Synthesis, Characterization and Antimicrobial studies of some complexes with new Schiff base namely [2,2’-(1E,1’Z)-(1,2-phenylenebis(aza$$\text{n-1-yl-1-yl}$de)) bis (phenylmethan-1-yl-1-ylidene) dibenzoic acid]

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ABSTRACT: A new tetradentate schiff-base derived from 2-benzoxy benzoic acid with O-phenylene diamine have namely[2,2’-(1E,1’Z)-(1,2-phenylenebis(aza$$\text{n-1-yl-1-yl}$de))bis(phenylmethan -1-yl-1-ylidene)dbibensaoic acid]. Tetradental Schiff base type (NNOO) have been synthesized and characterized using IR, UV, 'H & 13C-NMR spectroscopy, elemental analysis. The ligand used in the preparation of five transition metal complexes with Co(II), Ni(II), Mn(II), Hg(II) &Cu(II) and characterized via elemental analysis, FT-IR, UV, magnetic susceptibility and molar conductivity from the above data the geometrical formula was the square planar has suggested for Cu, Ni and Hg complexes and the tetrahedral geometry has suggested for Cu and Mm complexes and in vitro tests for antibacterial activity showed that most of the prepared compounds display a good activity to (Staphylococcus aureus), (Escherichia coli), (Bacillus ) and (Pseudomonas).

KEYWORDS: Tetradentate Schiff base, 2-Benzoyl benzoic acid, O-Phenylene diamine and Antimicrobial studies.

I INTRODUCTION

The coordination chemistry tis are a great area of intense study of inorganic chemistry$^{[1-4]}$. The aspect of interest in ligands is raised from features such as the nature, number and arrangement of ligand donors, as well as ligand conjugation, substitution and flexibility, which produce different types of macrocyclic molecules suitable for specific uses$^{[5]}$. Recently, there was great interest in the field of chemistry of metal complexes of Schiff bases containing O and N donors$^{[6-11]}$. This can be attributed to increased activity in this field to their remarkable structural features antimicrobial activity of these tis donor complexes derived from them$^{[12,13]}$. The ligands chelating contain O and N donor atoms show broad biological activity and are particularly important in view of the variety of ways that are bonded to metal ions. It is known that the presence of bonded metal ions of biologically active compounds may lead to strengthen its activities$^{[14]}$. A wide range of Schiff base macro cycles has evolved from the early studies$^{[15-20]}$, over the last few years working extensive research in many laboratories all over the world, and macrocyclic chemistry is well-established and highly recognized branch of science and thus it provided Schiff bases foundation stone for the building of modern chemistry macrocyclic$^{[21]}$.

The role of the metal ion in these metal-ion tern plated cyclizations is to control the supra molecular assembly of pre-cyclization fragments, most likely through the formation of metal complexes derived from the precursors. Then the results desired product cyclization of intramolecular interaction in the transition state. It is worth mentioning that the Schiff base metal complexes are coordination organometallic compounds and are excellent for building supra molecular complexes because of powerful applications in the field of catalysis, magnetic materials, chemistry, bio-inorganic$^{[22-29]}$. The Schiff base obtained has been versatile in forming a series of complexes with transition metal ions under well defined conditions and these complexes have been investigated with particular reference to the structural aspects of the ligand moiety in the metal complexes.

Transition metal complexes of tetradeative Schiff base ligands find applications in catalysis$^{[26-28]}$ and as bio-mimetic enzyme models,

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Although a large number of compounds of the type M(N$_2$O$_2$) $^{[30-34]}$ (using the binding atom representation), have been synthesized and characterized there are scarce reports on the N$_2$O$_2$ tetra dentate Schiff base complexes of Manganese(III) $^{[35]}$. 

http://www.ijser.org
In this paper, we describe the preparation and characterization of ligand namely [2,2‘-(1E,1’Z)-(1,2-phenylenbis(azan-1-yl-1- ylidene)) bis (phenylmethan-1-yl-1-ylidene) dibenzoic acid] by condensation of 2-Benzoyl benzoic acid with O-Phenylene diamine (2:1) and its complexes with Mn(II),Co(II), Ni(II), Cu(II) and Hg(II) were prepared. Also the antimicrobial studies of the Schiff base and the complexes were done by disc diffusion method.

II EXPERIMENTAL

2.1 Materials and Methods: The reagents 2-benzoylbenzoic acid and O-phenylenediamine were Merck products and used as supplied. Commercial solvents were distilled and then used for the preparation of ligand and its complexes. Other solvents were dried and purified before being used. The complexes were formed by using metal salts: Cobalt(II) chloride hexahydrate; Nickel(II) chloride hexahydrate; Copper (II) chloride dihydrate; Mercury (II) chloride and Manganese (II) chloride tetrahydrate were obtained from (BDH) British Drug House chemical limited company.

2.2 Instrumentation: Products were examined by FT-IR spectra were recorded on SHIMADZU FTIR–8400 Fourier Transform Infrared spectrophotometer as KBr disc. 1HNMR and 13C NMR spectra were recorded on Bruker spectrospin ultra shield magnets 300 MHz instrument. Conductivity measurements were obtained from WTW conductivity meter by using ethanol as a solvent. Melting points were determined in Gallen Kamp melting point apparatus and were uncorrected. Antibacterial screening was done at central laboratory in biological department, college of science, university of Baghdad, using agar diffusion technique.

III Synthesis

3.1 Synthesis of Schiff base Ligand
The ligand was prepared by mixing an ethanolic solution of O-Phenylene diamine with ethanolic solution of respective 2-Benzoyl benzoic acid in a 1:2 molar ratio. To this solution added three drops from glacial acetic acid and the resulting mixture was heated at (80°C) refluxed with stirring for 7 hrs. The resulting solution was evaporation to half volume and precipitated product was collected by filtered off, purified by recrystallization from a hot mixture of [(5mL) acetone, (5mL) ethanol and (2mL) distilled water], wished with dry diethyl ether, and dried at room temperature. The reagents 2-benzoylbenzoic acid and O-phenylenediamine were Merck products and used as supplied. Commercial solvents were distilled and then used for the preparation of ligand and its complexes. Other solvents were dried and purified before being used. The complexes were formed by using metal salts: Cobalt(II) chloride hexahydrate; Nickel(II) chloride hexahydrate; Copper (II) chloride dihydrate; Mercury (II) chloride and Manganese (II) chloride tetrahydrate were obtained from (BDH) British Drug House chemical limited company.

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Spectroscopic and Analytical Data for Schiff Base Compounds

For (C₄H₄N₂O₄) \text{Mwt: (524.17)}, \text{Coulor: (brown)} \text{ Yield(%): 72 ; M.p.: 211°C, Anal. Cal. (%):} C, 68.70; H, 4.58; N, 5.34; O, 12.21 Found (%): C, 67.98; H, 4.02; N, 4.82; O, 11.75

IR (KBr, cm⁻¹): 3167-2532,(O─H)ₐₘ; 3010,(C─H)ₕ; 2941,(C─H)ₕ; 1653, (C=N)ₐ; 1624, (C=C)ₐ; 1700,(COO⁻)ₐ.

UV-Vis (DMSO, \lambda_{max} (nm) (cm⁻²)): 296 (33783).

¹H NMR (300MHZ, δ): DMSO(s, 2.48), =CHₐₚ; COOH(s, 12.48).

¹³C NMR spectra: DMSO(s,40.01), =Cₐₚ (m, 121.96-136.64), COOH(s,169.66), C=N(s,180.46).

For [Co(C₄H₄N₂O₄)Cl₂ \text{Mwt: (621.5)}, \text{Coulor: (pale green)} \text{ Yield(%): 79 , M.p.: 245°C, Anal. Cal. (%):} C, 69.49; H, 65.65; H, 3.54; N, 4.50; O, 10.30 Found (%): C, 67.98; H, 4.02; N, 4.82; O, 11.75

IR (KBr, cm⁻¹): 3012,(C─H)ₕ; 2978,(C─H)ₕ; 1658, (C=N)ₐ; 1625, (C=C)ₐ; 1535 & 1458 (COO⁻)ₐ; 548, (M—N)ₐ; 421, (M—O)ₐ.

UV-Vis (DMSO, \lambda_{max} (nm) (cm⁻²)): 272 (36764), 463 (21598), 630 (18573).

Molar conductance (DMSO): 73.7 S. mol⁻¹. cm², \mu_{off} (BM): 3.9.

For[Cu(C₄H₄N₂O₄)Cl₂ \text{Mwt: (621.5)}, \text{Coulor: (reddish brown)}, \text{Yield(%):72 , M.p.: 233°C, Anal. Cal. (%):} Cu,10.22; C, 65.65; H, 3.54; N, 4.51; O, 10.30
Molar conductance: Through the two oxygen atoms of the carboxylic acid groups and two nitrogen atoms of C=N azomethine to give square planar and tetrahedral geometry has suggested for Co and Mn complexes.

IR (KBr, cm⁻¹): 3037, (C=H) aromatic; 2995, (C=H) aliphatic; 1662, (C=N)ₜ; 1620, (C=C)ₜ; 1537 & 1440 (COO)ₜ; 553, (M─N)ₜ; 455, (M─O)ₜ.
UV-Vis (DMSO, λmax (nm) (cm⁻¹)): 275 (36363), 377 (26525), 498 (20080).
Molar conductance (DMSO): 70.42 S. mol⁻¹. cm², μ_eff (BM): 1.76.

For [Mn(C₃H₂₂N₂O₄)]Cl₂: Mwt: (613.17), Coulor: (pale brown), Yield(%): 80, M.p.: 250°C
Anal. Cal. (%): Mn, 8.97; C, 66.56; H, 3.59; N, 4.57; O, 10.44.
Found (%): Mn, 8.06; C, 65.98; H, 3.12; N, 4.02; O, 10.09.

IR (KBr, cm⁻¹): 3008, (C=H) aromatic; 2981, (C=H) aliphatic; 1668, (C=N)ₜ; 1625, (C=C)ₜ; 1535 & 1429 (COO)ₜ; 559, (M─N)ₜ; 441, (M─O)ₜ.
UV-Vis (DMSO, λmax (nm) (cm⁻¹)): 276 (36231), 412 (24271), 559 (17889), 412 nm.
Molar conductance (DMSO): 78.21 S. mol⁻¹. cm², μ_eff (BM): 5.50.

For [Hg(C₃H₂₂N₂O₄)]Cl₂: Mwt: (758.6), Coulor: (purple), Yield(%): 72, M.p.: 243°C
Anal. Cal. (%): Hg, 26.44; C, 53.78; H, 2.90; N, 3.69; O, 8.44.
Found (%): Hg, 25.94; C, 52.98; H, 2.92; N, 3.02; O, 7.75.

IR (KBr, cm⁻¹): 3022, (C=H) aromatic; 2972, (C=H) aliphatic; 1663, (C=N)ₜ; 1624, (C=C)ₜ; 1535 & 1445 (COO)ₜ; 558, (M─N)ₜ; 426, (M─O)ₜ.
UV-Vis (DMSO, λmax (nm) (cm⁻¹)): 274 (36496), 458 (21834).
Molar conductance (DMSO): 82.45 S. mol⁻¹. cm², μ_eff (BM): diam.

For [Ni(C₃H₂₂N₂O₄)]Cl₂: Mwt: (617.17), Coulor: (purple), Yield(%): 78, M.p.: dec.< 260°C,
Anal. Cal. (%): Ni, 9.56; C, 66.13; H, 3.57; N, 4.54; O, 10.37.
Found (%): Ni, 9.18; C, 65.76; H, 3.21; N, 4.21; O, 9.89.

IR (KBr, cm⁻¹): 3030, (C=H) aromatic; 2999, (C=H) aliphatic; 1667, (C=N)ₜ; 1624, (C=C)ₜ; 1537 & 1449 (COO)ₜ; 557, (M─N)ₜ; 428, (M─O)ₜ.
UV-Vis (DMSO, λmax (nm) (cm⁻¹)): 260 (38461), 443 (22573).
Molar conductance (DMSO): 80.12 S. mol⁻¹. cm², μ_eff (BM): diam.

IV Results and Discussion

Elemental Analysis: The compositional data of [2,2′-(1E,1′Z)-(1,2-phenylenebis(azan-1-yl)-1-yli dine)) bis (phenylmethan-1-yl-1-ylidene) dibenzoic acid] compound is in good agreement with it's suggested stoichiometries. The analytical data of the complexes correspond well with the general formula ML where M = Cu(II), Ni(II), Co(II), Mn(II) and Hg(II) Mn(II); L = C₃H₂₂N₂O₄. The formulation of the complexes was based on IR data, molar conductivity values, elemental analyses, which were in close agreement with the values calculated for the assigned molecular formulae. The elemental analytical data were in good agreement with the molecular formulae arrived for the ligand and its complexes. The elemental analyses agreed well with 1:1 metal to ligand stoichiometry for all the complexes. The complexes are of various colours varied colour different from the colour of the ligand indicating that the colours formed depend on the metal ions. The melting points of the complexes are different (higher) than that of the Schiff base ligand, an evidence for complexation. All complexes are soluble in common organic solvents such as chloroform, acetone, water, dimethylsulfoxamide and dimethylformamide. In all the complexes [2,2′-(1E,1′Z)-(1,2-phenylenebis(azan-1-yl-1-yli dine)) bis (phenylmethan-1-yl-1-ylidene) dibenzoic acid] acts as a tetradeinate ligand and binds through the two oxygen atoms of the carboxylic acid groups and two nitrogen atoms of C=N azomethines to give square planar and tetrahedral geometries.

Molar conductance: One of the measurements commonly employed in the determination of charge type of the complexes is to determine the equivalent conductance at infinite dilution of those complexes. The chelates complexes prepared in the work showed conductivity values ranged between (70.42–82.45 S. mol⁻¹. cm² ) in DMSO at room temperature. These values indicating that conductive species exist[27]. According to these results the structural formulas of these ligand and complexes. Thus the suxare planar geometry has suggested for Cu, Ni and Hg complexes and the tetrahedral geometry has suggested for Co and Mn complexes[18-20].

NMR spectrum for the ligand (L): ¹H NMR spectrum of H₂L in DMSO–d₆ solution shows the following signals: C₆H₆ as multiplet at δₖ 6.02–7.40. The peak observed at δₖ 12.48 is attributable to the carboxylic (COOH) group present of the Schiff base Fig(3).

¹³C–NMR of the free ligand(H₂L) Fig(4) shows the HC=N peak at (180.46 ppm), the COOH peak at (159.83 )ppm, and carbon peaks for aromatic are detected at (121.96 ~136.64) ppm. The peak at (40.01) ppm. assigned to DMSO[7-8].
IR spectra: In order to study the binding mode of the Schiff base to the metal ion of the complexes, the IR spectrum of the free ligand was compared with the spectra of the complexes. The IR spectrum of the ligand shows a weak broad band in the region 3167–2532 cm⁻¹ assignable to hydrogen bonding for acidic -OH group[15–17]. Absence of this band in complexes indicates the deprotonation of the hydrogen bonded acidic -OH group upon complexation. The ligand shows its characteristic =C=N- band at ca. 1653 cm⁻¹, which are also shifted to higher frequencies in the spectra of the complexes (1667–1658 cm⁻¹). The metal chelates show some new bands in the region 559–548 cm⁻¹ and 428–420 cm⁻¹ which are due to the formation of M-O and M-N bonds respectively[18–21]. Hence, it is concluded that the coordination to the metal ion occurs through the enolizable acidic group of 2-benzoyl benzoic acid and the azomethine nitrogen atoms of the Schiff base. The free ligand (H₂L) exhibits an absorption bands at (1700)cm⁻¹ due to the stretching vibration of v(C=O) of carboxylic group[22]. This band disappeared in the spectra of the complexes by the appearance of one of two bands in the (1537–1535)cm⁻¹ range due to vₐₚₚ(COO⁻) and another bands in the (1458–1429)cm⁻¹ assigned to vₐₚₚ(COO), Δν = (106–77) cm⁻¹[23]. The values of (Δν) for all complexes refer to the carboxylate group behaves as tetradentate group. In addition to other bands, shows in IR spectra for the ligand and its complexes[27].

The electronic absorption spectra and magnetic studies: The electronic absorption spectra of the Schiff base and its Cu(II), Ni(II), Co(II), Hg(II) and Mn(II) complexes were recorded at 300 K. The UV-Vis spectrum of the Schiff base ligand is characterized mainly by two absorption bands at 296 nm (33783 cm⁻¹), which may be assigned to n → π* transition. This transition was also found in the spectra of the complexes, but they were shifted towards lower in range (244 - 275) nm[13].

i) Cobalt(II) complex shows magnetic moment (3.90 B.M) corresponding to three unpaired electrons. The electronic spectrum of the cobalt(II) complex exhibits absorption at 463nm (15314 cm⁻¹) and 630 nm (15873 cm⁻¹). These bands may be assigned to the transitions: \( ^4A_{2g} \rightarrow ^4T_{1g} \) and \( ^4A_{2g} \rightarrow ^4T_{2g} \), respectively. The position of these bands suggests a tetrahedral environment around the Co(II) ion.[12]

ii) Copper(II) complex has magnetic moment value (1.76 B.M) which may suggest square planar structure. Its electronic spectrum shows band centered at 377nm (26525 cm⁻¹) and 498nm (20080 cm⁻¹) assigned to \( ^4B_{1g} \rightarrow ^4E_g \) and \( ^4B_{1g} \rightarrow ^4A_{2g} \) transitions in square planar[18].

iii) Manganese(II) complex shows magnetic moment (5.50 B.M) at room temperature corresponding to one unpaired electrons. The electronic spectrum exhibits two absorption bands at 412 nm (24271 cm⁻¹) and 559 nm (17889 cm⁻¹). These bands may be assigned to the transitions: \( ^4A_1 \rightarrow ^4T_2 \) (G) and \( ^4A_1 \rightarrow ^4E \) (G) transitions respectively. Position of these bands indicates a tetrahedral geometry around the Mn(II) ion[21].

iv) Mercury(II) complex is diamagnetic moments for d¹⁰ ion and the electronic spectra there complex do not show any d-d band[25]. The complex of Hg(II) is diamagnetic. According to the empirical formula, square planar geometry is proposed for this complex.

v) Nickel(II) complex shows magnetic moment (3.12 B.M) at room temperature corresponding to two unpaired electrons. The electronic spectrum exhibits two absorptions at 508 nm (19685 cm⁻¹) and 557nm (19685 cm⁻¹). These bands may be assigned: \( ^4A_{1g} \rightarrow ^4A_{2g} \) and \( ^4A_{1g} \rightarrow ^4B_{1g} \) transitions[23]. Position of these band indicates square planar geometry around the Ni(II) ion[27].

Biological activities: The ligand driven by the condensation of 2-benzoyl benzoic acid and o-phenylene diamine and it’s Co(II), Mn(II), Cu(II) and Hg(II) and Ni complexes showed difference between the antimicrobial activity of ligand and metal complexes in Fig(6). The well diffusion method was used. Nutrient agar (NA) was used as medium for the cultured bacteria. The sample solutions (10⁻² M) were prepared by dissolving the compound in water. The sample solutions were loaded on the wells of the culture and incubated at 37°C for 24 h for the bacterial culture. During the period, the test solution diffused and the growth of the inoculated microorganisms was affected. Antibacterial activity was evaluated by measuring the zone of inhibition. The zone of inhibition was calculated in centimeters.

V Conclusion:

The new Schiff ligand (H₂L) and chelate complexes have been prepared [Mn(L)Cl₂], [Co(L)Cl₂], [Ni(L)Cl₂], [Cu(L)Cl₂] and [Hg(L)Cl₂]. All the complexes are stable and ionic. The metal (II) ions are coordinated by two carboxylate-O atoms and two imine(H=C=N) atoms. Spectroscopic, structural and magnetic data show that all complexes are four-coordinate metaes owing to theigation of tetradeinate schiff base moieties shows Fig(5)(a)&(b).

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Scheme(1): Synthetic route of the Schiff base ligand (L)

Scheme(2): Synthetic route of the ligand’s metal complexes

VI Acknowledgement

We are thankful to Department of Chemistry, College of Education for Pur Sciences, Ibn-Al -Haitham, University of Baghdad for providing financial support.

Table (1): Electronic spectral data of the ligand (H₂L) and its metal complexes

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<th>Compound</th>
<th>λnm</th>
<th>υ–wave number cm⁻¹</th>
<th>Assignments</th>
<th>Proposed structure</th>
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<td>H₂L</td>
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<td>45045</td>
<td>π→π*</td>
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<td>[Co(L)]Cl₂</td>
<td>272</td>
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<td>17889</td>
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<tr>
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Table (2) Diameter of zone of inhibition (mm)

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<th>L</th>
<th>Col</th>
<th>CuL</th>
<th>MnL</th>
<th>NiL</th>
<th>HgL</th>
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<td><em>Escherichia. Coli</em>(G-)</td>
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<td>4</td>
<td>2</td>
<td>5</td>
<td>-</td>
<td>6</td>
</tr>
<tr>
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<td>-</td>
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<td>3</td>
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<tr>
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<td>7</td>
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<td>6</td>
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<td>-</td>
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<tr>
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Fig(1) IR spectra of Ligand and it's complexes

Fig(2) UV. Vis spectra of Ligand and it's complexes
Fig.(3)$^1\text{H}$- NMR of Ligand ($\text{H}_2\text{L}$)

Fig.(4)$^{13}\text{C}$- NMR of Ligand ($\text{H}_2\text{L}$)
Fig(5) Molecular structure of (a) [2,2‘-(1E,1’Z)-(1,2-phenylenebis(azan-1-yl-1-ylidene)) bis(phenylmethan-1-yl-1-ylidene) dibenzoic acid] and (b) of the metal chelate complexes

Fig.(6) Difference between the antimicrobial activity of ligand & metal complexes

VII REFERENCES


