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Original Article

Synthesis of 3-hydroxy-4-{[(2E)-2-(hydroxyimino)ethylidene]amino}-naphthalene-1-sulfonic acid for Complex formation of Cr3+, Co2+, Ni2+ and Cu2+ ions

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Abstract

Purpose: This study aimed to synthesize and characterize new Schiff-base complexes derived from 3-hydroxy-4-{[(2E)-2-(hydroxyimino)ethylidene]amino}-naphthalene-1-sulfonic acid and to evaluate their structural properties and antimicrobial activity.**Methods:** The complexes of Cr^{3+}, Co^{2+}, Ni^{2+}, and Cu^{2+} were synthesized and characterized using IR spectroscopy, molar conductivity, UV-Vis spectroscopy, magnetic susceptibility measurements, and elemental analysis (C, H, N, and S). The coordination behavior of the ligand was investigated based on spectral and magnetic data. **Results:** Elemental analysis confirmed a 1:1 metal-to-ligand stoichiometry in all complexes. Molar conductivity values indicated that the complexes are non-electrolytic. Magnetic moment data revealed that the Ni^{2+} complex is diamagnetic, while Co^{2+}, Cr^{3+}, and Cu^{2+} complexes are paramagnetic. IR spectral data suggested coordination through the hydroxyl and imino groups. Electronic spectra showed $n\rightarrow\pi^*$ and $\pi\rightarrow\pi^*$ transitions, supporting octahedral geometry for all complexes. **Conclusion:** The structural analysis confirmed the formation of stable, octahedral Schiff-base metal complexes. These compounds, including the free ligand, demonstrated notable antimicrobial activity against pathogenic bacteria such as Proteus, Bacillus subtilis, Staphylococci, and Pseudomonas aeruginosa.

Keywords: 4-(2-hydroxbenzylideneamino)-3-hydroxnaphtalene-1-sulfonic acid, Pathogenic bacteria, Schiff-base complexes.

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INTRODUCTION:

The condensation reaction between 4-(dimethylamino)benzaldehyde and 4-amino-3-hydroxynaphthalene-1-sulfonic acid produced Co(II) and Cr(III) metal complexes of Schiff bases. Elemental analysis, molar conductance measurements, infrared spectroscopy, electronic spectroscopy, and 1H NMR spectroscopy were used to examine the structures of the materials. The complexes appeared to have a 1:1 [M:L] ratio based on the elemental analysis data. The molar conductance measurements of the complexes revealed that they are electrolytic when DMSO is used as a solvent. The presence of an octahedral environment surrounding the metal ions in the complexes was confirmed by the absorption bands in the electronic spectra [1]. An array of 2-(4-(2-chloroacetyl)piperazin-1-yl)-N-(2-(4-chlorophenyl)-4-oxoquinazolin-3(4H)-yl) acetamide derivatives were created in this work, and their chemical structures were verified through spectral and physicochemical analyses [2]. A new Schiff base and a new series of complexes of Co(II), Ni(II), Cu(II), Cd(II), and Hg(II) were created by condensing diacetylmonoxime and naphthofuran-2-carbohydrazide. The Schiff base metal complexes were synthesized using the ethanolic chloride salts of Co(II), Ni(II), Cu(II), Cd(II), and Hg(II). Based on analytical data, IR, electronic, mass, 1H NMR, ESR spectral data, thermal studies, magnetic susceptibility, and molar conductance measurements, the ligand and its metal complexes were characterized. Based on the measured low-conductance data, the non-electrolytic behavior of the complexes was evaluated. The stoichiometries of the CuL_2Cl_2 and $MLCl_2$ catalysts, where M = Ni(II), Co(II), Cd(II), Hg(II), and L = Schiff base, were confirmed by elemental analysis of the complexes. The redox properties of the Cu(II) complex were examined through an electrochemical method employing cyclic voltammetry. These findings led to the assignment of octahedral structures to Co(II), Ni(II), and Cu(II) complexes and of tetrahedral structures to Cd(II), Hg(II), and Ni(II) complexes [3]. We synthesized and characterized Mn(II), Ni(II), and Cu(II) transition metal chelates with Schiff base. The data from the elemental analysis indicated that the ratio of the isolated chelates was 1:1 [M:L]. The non-electric nature of the chelates was demonstrated by the molar conductance values. Magnetic moment measurements revealed that the Ni(II) chelate is diamagnetic and that the chelates of Mn(II) and Cu(II) have unpaired electrons. The primary coordination sites of (2E, 3E)-3-((2-aminophenyl) imino)butan-2-one oxime toward Mn(II), Ni(II), and Cu(II) ions were visible in the infrared spectral data. The electronic spectrum of the Schiff base ligand and its chelates indicates that the Ni(II) chelate is square planar, while the Mn(II) and Cu(II) chelates have octahedral structures [4]. [2-(2-(Naphthalen-5-yl)diazenyl)-4-amino-3- hydroxynaphthalene-1-sulfonic acid] was used as a precursor obtained from the reaction of diazonium salt with 1-amino-2-naphtol-4-sulfonic acid (W) to synthesize the tridentate (ONO)-type ligand [H_2L] by reacting [W] with 1-hydroxynaphthalene-2- carbaldehyde to produce the ligand [H2L] [2-(2-(naphthalen-5-yl) dialzenyl-4-(2- hydroxynaphthalen-3-yl) methyleneamino)-3-hydroxy naphthalene-1-sulfonic acid]. The ligand was characterized by spectroscopic methods. Using the same methodology, previous researchers also prepared complexes by reacting ligands with [CrIII, MnII, and FeII] ion salts under reflux, with ethanol serving as a solvent and KOH serving as a base. These complexes were characterized using atomic absorption (AA), IR, and UV–vis spectroscopy and conductivity measurements of chloride content. Finally, the presence of crystallized H_2O molecules in the complex structure was verified by thermal analysis. $K[M(L)_2]$ is the suggested formula for the Cr complex. H_2O, whereas complexes of Mn and Fe have $K_2[M(L)_2]$. H_2O formula. The biological activities of the prepared compounds were examined [5]. By reacting two distinct amino-substituted compounds with substituted aldehydes in methanol, three new series of biologically active amino-substituted Schiff bases were created. Different physicochemical techniques were used to characterize the products. The ability of the free ligands and their metal complexes to inhibit bacteria, fungi, and yeast in vitro was tested. Using *Aspergillus niger*, the physiological and antifungal activities of the ligand and metal complexes were evaluated. Compared to Schiff base ligands [6], metal complexes exhibit more potent activities. The molar conductance values of the 4-(2-hydroxybenzylidene amino) -3-hydroxnaphthalene-1-sulfonic acid study showed that all of the complexes were nonelectrolytic in nature. Magnetic moment measurements reveal that the Ni^{2+} complex is diamagnetic, whereas the Co^{2+}, Ni^{2+}, and Cu^{2+} complexes are paramagnetic. The primary coordination sites of 4-(2-hydroxbenzylideneamino)-3-hydroxnaphthalene-1-sulfonic acid to Cr^{3+}, Co^{2+}, Ni2+, and Cu2+ ions are suggested by the IR spectral data. The electronic spectra of the ligand and its complexes exhibit n→π* (HC=N) and π→π* (phenyl ring) transitions. Furthermore, the data indicate that the Ni^{2+} complex has a square-planar structure, whereas the Cr^{3+}, Co^{2+}, and Cu^{2+} complexes have octahedral structures.

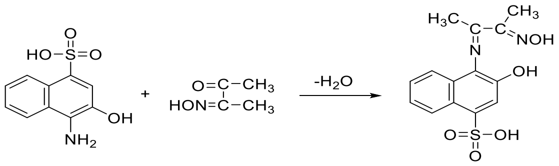
Staphylococci, *Proteus*, *Bacillus subtilis*, and *Pseudomonas aeruginosa* are among the pathogenic bacteria against which the synthesized Schiff base and its complexes were tested [7].

MATERIALS AND METHODS:

All the chemicals used in this investigation were Aldrich or BDH reagents, including diacetyl monoxime, 4-amino-3-hydroxynaphthalene-1-sulfonic acid, ammonium solution, ether, and EtOH.

Synthesis of Schiff base

3-hydroxy-4-{[(2*E*)-2-(hydroxyimino) ethylidene]amino}-naphthalene-1-sulfonic acid (H_2L) was synthesized by adding (2.39 g, 0.01 mmole) 4-amino-3-hydroxy-naphthalene-1-sulfonic acid dropwise to diacetylmmonoxime (1.011 g, 0.01 mmole) in 50 mL of absolute Et-OH. The reaction mixture was refluxed for 3 h. The product was allowed to cool at room temperature, filtered off, and recrystallized from Et-OH and then dried under vacuum to obtain a pale pink precipitate (m.p. 185 °C; yield 79%).



Synthesis of Complexes

The Schiff base complexes under investigation were synthesized by adding 3-hydroxy-4-{[(2*E*)-2-(hydroxyimino) ethylidene]amino}-naphthalene-1-sulfonic acid (H_2L) (3.3.22 g, 0.01 mmole) in 50 mL of absolute Et-OH to 0.05 mmole of the salts $CrCl_3.6H_2O$ (2.66 g, 0.01 mmole), $CoCl_2.6H_2O$ (2.37 g, 0.01 M), $NiCl_2.6H_2O$ (2.37 g, 0.01 M) and $CuSO_4.5H_2O$ (2.49 g, 0.01 M) in the same amount of absolute Et-OH or bidistilled water. The reaction mixture was heated under reflux for 3 h. The complexes were filtered off, recrystallized from the suitable solvent, and finally kept in a desiccator over silica gel.

Bacterial assay

The Schiff base and its complexes with Cr^{3+}, Co^{3+}, Ni^{3+}, and Cu^{2+} were tested against *Pseudomonas aeruginosa* and *Bacillus subtilis* Bacterium species in a mixture of DMF and H_2O. Diffusion methods were used for antibacterial activity determination.

Antibacterial activity

The Schiff base and its complexes inhibited all the bacteria tested (*Pseudomonas aeruginosa* and *Bacillus subtilis* bacteria) because of the free Schiff base 18-13 mm, and its complex 08-16 mm. The antibacterial results (mm) are presented in Table 1. The study of the antibacterial activity of the Schiff bases and their complexes showed that they had greater activity against the bacteria tested [8,9].

Table (1): Antibacterial activity results (mm) of Schiff base and its complexes.

L and its complexes	**Bacteria**	
	Pseudomonas Aeruginosa	*Bacillus Subtilis*
H_2L; ($C_{17}H_{13}NO_5S$)	18	13
$[CrL(Cl)(H_2O)_2].2H_2O$	12	16
$[CoL(H_2O)_3].2H_2O$	08	13
$[NiLH_2O)].3H_2O$	11	08
$[CuL(H_2O)_3].H_2O$	10	10

RESULT:

The synthesis of 3-hydroxy-4-{[(2*E*)-2-(hydroxyimino)ethylidene]amino}-naphthalene-1-sulfonic acid (H_2L) yields only one product (Scheme 1).

Table (2): Microanalysis and molar conductance measurements of the Schiff base L_2H and its complexes.

Compound	%Calc. (Found)				Λ (μs)	
	C%	H%	N%	S%		BM
H_2L ($C_{14}H_{14}N2O_5S$)	52.17(52.42)	4.38(4.58)	8.69(8.92)	9.95(9.46)	-	-
$[Cr(C_{14}H_{17}N_2O_7SCl_2)]\ 3H_2O$	31.47(31.74)	4.34(4.42)	5.24(5.17)	6.01(6.33)	33.45	3.73
$[Co(C_{14}H_{19}N_2O_8SCl)]\ 2H_2O$	33.24(33.77)	4.58(5.27)	5.58(5.22)	6.34(6.39)	17	2.76
$[Ni(C_{14}H_{21}N_2O_9SCl)]\ 4H_2O$	30.05(31.21)	5.22(5.34)	5.01(5.65)	5.73(5.52)	11.32	2.72
$[Cu(C_{14}H_{22}N_2O_{13}S_2)]SO_4$	30.35(31.06)	4.00(4.41)	5.06(5.19)	11.58(11.34)	105	1.67

Microanalysis and molar conductance measurements

The elemental analysis data and some physical properties of the Schiff base and its complexes are summarized in Table 1, where the results confirm the proposed composition. The synthesized complexes were formed in a 1:1 (M:L) ratio. The obtained molar conductance values of the complexes in DMF solvent ranged from 11.32.105 ohm^{-1} cm^{2} mol$^{-1,}$ indicating $[Cr(C_{14}H_{17}N_2O_7SCl_2)]$ $3H_2O$, $[Co(C_{14}H_{19}N_2O_8SCl)]$ $2H_2O$, $[Ni(C_{14}H_{21}N_2O_9SCl)]4H_2O$ non-electrolytic, and $[Cu(C_{14}H_{22}N_2O_{13}S_2)]SO_4$ electrolytic behavior. Thus, the complexes may be formulated as $[M (L)Cl_n(H_2O)_n]$. nH_2O, where M = (Cr^{3+}, Co^{2+}, Ni^{2+}, and Cu^{2+}) [10].

NMR spectra of the Schiff base and its complexes

The ^{1}H-NMR spectrum was recorded in d^6 DMSO as a solvent via a Jeol-90 Fourier transform (200 MHz). H_2L shows peaks at 1.05, 1,884, 2.376, 5.656, and 7.181 – 8.343 ppm downfield of TMS, assignable to the protons of CH_3, CH_3, OH(NOH), OH(phenyl), and 5H (aromatic) and the phenyl ring, respectively [11].

Electronic spectra of H_2L and its complexes

The electronic absorption spectra data of the ligand ($C_{14}H_{14}N_2O_5S$) H_2L and its complexes with Cr(III), Co(II), Ni(II), and Cu(II) are summarized in Table and Figures 3-7. The electronic absorption spectrum of the ligand showed one very intense band at 35715 and 37000 cm^{-1} assigned to the $n\rightarrow\pi^*$ and $\sigma\rightarrow\sigma^*$ transitions, respectively [12].

Electronic (UV–Vis) Spectra

The electronic absorption spectrum of the Cr(III) complex shows a band in the visible region at 18180 cm^{-1} assignable to the $^3T_{2g} \rightarrow {}^3A_{1g}$ transition, which is characteristic of octahedral geometry, and an absorption at 27570 cm^{-1} assigned to M–L charge transfer[13]. The electronic absorption spectrum of the Co(II) complex shows a band in the visible region at 21505 cm^{-1} and one very strong band at 33000 cm$^{-1,}$ which is attributed to ligand-to-metal charge transfer transition. The electronic absorption of the Ni(II) complex exhibited bands at 26980 cm^{-1} and 23300 cm-1, which may be assigned to $^3T_{1(F)} \rightarrow {}^3T_{2(P)}$ [14]. The spectrum also showed an intense, sharp band at 33333 cm$^{-1,}$ which is attributed to ligand-to-metal charge transfer transition, as observed in most tetrahedral complexes. The electronic absorption spectrum of the Cu(II) complex with ligand (L) exhibited bands at 18200 cm^{-1}, 27027 and 34120 cm-1, which may be assigned to $4A_2 \rightarrow 4T_1$ (P), and the other two transitions, 27027 and 34120 cm$^{-1,}$ are attributed to charge transfer transitions [15].

Table. 3: Electronic spectra of H_2L and their synthesized complexes

Ligand/complexes	Electronic spectra
	UV/Vis nm
L2 ($C_{14}H_{14}N_2O_5S$)	270, 280
$[Cr(C_{14}H_{17}N_2O_7SCl_2)$ $3H_2O$	362, 550
$[Co(C_{14}H_{19}N_2O_8SCl)]2H_2O$	303, 464
$[Ni(C_{14}H_{21}N_2O_9SCl)]4H_2O$	3300, 429
$[Cu(C_{14}H_{22}N_2O_{13}S_2)]SO_4$	293, 370, 549
	293, 370, 549

Infrared spectra of the Schiff base H2L and its complexes

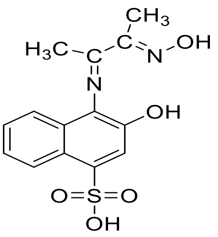
The Schiff base and its complexes' IR spectral data are summarized in Table 4. The IR spectrum has bands in the range of 3507 cm-1, which could be due to the presence of OH groups in the Schiff base [16]. However, the band at 1602 cm^{-1} is attributed to the νC=N group [17]. The Schiff base L2 is characterized by the stretching frequency of νOH of the phenolic group at 3418 cm^{-1}. This group is associated with complex formation, as shown in the IR spectra of all the complexes (Table 4), indicating the involvement of the –OH group in chelation through the oxygen atom. This was confirmed by the existence of new bands in the range of 549 – 593 cm^{-1} assigned to ν M-O vibrations, which are absent in the spectrum of the Schiff base. The band at 1615 cm^{-1} is due to the νC =N group of L2, which is shifted to a lower wave number in the spectra of the complexes, indicating its participation in coordination via nitrogen atoms. This can be supported by the existence of new bands in the range of 611–700 cm$^{-1,}$ which could be assigned to νM-N vibrations [18,19]. L2 in complex with Cr(III), Co(II), Ni(II) and Cu(II) exhibited bands at 3418 cm^{-1}, 3432 cm^{-1}, 3475 cm^{-1} and 3407 cm^{-1}, respectively, corresponding to the ν(OH) stretching vibration. The C=N peak at 1602 cm^{-1} for the free ligand is shifted to 1567 cm^{-1}, 1562 cm^{-1}, 1565 cm^{-1}, and 1569 cm^{-1} for the Cr(III), Co(II), Ni(II) and Cu(II) complexes, respectively. New bands in the complexes appears at 691 cm^{-1}, 632 cm^{-1}, 611 cm^{-1} and 700 cm^{-1}, respectively are assigned to the ν(M –N) and at 595 cm^{-1}, 515 cm^{-1}, 505 cm^{-1} and 549 cm^{-1} are assigned to ν(M –O) vibration , respectively [20-23].

Table 4: Infrared spectral data of the ligand (L2) and its synthesized complexes

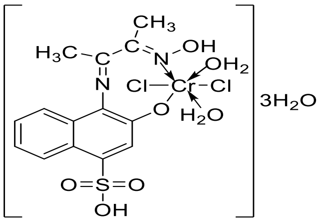
Ligand /Complexes	IR (cm^{-1})			
	OH	ν C=N	νM-N	νM-O
L2 ($C_{14}H_{14}N_2O_5S$)	3507	1602	-	-
[Cr(L)(H2O)$_2$(Cl)$_2$)]3H_2O	3418	1567	691	593
[Co(L)(H_2O)$_3$Cl)]2H_2O	3432	1562	632	515
[Ni(L)(H_2O)$_3$Cl)]4H_2O	3475	1565	611	505
[Cu(L)(H_2O)$_3$]SO_4	3407	1569	700	549

DISCUSSION:

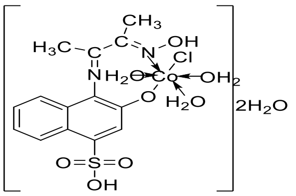
Analytical and spectroscopic results confirmed the formation and structural characteristics of the Schiff base ligand (H_2L) and its metal complexes. The close agreement between the calculated and observed elemental values supported the proposed formulas. Molar conductance studies suggest that the Cu(II) complex is a 1:1 electrolyte, likely due to the presence of the SO_4^{2-} counterion, whereas the other complexes are neutral. The NMR signals confirmed the presence of functional groups involved in coordination, particularly the phenolic and oxime OH groups, aligned with the expected structure of the Schiff base. The observed electronic transitions are consistent with the octahedral coordination of Cr(III), Co(II), and Ni(II) complexes and a potentially distorted octahedral geometry for the Cu(II) complex. The charge-transfer bands further confirmed the metal-ligand interactions. Coordination through both nitrogen (C=N) and oxygen (OH) atoms was confirmed by the shift of the ν(C=N) stretching frequency to lower wavenumbers, disappearance or broadening of the ν(OH) bands, and emergence of ν(M–O) and ν(M–N) vibration bands, which are absent in the free ligand. Collectively, these spectral features confirm that the Schiff base ligand acts as a bidentate or potentially tetradentate ligand, coordinated via the phenolic OH and azomethine nitrogen atoms. The coordination environment around the metal centers supported the proposed geometries of the synthesized complexes.



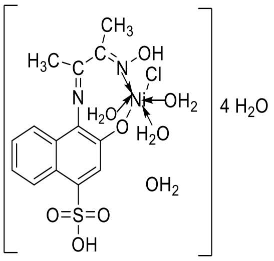
Structure (1) L $C_{14}H_{14}N_2O_5S$



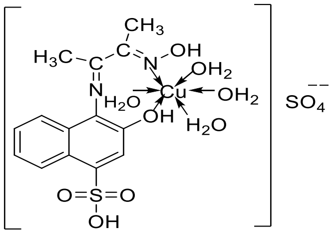
Structure (2)[Cr(L) (Cl)$_2$(H_2O)$_2$]$_3$$H_2O$



Structure (3) [Co(L)(Cl)(H_2O)$_3$]$_2$$H_2O$



Structure (4)[Ni(L)Cl(H_2O)$_3$]$_4$$H_2O$



Structure (5) [Cu(L)(H_2O)$_4$]SO_4

CONCLUSION:

Four Schiff base complexes of Cr(III), Co(II), Ni(II), and Cu(II) with 3-hydroxy-4-{[(*2E*)-2-(hydroxyimino)ethylidene]amino}-naphthalene-1-sulfon*ic* acid were synthesized and characterized, and the yields of the synthesized ligands and their complexes were determined. The synthesized complexes were characterized using several techniques (elemental analysis, mass spectrometry

(MS), IR, UV–Vis, and ^{1}H-NMR). The following geometrical structures (1-5) were suggested for the synthesized complexes because of their octahedral and antibacterial activities.

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