

Synthesis, And Characterization Of Five New Metal Transition Complexes Derived From The Schiff Base Ligand N^1, N^4 -Bis(2-Hydroxybenzylidene)Succinohydrazide (H_4L)

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Abstract

Schiff base ligand N^1, N^4 -bis(2-hydroxybenzylidene)succinohydrazide (H_4L) was synthesized via condensation of salicylaldehyde and succinohydrazide in ethanol. The complexes were obtained from 1:2:1 ($H_4L/LiOH/MCl_2 \cdot nH_2O$) molar ratio reactions, yielding compound formulated as $[M_2(H_2L)_2] \cdot nH_2O$, where $M = Mn(II), Fe(II), Co(II), Ni(II)$ and $Cu(II)$. Elemental analysis, 1H and ^{13}C NMR, FT-IR and UV-Visible spectroscopies were used for the characterization of the ligand. The complexes were structurally studied using FT-IR, UV-Visible spectroscopies, conductance, and magnetic susceptibility measurements. All complexes are non-electrolytes in DMF solutions. In each complex unit two ligand molecules acting in hexadentate fashion bridges two metal ions are present. Thus, each metal ion of the binuclear unit is coordinated to two azomethine nitrogen atoms, two phenolate oxygen atoms and two carbonyl oxygen atoms, resulting in a hexacoordinated metal ion. The environment around each metal ion is described as octahedral geometry owing to the results of the magnetic susceptibility measurement and the UV-visible spectra analyses.

Keywords: Salicylaldehyde, Succinohydrazide, Complex, FTIR, UV-Visible, Magnetic moments.

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I. Introduction

Dihydrazone compounds are an important class of compounds in the field of coordination chemistry because of the presence of multiple coordination sites and the compounds they can generate after complexation [1–5]. The presence of nitrogen and oxygen atoms in dihydrazones and the ability of these molecules to act in neutral or deprotonated form allow the synthesis of interesting coordination complexes with very varied structures [6–8]. Indeed, the complexes formed by these types of Schiff bases can not only present fascinating structures but also interesting properties. Some of these Schiff bases exhibit important biological activities that can be enhanced after complexation with transition metals [9–14]. Schiff bases obtained from succinohydrazide have the particularity of being stable due to the strong delocalization and of having two flexible arms that allow to capture metal ions easily. The complexes generated by these Schiff bases derived from succinohydrazide have found various applications in the fields of magnetism [15, 16], luminescence [17], optics [18], catalysis [19, 20] and Interactions with biomacromolecules [21]. The use of succinohydrazide as a precursor in the synthesis of Schiff bases having two symmetrical arms has allowed us, recently, to prepare dinuclear complexes of transition metals in which the ligand acts in its iminol form [22] as observed in tin complexes [23]. Continuing our work we used the Schiff base N^1, N^4 -bis(2-hydroxybenzylidene)succinohydrazide (H_4L) to prepare new transition metal complexes which we characterized using FT-IR, UV-Visible spectroscopies, conductance, and magnetic susceptibility measurements.

II. Material And Methods

Starting materials and Instrumentations

Salicylaldehyde, succinohydrazide, manganese chloride tetrahydrate, cobalt chloride hexahydrate, nickel chloride hexahydrate, copper chloride dihydrate, and zinc chloride were commercial products (from Aldrich) and were used without further purifications. The solvents were reagent grade and were purified by usual methods. Elemental analyses were carried out using a VxRio EL Instrument. The FTIR spectra were recorded on

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a FTIR Spectrum Two of Perkin Elmer (4000–400 cm^{-1}). The UV–Vis spectra were run on a Perkin-Elmer UV/Visible spectrophotometer Lambda 365 (1000–200 nm). The ^1H and ^{13}C NMR spectra of the Schiff base were recorded in DMSO-d_6 on a BRUKER 500 MHz spectrometer at room temperature using TMS as an internal reference. The molar conductance of 10^{-3} M solutions of the metal complexes in DMF were measured at 25 $^\circ\text{C}$ using a WTW LF-330 conductivity meter with a WTW conductivity cell. Magnetic measurements for complexes were performed at room temperature by using a Johnson Matthey scientific magnetic susceptibility balance (Calibrant: $\text{Hg}[\text{Co}(\text{SCN})_4]$).

Synthesis of the ligand H_2L

In a 250 mL round-bottomed flask containing 30 mL of methanol, 2 g (13.7 mmol) of succinohydrazide were added and the mixture was stirred for 30 min before adding 3.34 g (27.37 mmol) of salicylaldehyde. The mixture was refluxed for three hours. After cooling, the white solid was recovered by filtration and washed with ether (2 x 20 mL), then dried in the open air. The compound is soluble DMSO. M.P. 247 $^\circ\text{C}$. Yield : 82%. IR (ν , cm^{-1}) : 3453 (OH), 3180 (NH) ; 3054 ($\text{C}_{\text{sp}^2}\text{-H}$), 2981 ($\text{C}_{\text{sp}^3}\text{-H}$), 1667 (C=O), 1618 (C=N), 1566 cm^{-1} ($\text{C}_{\text{Ar}}=\text{C}_{\text{Ar}}$), 1403 cm^{-1} (δ_{CH_2}) 1275 cm^{-1} (C-O) ; 1072 cm^{-1} (N-N). RMN^1H (DMSO-d_6 , δ (ppm)): 2.58 (s, 4H, $-\text{CH}_2$), 6.87-7.50 (m, 8H, $-\text{H}_{\text{Ar}}$), 8.33 (s, 2H, $\text{HC}=\text{N}$), 10.15 (s, 2H, $-\text{NH}$); 11.18 (s, 2H, $-\text{OH}$). RMN^{13}C (DMSO-d_6 , δ (ppm)) : 173.05 (C=O), 157.23 ($\text{C}_{\text{Ar}}\text{-OH}$); 146.30 ($-\text{CH}=\text{N}$), 132.46 (C_{Ar}), 131.13 (C_{Ar}), 119.23 (C_{Ar}), 118.56 (C_{Ar}), 116.06 (C_{Ar}), 34.3 ($-\text{CH}_2-$).

Synthesis of the complexes of H_4L

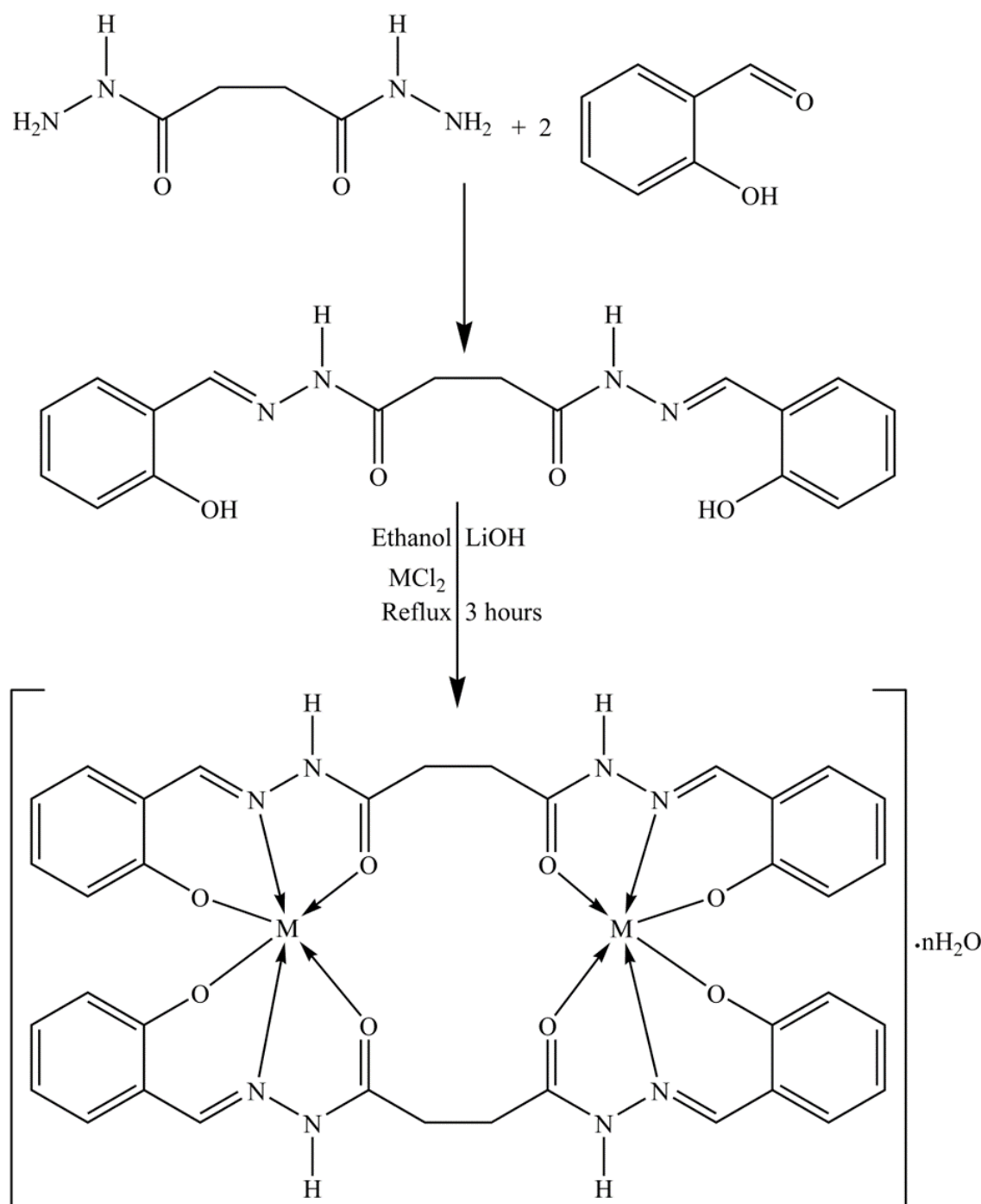
0.1172 g (0.5 mmol) of the ligand were introduced into a flask containing 20 mL of ethanol and 0.4195 g (1 mmol) of $\text{LiOH}\cdot\text{H}_2\text{O}$. A 10 mL ethanol solution containing 0.5 mmol of transition metal chloride salt ($\text{M} = \text{Mn, Fe, Co, Ni, Cu}$) was added to the mixture. The mixture was refluxed for three hours and a change in color was observed. After cooling, the solid which appears was recovered by filtration and then washed with cold ethanol and diethyl ether before dried in desiccator under P_2O_5 . (Table 1).

Table 1. Analytical data, room temperature magnetic moments and conductance of complexes **1-5**.

Compounds	Yield (%)	Color	MP ($^\circ\text{C}$)	% C Calc. (Found)	% H Calc. (Found)	% N Calc. (Found)	Λ ($\Omega^{-1}\cdot\text{cm}^2\cdot\text{mol}^{-1}$)	μ_{eff} (μ_{B})
H_2L	72	White	215	61.01 (59.98)	5.12 (5.09)	15.78 (15.75)	-	-
$[(\text{Mn}_2(\text{H}_2\text{L})_2)\cdot 2\text{H}_2\text{O}$ (1) $\text{C}_{36}\text{H}_{36}\text{Mn}_2\text{N}_8\text{O}_{10}$	41	Pink	> 260	50.83 (50.80)	4.27 (4.23)	13.17 (13.12)	13	11.87
$[(\text{Fe}_2(\text{H}_2\text{L})_2)\cdot 2\text{H}_2\text{O}$ (2) $\text{C}_{36}\text{H}_{40}\text{Fe}_2\text{N}_{12}\text{O}_6$	36	Red	> 260	50.73 (50.70)	4.26 (4.22)	13.15 (13.10)	9	10.55
$[(\text{Co}_2(\text{H}_2\text{L})_2)\cdot 2\text{H}_2\text{O}$ (3) $\text{C}_{36}\text{H}_{36}\text{Co}_2\text{N}_8\text{O}_{10}$	40	Red	> 260	50.36 (50.33)	4.23 (4.20)	13.05 (13.01)	21	9.71
$[(\text{Ni}_2(\text{H}_2\text{L})_2)\cdot 4\text{H}_2\text{O}$ (4) $\text{C}_{36}\text{H}_{40}\text{Ni}_2\text{N}_8\text{O}_{12}$	22	Green	> 260	48.36 (48.32)	4.51 (4.48)	12.53 (12.53)	15	5.78
$[(\text{Cu}_2(\text{H}_2\text{L})_2)\cdot 5\text{H}_2\text{O}$ (5) $\text{C}_{36}\text{H}_{42}\text{Cu}_2\text{N}_8\text{O}_{13}$	39	Green	> 260	46.90 (46.87)	4.59 (4.56)	12.6 (12.13)	13	3.60

Table 2. Main FTIR and UV-visible bands for H_4L and complexes **1-5**.

Compounds	$\nu(\text{O-H})$	$\nu(\text{N-H})$	$\nu(\text{C=O})$	$\nu(\text{C=N})$	$\nu(\text{C-O})$	$\nu_s(\text{N-N})$	$\lambda(\text{nm})$
H_4L	3453	3180	1667	1618	1275	1072	215, 265, 302
(1)	3427	3192	1716	1602	1245	1052	213, 260, 297, 349, 411, 652
(2)	3429	3180	1727	1601	1285	1065	208, 259, 301, 345, 400, 780, 985
(3)	3429	3199	1745	1597	1290	1073	207, 267, 299, 350, 417, 480, 915
(4)	3445	3200	1722	1620	1295	1043	213, 260, 300, 344, 428, 552, 821
(5)	3428	3205	1725	1644	1195	1039	218, 272, 303, 341, 425, 468



M = Mn (n = 2), Fe (n = 2), Co (n = 2), Ni (n = 4), Cu (n = 5)

Scheme 1. Synthetic scheme for the ligand and its metal transition complexes preparation.

III. Results And Discussion

General study

The elemental analysis of the ligand and the complexes which are presented in Table 1 are in accordance with the proposed formula. The infrared spectrum of the ligand shows a broad band at 3453 cm^{-1} which is attributed to the elongation vibration of the OH groups. A medium intensity band at 3180 cm^{-1} is attributed to the elongation vibration of the NH bond of the amide group. The characteristic band of the C=O function is noted at 16657 cm^{-1} . The band appearing at 1618 cm^{-1} corresponds to the elongation vibration of the C=N moiety. The bands in the region $1566\text{ cm}^{-1} - 1450\text{ cm}^{-1}$ are due to the C=C vibration of the benzene ring. The band due to the C-O is pointed

at 1275 cm^{-1} . The ^1H NMR spectrum shows a signal at 2.58 ppm which is attributed to $-\text{CH}_2-$ groups. Between 6.87 ppm and 7.80 ppm, the observed signals are assigned to the aromatic protons. The signals at 10.15 ppm and 11.7 ppm corresponds, respectively, to the HN- and the phenol protons OH. The signal of the azomethine protons appear at 8.33 ppm. In ^{13}C NMR the signal of the methylene groups appears at 34.3 ppm. The carbon atoms of C=O and C=N moieties are identified at 173.5 ppm and 146 ppm, respectively. The signal at 157.23 ppm is attributed to aromatic carbon which support the hydroxy group. The signals which appears in the region 116 ppm - 146 ppm are attributed to the aromatic carbon atoms. In the IR spectra of the complexes, phenolic oxygen is coordinated to copper by loss of the $-\text{OH}$ proton. The $\nu_{\text{O-H}}$ band (3453 cm^{-1} for H_4L) of the free ligand disappear on complex formation which supports coordination through deprotonated phenolate oxygen. The broad bands centered in the range $3180\text{-}3205\text{ cm}^{-1}$ are probably due to the stretching of $\nu_{\text{N-H}}$ [24] bands which appear in the range $3427\text{-}3345\text{ cm}^{-1}$ are due to the presence of free water molecules in the complexes. The bands due to the C=O were shifted to high frequencies [$1716\text{-}1727\text{ cm}^{-1}$] indicating the involvement of the oxygen atoms of the carbonyl groups in the coordination. The shift of the $\nu_{\text{C=N}}$ in the spectrum of the complexes is indicative of the coordination of the ligand through the azomethine atoms of the ligand. The UV-Vis absorption spectra of H_4L is recorded in DMF solutions ($10^{-5}\text{ mol}\cdot\text{L}^{-1}$) at room temperature (Table 2). Three absorption bands are pointed at 215 nm, 265 nm and 302 nm in the UV-Vis spectrum of H_4L . These absorption are due to the $\pi-\pi^*$ transitions of the phenyl rings and the C=N bonds of intra-ligand [25], respectively.

Molar conductance

The molar conductivity of the complexes **1-5**, which are in the range $22\text{-}42\text{ S}\cdot\text{cm}^2\cdot\text{mol}^{-1}$ are indicative of neutral complexes electrolyte in DMF solution [26]. Conductivity values are quite constant after 15 days. This fact shows that the complexes are stable in DMF solutions.

Magnetism

The value of the magnetic moment at room temperature of the manganese complex of 11.87 B.M. is consistent with the presence of two high-spin Mn(II) ions in octahedral environment [27]. The iron(II) complex has a magnetic moment value of 10.55 B.M. which is indicative of the presence of two high-spin uncoupled high-spin iron(II) in the molecule complex [27, 28]. The cobalt complex present a magnetic moment value of 9.71 B.M. This value is in accordance with the presence of two uncoupled cobalt(II) in octahedral environment [29]. The nickel complex is a dinuclear complex with a magnetic moment value of 5.78 B.M. which indicates that the two nickel(II) ions are in octahedral environment [27]. The copper complex present a magnetic moment value of 3.80 B.M. which is indicative of the presence of two copper(II) ions in octahedral environment, without magnetic exchange [30].

Electronic spectra

The electronic spectra data of the complexes **1-5** are recorded in DMF ($10^{-5}\text{ mol}\cdot\text{L}^{-1}$) solutions and listed in Table 2. Three absorption bands pointed at 215 nm, 265 nm and 302 nm in UV-Visible spectrum are due to the $\pi-\pi^*$ transitions of the phenyl rings and the C=N bonds of intra-ligand (**ref**). These absorption peaks are slightly shifted upon coordination of the ligand to the metals ions and are pointed in the spectra of the complexes in the range [207-218 nm], [250-272nm] and [297-303 nm]. The fact indicates clearly the coordination of the ligand to the metals ions. For the complexes **1-5**, the new bands, pointed in the range [341-350 nm] are assigned to the $\pi\rightarrow\pi^*$ transitions of the azomethine C=N moiety. Moreover, there is a weak absorption band at around 411 nm for complex **1**, 400 nm for complex **2**, 417 nm for complex **3**, 428 nm for complex **4** and at 425 nm for complex **5**, which may be ascribed to MLCT transitions. For the manganese complex (**1**) the band pointed at 652 nm is attributable to the $^6\text{A}_{1g} \rightarrow ^4\text{T}_{1g}$ transition which is characteristic of a Mn(II) ion in a distorted octahedral environment [31]. The electronic spectrum of the Fe(II) complex shows two additional low intensity bands pointed at 985 nm and 780 nm. These band are similar to those reported for octahedral iron(II) complex with Jahn-Teller effect [32, 33]. The spectrum of the Co(II) complex shows two absorption bands, pointed at 915 nm and 480 nm attributed to $^4\text{T}_{1g}(\text{F}) \rightarrow ^4\text{T}_{2g}(\text{F})$ and $^4\text{T}_{1g}(\text{F}) \rightarrow ^4\text{T}_{1g}(\text{P})$ transitions, respectively. These values are in the range reported for octahedral Co(II) complex [34]. The electronic spectrum of the Ni(II) complex exhibits two d \rightarrow d bands pointed at 821 nm and 552 nm. These bands are assigned, respectively, to the $^3\text{A}_{2g}\rightarrow^2\text{T}_{2g}$ and $^3\text{A}_{2g}\rightarrow^3\text{T}_{1g}$ transitions which are characteristic of a Ni(II) in an octahedral environment [35]. An additional low intensity bands pointed at 468 nm is exhibited in the spectrum of the Cu(II) complex. This band is assigned to $^2\text{B}_{1g} \rightarrow ^2\text{B}_{2g}$ transition, indicating that the Cu(II) ion is situated in a distorted octahedral environment [36].

IV. Conclusion

In the present work, we report the synthesis and characterization of five new complexes of manganese(II), iron(II), cobalt(II), nickel(II) and copper(II) prepared from the tetradentate ligand *N*'1,*N*'4-bis(2-

hydroxybenzylidene)succinohydrazide (H_4L). The ligand was characterized by elemental analysis, 1H and ^{13}N NMR, FTIR and UV-visible spectroscopies. The complexes **1-5** were characterized by elemental analysis, FTIR and UV-Visible spectroscopies, conductance measurement and room temperature magnetic moment determination. All the complexes are dinuclear and neutral electrolyte in DMF solution. The FTIR spectral data showed that the ligand is coordinated with the metal center through the imino nitrogen atoms, the phenolate oxygen atoms and the carbonyl oxygen atoms. The electronic spectra show bands due to the ligand and the MLCT for all complexes. The d-d bands for complexes **1-5** are in accordance with octahedral structure. Room temperature magnetic susceptibility measurement indicated that the complexes **1-5** are diamagnetic and present two non-coupled metal ions. Conductance measurement shows that the complexes **1-6** are non-electrolyte in nature. The structures of the five complexes are similar and formulated as $[(Mn_2(H_2L)_2) \cdot 2H_2O$ (**1**), $[(Fe_2(H_2L)_2) \cdot 2H_2O$ (**2**), $[(Co_2(H_2L)_2) \cdot 2H_2O$ (**3**), $[(Ni_2(H_2L)_2) \cdot 4H_2O$ (**4**), $[(Cu_2(H_2L)_2) \cdot 5H_2O$ (**5**). In the complexes each of the two symmetrical ligand acts as a bridge between two metal ions.

In Memoriam

Cheikh Halidou KANE 1963–2021. The death of Dr. KANE has deeply shocked us. Dr. KANE was a very talented chemist who was deeply involved in research and supervision of doctoral students. His contribution is greatly missed by our team. This article is dedicated to his memory.

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