complex

The same method used to prepare $[Ni(L)Cl_2]$ complex was used to prepare $[Co(L)Cl_2]$ complex but with a solution of 0.13 g, 0.546 mmole of CoCl₂.6H₂O to form (0.13 g) deep brown precipitate. Yield 86.6%, 118°C. Table I shows the microanalysis results and some physical properties for the ligand and its complexes.

III. RESULTS AND DISCUSSION

A. Synthesis of the ligand [HL]

The (FT-I.R) spectra for the starting materials of the ligand [HL], the essential infrared data are summarized in Table II.

In the spectrum of 3-aminoacetophenone, there are two sharp absorption bands at 3470 and 3450/cm that assigned to the stretching vibration of v_{sy} (N-H) and v_{asy} (N-H) of the primary amine (R-NH₂) group (Nakamoto, 1997) (Fig. 2). These bands have been disappeared in the spectrum of the ligand [HL], because of its condensation reaction with Istian.

In the spectrum of Isatin Fig. 3., there is a band at 3450.6/cm due to the v(N-H) of the secondary amine (R₂-NH) group (Rostkowska, et al., 1993), is shifted to lower frequency about (50/cm) as shown in the spectrum of the ligand [HL] as a consequence of the coordination between both of Isatin and 3-aminoacetophenone.

The appearance of a new band at 1550/cm range in the spectrum of [HL] ligand was assigned to the v(C=N)stretching, indicating formation a new compound and Schiff base reactions (Ali, et al., 1981; Ivanov and Nikolova, 2008) 3-aminoacetophenone and Istian. Figs. 2 and 3 displayed strong bands at 1728/cm and 1695/cm range, assigned to the stretching vibration of v(C=O) group, respectively, which are shifted to lower frequency at 1650/cm compared with the spectra of the ligand [HL] (Anuradha and Rajarel, 2011; Colchoubian, et al., 1999) (Fig. 4).

U.V-Vis spectrum for [HL], Fig.5. exhibits a high intense absorption peak at 240 nm (41666/cm) ($\in_{max} = 450/molar/cm$), assigned for $n \rightarrow \pi^*$. A shoulder peaks at 290 nm (34482/cm) ($\in_{max} = 366/molar/cm$) and (339 nm) (29498/cm) ($\in_{max} = 120/molar/cm$) were assigned to $\pi \rightarrow \pi^*$ transitions (Colchoubian, et al., 1999; Kindeel, et al., 2013) (Table III).

B. Synthesis of the complexes

The reaction of the ligand [HL] with Co^{II} and Ni^{II} was carried out in methanol under reflux. All complexes are stable in the solid state. The analytical and physical data, Table I and spectral data Table III are compatible with the suggested structures. FT-I.R spectra of the complexes are shown in Figs. 6 and 7 and the predominant bands are summarized in Table II.

TABLE I THE MICROANALYSIS RESULTS AND SOME PHYSICAL PROPERTIES OF [HL] AND ITS COMPLEXES

M.W	Yield%	M p.°C	Color	Formed (cal. %)-(theo. %)				
				С	Н	Ν	Cl	Metal
264.28	66.6	63	Pale-Brown	71.55 72.72	4.01 4.58	9.16 10.60	-	-
463.78	73	134	Pale-Yellow	45.65 48.91	2.122.82	6.01 7.13	16.89 18.05	13.22 14.94
464.02	86.6	118°	Deep-Brown	44.01	2.31	6.11	17.00	13.06
	M.W 264.28 463.78 464.02	M.W Yield% 264.28 66.6 463.78 73 464.02 86.6	M.W Yield% M p.°C 264.28 66.6 63 463.78 73 134 464.02 86.6 118°	M.W Yield% M p.°C Color 264.28 66.6 63 Pale-Brown 463.78 73 134 Pale-Yellow 464.02 86.6 118° Deep-Brown	M.W Yield% M p.°C Color 264.28 66.6 63 Pale-Brown 71.55 72.72 463.78 73 134 Pale-Yellow 45.65 48.91 464.02 86.6 118° Deep-Brown 44.01	$ \begin{array}{c c c c c c c c c c c c c c c c c c c $	$ \begin{array}{c c c c c c c c c c c c c c c c c c c $	$ \begin{array}{c c c c c c c c c c c c c c c c c c c $

 Table II

 FT-I.R spectral data (wave number y)/cm of the derivative ligand [HL] and its precursors with the complex

Compound	υ(N-H) Primary-R-NH ₂	υ(N-H) Secondary R ₂ -NH	υ(C=O)	v(C=N)-Imine	M-O M-N
3-aminoacetophenone	3470 3450	-	1695	-	-
Istian	-	3450.6(sh)	1728(sh)	-	-
[HL]	-	3400	1650	1550	-
[Ni (L) CL ₂]	-	-	1670	1592	530 590
$[\text{Co}(\text{L})\text{Cl}_2]$	-	-	1665	1595	526 580

TABLE III

Electronic spectral data and conductance measurements of the ligand and its complexes

Compound	λnm	cm ⁻¹ υ wave number	∈ _{Max} /molar/cm	Assignments	conductance	B.M
[HL]	240	41666	450	n→π*		
	290	34482	366	$\pi \rightarrow \pi^*$		
	339	29498	120	$\pi \rightarrow \pi^*$		
[Ni (L) CL ₂]	240	41666	1141	Ligand field	20	2.9
2-	285	35087	440	C.T		
	410	24390	200	$^{1}A_{1} \rightarrow ^{1}E'$		
	425	23529	180	$^{1}A_{1} \rightarrow ^{1}E$		
$[Co(L)Cl_2]$	255	39215	904	Ligand field	16	1.6
	291	34364	1122	C.T		
	394	25380	309	${}^{4}A_{2}g \rightarrow {}^{4}T_{1}g$ (F		
	447	22371	103	${}^{4}A_{2}g \rightarrow {}^{4}T_{2}g$		



Fig. 2. The Fourier transform infrared spectrum of 3-aminoacetophenone

The band at 1650/cm⁻¹ of the v(C=O) stretching vibration (Ali, et al., 1981; Ivanov and Nikolova, 2008) of the ligand is shifted

to higher frequencies at 1670–1665 range for Ni^{II} and Co^{II} , complexes because of the coordination with the metal ions.



Fig. 3. The Fourier transform infrared spectrum of Isatin



Fig.4. The Fourier transform infrared spectrum of [HL] ligand



Fig. 5. The ultraviolet-visible spectrum of [HL] ligand

Moreover, the band at $1550/\text{cm}^1$ of $\upsilon(\text{C=N})$ imine (Anuradha and Rajarel, 2011; Colchoubian, et al., 1999) is shifted to higher frequencies about (40/cm) range for the complexes due to the forming ring system with the metal ions. Metal oxygen and metal nitrogen band further confirmed by the presence of

peaks at 530–590/cm and 526–580/cm range were assigned to ν (M-O) and ν (M-N) (Halli, et al., 2012; Jakels, et al., 1983) stretches for the Ni^{II} and Co^{II} complexes, respectively.

UV-Vis spectra of Ni^{II} and Co^{II} complexes Figs. 8 and 9, respectively, showed two intense peaks in the range 240 nm,



Fig.6. The Fourier transform infrared spectrum of [Ni(L)Cl2] complex



Fig. 7. The Fourier transform infrared spectrum of [Co(L)Cl2] complex



Fig. 8. The ultraviolet-visible spectrum of [Ni(L)Cl2] complex

(41666/cm), ($\in_{max} = 1141/molar/cm$) and 255 nm, (39215/cm⁻¹), ($\in_{max} = 904/molar/cm$) range assigned to the ligand field for Ni^{II} and Co^{II}, respectively (Lever, 1984). Another two peaks at 285 nm, (35087/cm), ($\in_{max} = 440/molar/cm$) and (291 nm), (34364/cm), ($\in_{max} = 1122/molar/cm$) range assigned to the charge transfer transition for Ni^{II} and Co^{II}, respectively (Al-Shihri, 2004). Another two peaks are detected in the visible region for Ni^{II} complex at 410 nm, (24390/cm), ($\in_{max} = 200/$

molar/cm) and (425 nm), (23529/cm), ($\in_{\max} = 180/\text{molar/cm}$) are due to $({}^{I}A_{I} \rightarrow {}^{I}E')$ and $({}^{I}A_{I} \rightarrow {}^{I}E')$ (" transitions indicating a tetrahedral structure (Lever, 1984) around Ni^{II}. So that for the two peaks of Co^{II} complex at 394 nm, (25380/cm), ($\in_{\max} = 309/\text{molar/cm}$) and (447 nm), (22371/cm), ($\in_{\max} = 103/\text{molar/}$ cm) are assigned to (${}^{4}A_{g}g \rightarrow {}^{4}T_{I}g_{(F)}$) and (${}^{4}A_{g}g \rightarrow {}^{4}T_{g}g$) transitions (AL-Shihri, 2004), suggesting a tetrahedral structure around Co^{II} ion. is assigned to d-d transitions (AL-Shihri, 2004).



Fig. 9. The ultraviolet-visible spectrum of [Co(L)Cl₂] complex



Fig. 10. The thin-layer chromatography for the ligand and its Ni^{II}, Co^{II} complexes

Thin-layer chromatography (TLC) measurement for the derivative ligands [HL] and its complexes were performed with Co^{II} and Ni^{II} is showed in Fig. 10. and Table IV. The appearance of new spots with different R_f compared with the R_f of the ligands, for Co^{II} and Ni^{II} indicated the formation of the complexes. The spots positions belong to Co^{II} , and Ni^{II} ion complexes are differ from the positions of the ligands spot.

Biological activity of the [HL] ligand and its two complexes was tested on two types of pathogenic bacteria using inhibition method (Anacona, 2006; Sultana and Arayne,2007). The two types of bacteria were Grampositive *S. aureus* and *B. subtilis*. The ligand [HL] showed inhibition diameter against the two types of bacterial after 24 h and this inhibition diameter was increased after 48 h (Fig. 11). Furthermore, experimental results indicated that



Fig. 11. The biological activity of [HL] and its $\rm Ni^{II},\, \rm Co^{II}$ complexes



Fig. 12. The X-ray powder diffraction of [Co(L)Cl2] complex

the complexes show more activity than the ligand under similar experimental conditions with the same kinds of bacteria.

Molar conductance in DMSO solutions lie in the (16 and 20/cm/mole) range indicating its electrolytic nature with (1:1)



Fig. 13. The mass spectrum of [HL]

TABLE IV THE TLC MEASUREMENTS FOR THE [HL] LIGAND AND ITS CO^{II} AND NI^{II} COMPLEXES

Compound	Range of R _f (mm)
[HL]	0.7
[Co (L)	3.3
$[N_1(I_1)]$	2.2
<u>CL₂]</u>	

TLC: Thin-layer chromatography

of the Coⁿ and Niⁿ, metal ion complexes (Kai, et al., 2009; Refat and Struct, 2007) (Table I).

Magnetic moment (1.6 and 2.9 B.M) value of the Coⁿ and Niⁿ ion complexes, respectively, as well as the other analytical data Table III is in agreement with the suggested structure of square planar geometry for the two complexes in the solid state (Uppadin, et al., 2001; Al-Jeboori, et al., 2010).

X-ray powder diffraction (XRD) pattern of Coⁿ complex shows well-defined crystalline peaks indicating that the sample is 37% crystalline in nature (Dokken, et al., 2009). An XRD powder diffraction pattern of the copper complex has been given in Fig. 12 of different scale particles are well coincident with each other, and it means that different forms of complexes have the same structure (Guillemet-Fritsch, et al., 2006). The sample has been dried and then scanned in the 20 range of 10–80° confirming square planar geometry around Coⁿ ion complex (Kavitha and Lakshm, 2017; Zheng, et al., 2017).

Mass spectrum shows the base peak at 264 related to the molecular weight of the ligand. Moreover, all the other fragmentations are compatible with the value of the fragments of the ligand as shown in Fig. 13.

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