



Original Research Article

Formation, Characterization and Thermal Study of Novel Schiff Base Ligand from Sulfonic Acid and Its Complexes with Co(II), Ni(II), Cu(II), Zn(II) and Hg(II) Type NO

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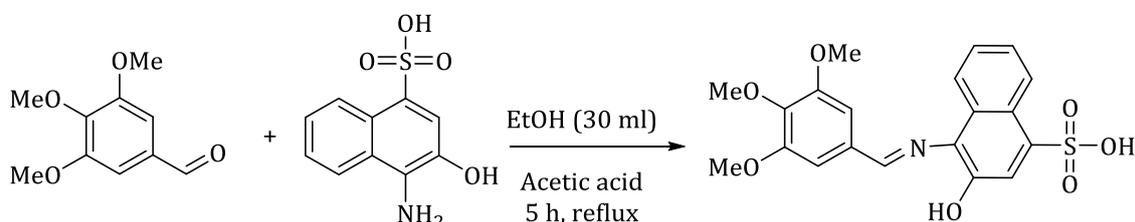
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ABSTRACT

The new ligand Schiff base named [(E)-3-hydroxy-4-((3,4,5-trimethoxybenzylidene)amino) naphthalene-1-sulfonic acid] was synthesized from 3,4,5-trimethoxybenzaldehyde and 1-amino-2-naphthol-4-sulfonic acid in equal molar ratio. A series of new metal complexes of the common molecular formulation $[M(L)_2(H_2O)_2] \cdot H_2O$ are synthesized and characterized by IR, UV-Vis spectra, mass spectra, atomic absorption, elemental analyses, chloride content, magnetic susceptibility and conductivity measurements as well as thermo gravimetric analysis (TGA, DSC). Consistent with results of the magnetic and spectral studies, the advised geometrical structures for all of the prepared complexes have been octahedral formula.

GRAPHICAL ABSTRACT



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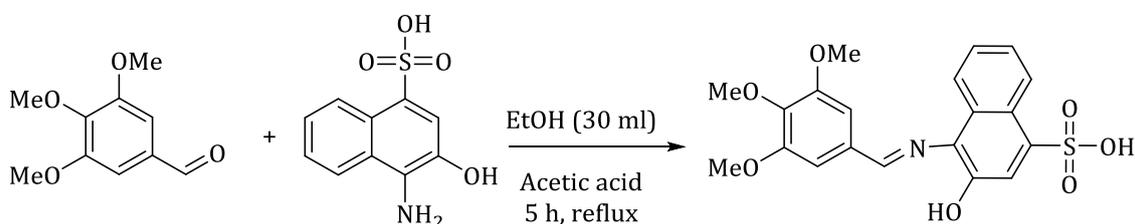
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Introduction

Schiff base composites are considered with the presence of azomethine group (-CH=N-) as per consequence of reflux among aldehyde or ketone with primary amin [1]. Studies have shown that the azomethine group (-CH=N-) is a donor-acceptor group and is an acceptor through the π -orbital of the double bond, and a doner through the non-acceptor of electrons to the nitrogen atom of this group [2]. The Schiff base is derived from benzaldehyde or its derivatives with aniline or its derivatives; the Schiff base molecule is uneven, as the benzene ring attached to the nitrogen atom is outside the level of the rest of the molecule, and it turns out that the compensators on the benzaldehyde molecule are more influential in the electronic distribution than the compensators on aniline cycle [3]. Schiff bases mainly frolic unlike other zones like the pharmacology as anti-fungal, anti-bacterial and anti-cancer agents [4]. Further, they are applied in manufacturing things like thermal Initiations in the Radical Polymerization [5].

Materials and Methods



Scheme 1: Schiff base Ligand preparation

Preparation of metallic complexes [Co(II), Cu(II), Ni(II), Zn(II), Mercury] through ligand (L)

The chelate facilities of ligand and metal (1:2) were arranged *via* the dissolution of 0.95 mmol (0.4 g) of the Schiff base in 10ml of solvent and added to KOH dissolved in 10 ml of absolute solvent. The consistent hydrated metal chloride salt of $\text{CoCl}_2 \cdot 6\text{H}_2\text{O}$, $\text{CuCl}_2 \cdot 2\text{H}_2\text{O}$ =0.081 g, $\text{NiCl}_2 \cdot 6\text{H}_2\text{O}$ =0.114 g, HgCl_2 =0.130 g and ZnCl_2 =0.060 g was liquefied in absolute ethanol 10

All composites used were of highest purity (BDH, Fluka).

Instrumentation

TGA-DSC was characterized and analyzed. The FTIR-Spectra were recorded on Shimadzu 8400-s in a range of ($400 \text{ cm}^{-1} - 4000 \text{ cm}^{-1}$) with the use of the KBr disk. $^1\text{H-NMR}$ spectra were characterized and analyzed. CHNS was accomplished on Euro-EA Elemental.

Preparations of facilities

Preparation of New Schiff base ligand (E)-3-hydroxy-4-((3,4,5-trimethoxybenzylidene)amino)naphthalene-1-sulfonic acid

In a round bottom flask, 3,4,5-dimethoxybenzaldehyde (5 mmol, 1 g) in 15 mL ethanol and 1-amino-2-aphthol-4-sulfonic acid (5 mmol, 1.219 g) in 15 ml ethanol was refluxed for 5 h. [6]. Then, the reaction flask was located in a freeze bath for several records, followed by filtering the precipitate and leaving it to dry, giving a pale green precipitate. The groundwork of Schiff base is illustrated in Scheme1.

mL varied with solvent of the ligand and refluxed for 3h on contents cooling. The facilities were divided out in every event. The produce was filtered, splashed with solvent then dehydrated below vacuity.

Results and discussion

Elemental analyses

Some of the physical and analytical characteristics of ligand as well as its complexes were examined, aimed at finding the chloride content.

Table 1: Physical characteristics and analytical data of ligands as well as their facilities

No.	Compounds	Color	M.P. (°C)	M.wt	Calc./Found%						
					C	H	N	S	O	M	Cl
1	C ₂₀ H ₁₉ NO ₇ S	Pala Green	261-263	417.43	57.49 (57.47)	4.55 (4.53)	3.35 (3.33)	7.66 (7.63)	26.83 (26.80)	-	-
2	[Co(L) ₂ (H ₂ O) ₂].H ₂ O	Brown	139-140	945.79	50.75 (50.73)	4.44 (4.42)	2.96 (2.94)	6.76 (6.74)	28.75 (28.73)	6.23 (6.20)	Nil
3	[Ni(L) ₂ (H ₂ O) ₂].H ₂ O	Dark Brown	>300	945.45	50.76 (50.73)	4.44 (4.41)	2.96 (2.94)	6.76 (6.73)	28.76 (28.74)	6.19 (6.18)	Nil
4	[Cu(L) ₂ (H ₂ O) ₂].H ₂ O	Red	147-148	950.4	50.50 (50.48)	4.41 (4.39)	2.94 (2.93)	6.73 (6.71)	28.61 (28.60)	6.68 (6.66)	Nil
5	[Zn(L) ₂ (H ₂ O) ₂].H ₂ O	Dark Brown	116-118	952.24	50.40 (50.39)	4.41 (4.40)	2.94 (2.92)	6.72 (6.70)	28.56 (28.53)	6.86 (6.84)	Nil
6	[Hg(L) ₂ (H ₂ O) ₂].H ₂ O	Yellowish Brown	>300	1087	44.15 (44.12)	3.86 (3.84)	2.57 (2.55)	5.88 (5.85)	25.02 (25.00)	18.45 (18.40)	Nil

FT-IR of ligand and Facilities

The infra-red spectral data of the Schiff base ligands as well as their complexes are listed in Table 2. Infra-red spectra of complexes were equated with those of free ligand for the purpose of regulating sites coordination, which could be complicated in the chelation. The band in infra-red spectrum of the free ligands at 1172 cm⁻¹ was transferrable to stretching shaking carbonyl group of Schiff base ligand that was lifted to lower frequency in IR spectra of facilities at 1168cm⁻¹. The appearance of band in the range of 582-489cm⁻¹ in infra-red spectra of all of the facilities came as a result of the $\nu(\text{M-O})$ validates [7]. The band at (1658 cm⁻¹) in free ligand's infra-red

spectrum might be a result of stretching vibration of the imine group $\nu(\text{C=N})$ that was changed to a lower or higher frequency; in infra-red spectra of facilities of Cu(II) and Hg(II) rose in to higher frequency, whereas the facilities of Ni(II), Co(II), and Zn(II) fell to lower frequency designated for the harmonization of ligand with metal ion through the N atom [8]. This was further validated by the presence of a newfangled band in range (613-594 cm⁻¹) transferrable to $\nu(\text{M-N})$ [9,10]. It was established that the ligand acted the role of bidentate ligand harmonized to metal ions through O atom of the carbonyl group of benzaldehyde and N atom of imine group for all facilities.

Table 2: Characteristics of infrared bands of absorption of ligand (L) as well as its facilities in cm⁻¹

Compounds	$\nu(\text{OH})$ $\nu(\text{OH})$	$\nu(\text{C=N})$	$\nu(\text{C-O})$	$\nu(\text{M-N})$	$\nu(\text{M-O})$
C ₂₀ H ₁₉ NO ₇ S	3240 1315	1658	1172	-----	-----
[Co(L) ₂ (H ₂ O) ₂].H ₂ O	3348 1315	1539	1168	609	489
[Ni(L) ₂ (H ₂ O) ₂].H ₂ O	3402 1361	1550	1161	605	528
[Cu(L) ₂ (H ₂ O) ₂].H ₂ O	3448 1365	1651	1168	613	582
[Zn (L) ₂ (H ₂ O) ₂].H ₂ O	3417	1546	1168	594	528

	1357				
[Hg(L) ₂ (H ₂ O) ₂].H ₂ O	3414 1354	1635	1168	597	513

UV-Vis

The UV-Vis of ligand and its Ni(II), Cu(II), Co(II), Zn(II) and Mercury facilities were deliberate and spectra1 data were registered as displayed in Table 3. UV-Vis of Schiff base ligands has been described mostly by three absorption top at (235

nm) consigned to (π→π*), at (345nm) that has been allocated to (n→π*) and at (470nm) allocated to Charge transfer [11]. Those electronic transitions were lifted near lower or higher frequency in electronic spectra of all of the set facilities [12-14].

Table 5: UV-Vis spectra of ligand as well as its chelate facilities

Compound	λ(nm)	υ(cm ⁻¹)	ε _{max} L/mol cm	Transition	Λ _m S.cm ² .mole ⁻¹
C ₂₀ H ₁₉ NO ₇ S	235	42553	457	π→π*	-----
	345	28985	1508	n→π*	
	470	21276	25	C.T	
[Co(L) ₂ (H ₂ O) ₂].H ₂ O	237	42194	4440	π→π*	3.41
	344	29069	2389	n→π*	
	473	21141	42	⁴ T _{1g} → ⁴ T _{1g(p)}	
	603	16583	15	⁴ T _{1g} → ⁴ A _{2g}	
	927	10787	4	⁴ T _{1g} → ⁴ T _{2g}	
[Ni(L) ₂ (H ₂ O) ₂].H ₂ O	241	41493	1419	π→π*	2.16
	344	29069	2377	n→π*	
	480	20833	217	³ A _{2g} → ³ T _{1g(P)}	
	745	13422	28	³ A _{2g} → ³ T _{1g}	
	789	12674	19	³ A _{2g} → ³ T _{2g}	
[Cu(L) ₂ (H ₂ O) ₂].H ₂ O	245	40816	1693	π→π*	17.72
	276	36231	1457	π→π*	
	344	29069	2416	n→π*	
	502	199203	2500	² B _{1g} → ² E _g	
	888	11261	9	² B _{1g} → ² A _{2g}	
[Zn (L) ₂ (H ₂ O) ₂].H ₂ O	241	41493	1133	π→π*	9.48
	281	35587	933	n→π*	
	419	23866	396	C.T	
[Hg(L) ₂ (H ₂ O) ₂].H ₂ O	237	42194	771	π→π*	14.17
	348	28735	682	n→π*	
	473	21141	130	C.T	

Magnetic measurement

$$\mu_{S+L} = \sqrt{4S(S+1) + L(L+1)} \quad B.M$$

$$\mu_S = \sqrt{4S(S+1)} \quad B.M, \quad S=n/2$$

$$\mu_S = \sqrt{n(n+2)} \quad B.M$$

$$X_M = X_g \times M_{wt} \quad (1)$$

$$X_A = X_M - D \quad (2)$$

$$\mu_{eff} = 2.82\sqrt{X A. T} \quad (3) \quad B.M$$

Co(II) composite displays magnetic moment μ_{eff} (4.82BM) that is consistent with 3 unpaired electrons. This significance submits octahedral environment around ion of Co (II) [15]. Ni(II) complex displays magnetic moment μ_{eff}. (2.80

BM) at room temperature that is resultant to 2 unpaired electrons. This significance directs octahedral geometry about Nickel(II) ion [16]. Cu(II) complex indications μ_{eff} (1.75 BM) conform to unpaired electrons. This value suggests octahedral environment around ion of Cu(II) [17]. The value of the magnetic moment μ_{eff} of Zn(II) associates with Mercury composite (0.00 B.M) for the reason of being diamagnetic in nature (d^{10}) [17].

¹HNMR

In mixture, as in solid state, this marvel was set by infra-red and NMR spectra. ¹HNMR ligand spectrum in the DMSO-*d*₆ revealed signal at chemical shift (δ H = 9.6 ppm, s) credited to the one proton of the hydroxyl group phenol δ (OH) [18], and at (8.7 ppm, s) credited to the proton of the hydroxyl group sulfonic δ (OH)). Resonance values at the chemical shifting (δ H = 6.7 -7.6 ppm, m) are flexible to the aromatic ring protons and at (3.0, 3.2 ppm, s) official to nine protons of (-OCH₃).

GC-mass of ligand and complexes

Electro-spray mass spectrum of ligand displays parent ion top at (M/Z)=(417) that agrees with [M⁺], other rubbles as well as their comparative abundance and disintegration pattern has been presented. Electro-spray mass spectrum of Co(II) complexes illustrates parent ion top at

(M/Z)=(928) that agrees with [M⁺], other fragments as well as their comparative abundance and disintegration configuration is exposed. Electro-spray mass spectrum of Ni(II) complexes confirm the parent ion top at (M/Z)=(927), corresponding to [M⁺], other fragments as well as their comparative abundance and disintegration patterns having been illustrated.

Thermal decomposition of ligands and Cu(II), Zn(II) Complexes

The thermogram of prepared ligand (C₂₀H₁₉NO₁₇S) was carried out by thermogravimetric analysis (TGA) and differential calorimetry (DSC), in an inert atmosphere using argon gas at an average temperature of 10°Cmin⁻¹ and a weight of (26mg) of the sample. The TGA curve of the prepared ligand (L) shows that it undergoes one phase of weight loss [19,20]. Thermal study of the complex CuC₄₀H₄₂N₂O₁₇S₂ was carried out in an inert atmosphere of argon at an average temperature of 10°Cmin⁻¹ with a weight of (21 mg). The TGA curve shows the complex going through four stages of weight loss.

Thermal study of the complex ZnC₄₀H₄₂N₂O₁₇S₂ was carried out in an inert atmosphere of argon at an average temperature of 10 °Cmin⁻¹ by the weight of 17mg [21]. The TGA curve shows that the complex had one phase of weight loss.

Table4: Thermal analysis of the ligand as well as its complexes

Compound	Stages	De-composition Temperature Initial-inal(°C)	Estimated(calculated)		Assignments
			Mass Loss	Total mass loss	
L	1	115-597.547	24.62(24.65)	24.62 (24.65)	-C ₂₀ H ₁₃ NO ₆ S
[Cu(L) ₂ (H ₂ O) ₂].H ₂ O	1	60-166.897	1.86 (1.87)	18.48 (18.50)	-C ₂ H ₁₂ O ₃
	2	166.879-246.4	2.18 (2.19)		H ₃ SO ₄ ⁻
	3	246.4-392.132	9.42 (9.43)		-C ₁₉ H ₂₂ O ₉ S
	4	392.132-596.753	5.02 (5.03)		-CuC ₁₁ H ₂ NO
[Zn(L) ₂ (H ₂ O) ₂].H ₂ O	1	120-594.869	14.95(14.96)	14.95 (14.96)	-C ₄₀ H ₃₉ NO ₁₅ S ₂

Conclusion

The aim of this research was to create a new ligand containing the azomethine group (-CH=N-) as a donor-acceptor group countered with around transition metal ions in Ni(II), Co(II), Cu(II), Zn(II)

and mercury to offer a series of new metal complexes of the common molecular formulation [M(L)₂(H₂O)₂].H₂O. The metal complexes were proved to have an octahedral formula.

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Authors' contributions

All authors contributed toward data analysis, drafting, and revising the paper and agreed to be responsible for all the aspects of this work.

Conflict of Interest

We have no conflicts of interest to disclose.

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