

PREPARATION, CHARACTERIZATION AND ANTIMICROBIAL STUDIES OF Cd(II)-Hg(II) HETEROBIMETALLIC COMPLEX

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ABSTRACT

This study focuses on the Preparation, characterization and antimicrobial studies of Cd(II)-Hg(II) heterobimetallic complex synthesized by combining two Monometallic complexes. Cd(II)- Schiff base monometallic complex derived from o-vanillin and ethylenediamine and Hg(II)- 1-nitroso-2-naphthol monometallic complex. Each Monometallic complex was individually synthesized in ethanol and then combined to obtain the heterobimetallic complex. characterization through IR, ¹H-NMR, UV-Visible spectroscopy and Metal percentage analysis confirmed successful co-ordination and complex formation. Biological studies showed that the heterobimetallic complex exhibited superior antibacterial and antifungal activity compared to the monometallic complexes.

KEYWORDS: Heterobimetallic complex, IR, ¹H-NMR, UV-Visible spectroscopy, Metal percentage analysis, antibacterial activities and antifungal activities.

1. INTRODUCTION

In coordination chemistry, complexes—particularly those of the first row transition metals—are studied in great detail because they can result in mono- and polynuclear species with some biological significance.^[1]

Because of its many uses, the chemistry of Schiff bases and related metal complexes has attracted a lot of attention. When primary amines condense with carbonyl molecules, schiff bases are produced.^[2] Because they are bound to metal ions in a variety of ways, chelating ligands with O and N donor atoms exhibit a wide range of biological activity and are particularly interesting.^[3,4]

The biological activity of metals that have been the subject of metal coordination chemistry is increased when metal ions are combined with Schiff base ligands to create metal complexes.^[5] Additionally, because transition metal complexes of Schiff's bases showed antibacterial and anti-inflammatory properties, they were synthesised and evaluated for biological activity.^[6-9] Through catalysis, substrate carriers, and dyes, their metal chelates have been crucial to the growth of chemical industries.^[10]

The synthesis, characterisation, and biological activity of bimetallic complexes have been extensively studied during the past few decades. Research on the synthesis and characterisation of novel bimetallic complexes is expanding quickly due to the numerous applications of these complexes. Pesticides, fungicides, and antifouling agents are only a few of the industrial and agricultural uses for bimetallic complexes.^[11]

2. EXPERIMENTAL

2.1 Materials

Without further purification, commercially available reagent-grade o-vanillin, ethylenediamine, Cadmium chloride, 1-Nitroso-2-naphthol, Mercuric chloride, and ethanol were used.

2.2 Physical measurements

FT-IR spectra were recorded using FT-IR.8300 Shimadzu in the range of (4000-200) cm⁻¹. The measurements were recorded using a concentration of (10⁻³M) of the complex in ethanol. NMR spectroscopic studies were recorded on a Bruker 400 MHz and 75.45 MHz. Elemental percentage was analyzed using PerkinElmer 2400 CHN elemental analyzer. UV/ Vis spectroscopic study was performed using a Shimadzu UV 1700

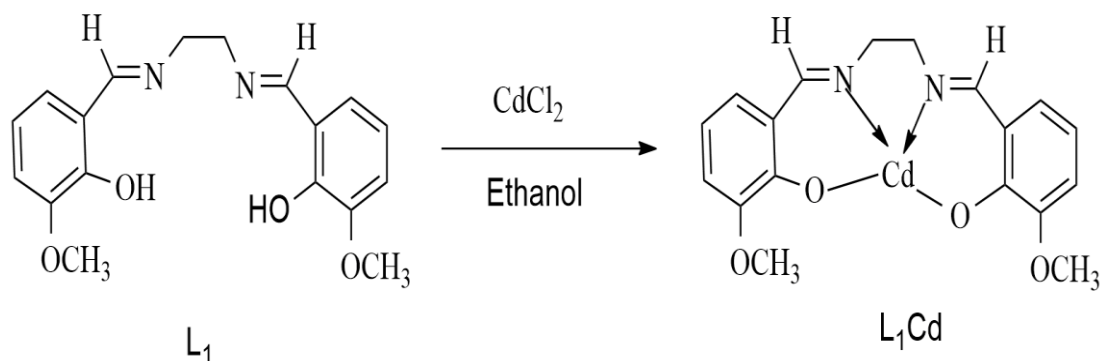
spectrophotometer.

2.3 Preparation and characterization of monometallic complex L_1Cd

[N,N'-Bis(o-vanillinidene) ethylenediamine] Cd(II)

The ligand N, N'-Bis(o-vanillinidene) ethylenediamine (L_1) was used to form a complex with Cd(II) to get the L_1Cd complex. An ethanolic solution of Cadmium

chloride was added in a 1:1 ratio to the ethanolic solution of ligand (L_1) and stirred for 20 minutes at room temperature. The faint yellow coloured precipitate was obtained. The product was filtered off and then dried under a vacuum.



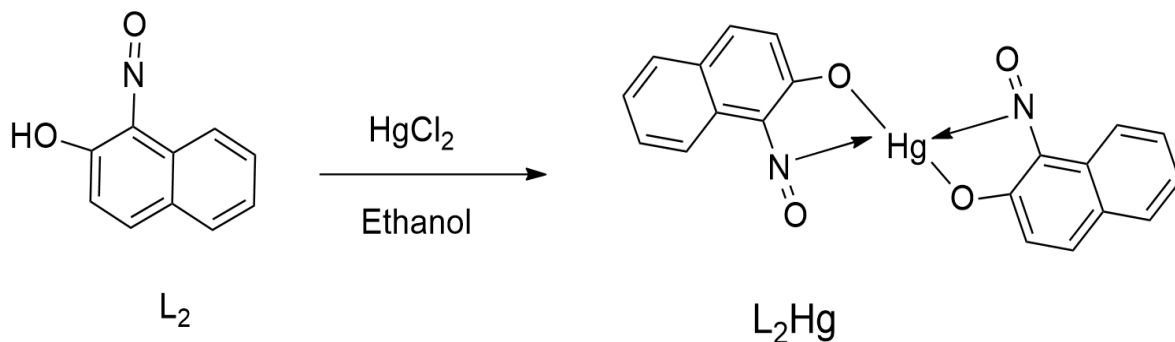
Scheme 1: Synthesis of monometallic complex L_1Cd .

2.4 Preparation and characterization of monometallic complex

L_2Hg [1-Nitroso-2-Naphthol] Hg(II)

The hot ethanolic (40° C) solution of (L_2) was added to the ethanolic solution of mercuric chloride with stirring. The yellow precipitate (L_2Hg) was formed upon stirring. Stirring was continued for one hour to ensure the

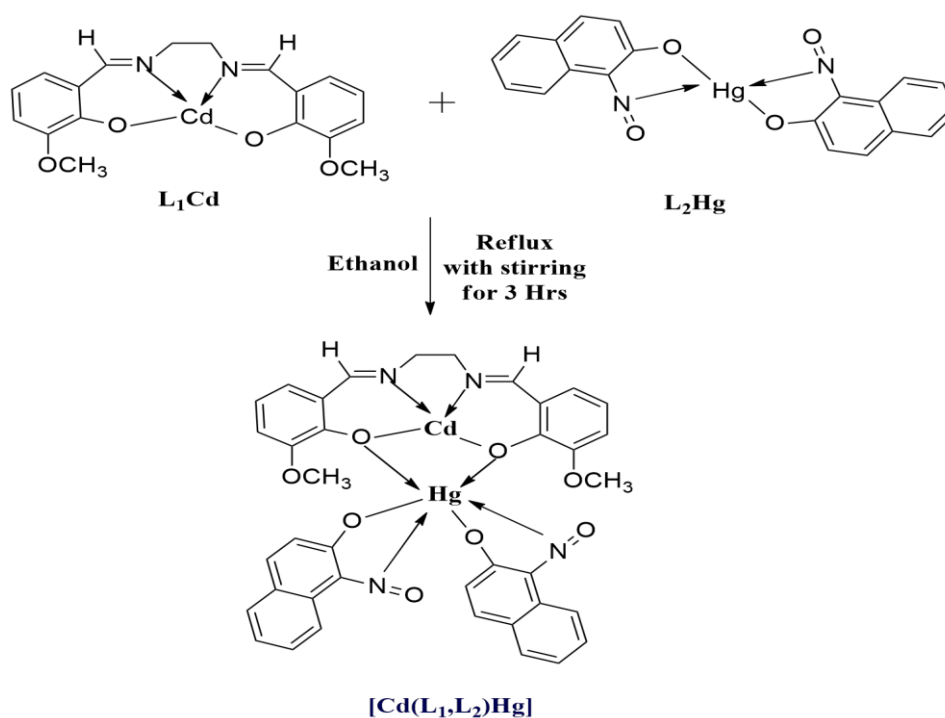
reaction was completed. The product was filtered off, washed with hot ethanol, followed by cold water, and then dried under a vacuum. The complex is purified with ethanol and the prepared complex is characterized by IR and elemental analysis.



Scheme 2: Synthesis of monometallic complex L_2Hg [1-Nitroso-2-Naphthol] Hg(II)

2.5 Preparation and characterization of hetero-bimetallic complex $[Cd(L_1,L_2)Hg]$

The L_1Cd complex was then added to the complex prepared by using the reagent 1-nitroso-2-naphthol. The reaction mixture of ethanolic solution of L_1Cd complex and the ethanolic solution of L_2Hg complex was then refluxed with stirring for three hours. The buff coloured product precipitated out, filtered at room temperature and washed with sufficient ethanol.

Scheme 3: Synthesis of hetero-bimetallic complex [Cd(L₁, L₂) Hg]

3. RESULTS AND DISCUSSION

3.1 Physical characteristics and elemental analysis

COMPLEX/ Ligand	MW (gmol ⁻¹)	COLOUR	% YIELD	% C	% H	% N	% Cd	% Hg
				Found (Calculated)				
(L ₁ Cd)	433.93	Faint yellow	58.2	55.21 (56.11)	4.05 (4.67)	6.56 (7.27)	14.96 (15.30)	--
(L ₂ Hg)	544.59	Black	72.6	40.80 (44.06)	1.80 (2.20)	4.80 (5.14)	--	25.24 (36.83)
[Cd(L ₁ ,L ₂)Hg]	982.9	Buff	56.1	45.95 (46.39)	2.84 (3.05)	5.17 (5.69)	11.01 (11.43)	19.80 (20.40)

3.2 Infrared spectroscopic study

In the L₁Cd complex a negative shift with reduced intensity were observed for (C=N) and (CH₂N) stretching bands. The sharp band due to the ligand's phenolic (OH) is absent in the complex L₁Cd and bimetallic complex [Cd(L₁,L₂)Hg] to indicate the coordination of the phenolic oxygen to the metal ion after deprotonation. Such coordination is also supported by shifting the

phenolic ν(C–O) band to lower wave numbers at 1430 cm⁻¹ in the complex L₁Cd and at 1344 in the bimetallic complex [Cd(L₁,L₂)Hg]. The coordination of the azomethine nitrogen and phenolic oxygen in bimetallic complex [Cd(L₁,L₂)Hg] is further supported by the appearance of two non-ligand bands at 510–680 and 400–560 cm⁻¹ due to ν(M–N) and ν(M–O), respectively.

Complex	ν Ar C=C	ν Ar C-H	ν CH ₂ -N	ν CH=N	ν N=O	ν C-O	ν M-O	ν M-N
(L ₁ Cd)	1422.1 (s)	3152.2 (m)	1070 (s)	1626.0 (s)	--	1430.8 (s)	573.7 (m)	475.2 (s)
(L ₂ Hg)	1434.4 (s)	908.1 (s)	--	1608.5 (s)	1538.8, (s)	1274.2 (m)	450.1 (s)	520.1 (m)
[Cd(L ₁ ,L ₂)Hg]	1431.2 (s)	3120.6 (m)	1211.5 (s)	1635.9 (s)	--	1344.1 (s)	522.7 (s)	677.8 (s)

3.3 NMR Spectroscopy

The ¹H NMR spectra of the metal complex (L₁Cd) demonstrated characteristic azomethine (CH=N) protons at 4.75 ppm as a singlet. As a singlet, the (CH₂) protons in the metal complex (L₁Cd) were observed at 3.35–

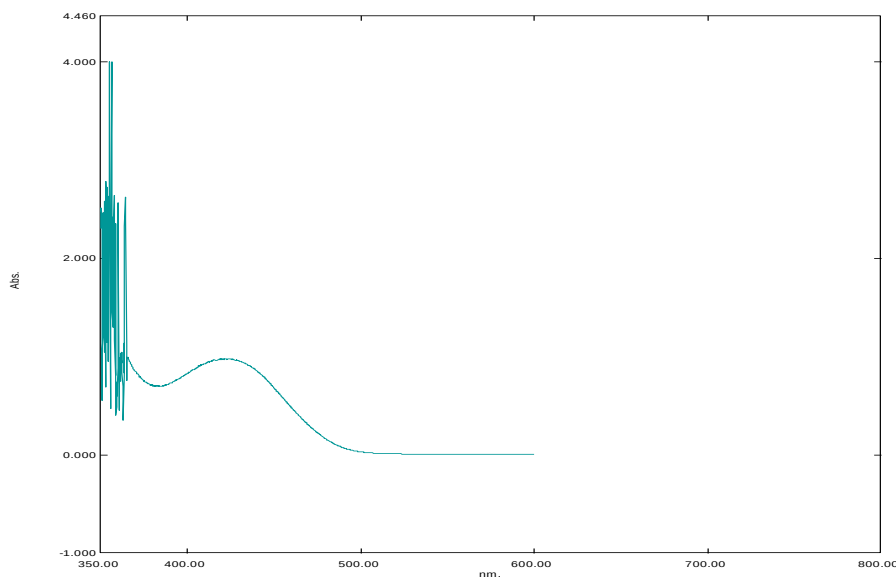
3.92 ppm.

3.4 UV/ Vis spectroscopic study

The UV/Vis spectrum of [Cd(L₁, L₂)Hg] was recorded in the 350–800 nm range. The UV–Vis absorption spectrum

exhibits a broad absorption band centered near 420 nm, corresponding to electronic transitions $\pi \rightarrow \pi^*$ (conjugated systems) within the complex. A sharp

decrease in absorbance beyond 480–500 nm indicates an optical absorption edge in this region. The estimated optical band gap is 2.5 eV.



3.5 Anti-bacterial

Antibacterial activity against *E. coli*, *Bacillus subtilis*, *S. Aureus*, *P. aeruginosa* bacteria by healthy diffusion method. The inoculums of the microorganism were prepared from the bacterial cultures. 15 ml of nutrient agar (Hi media) medium was poured into clean sterilized Petri plates and allowed to cool and solidify. 100 μ l of

broth of bacterial strain was pipetted out and spread over the medium evenly with a spreading rod till it dried properly. Once the agar was hardened, the sample slides were placed on the plate in the manner, and the plates were incubated at 37 $^{\circ}$ C for 24 h. Antibacterial activity was evaluated by measuring the diameters of the zone of inhibitions (ZI).^[12]

Antibacterial Activity of samples against *E.coli*, *Bacillus subtilis*, *S Aureus*, and *S.typhi*.

Sr. No	SAMPLES	CONCENTRATION (mg/ml)	ZONE IN DIAMETER (mm) <i>E.coli</i>	ZONE IN DIAMETER (mm) <i>Bacillus subtilis</i>	ZONE IN DIAMETER (mm) <i>Staphylococcus Aureus</i>	ZONE IN DIAMETER (mm) <i>S.typhi</i>
1	Standard (streptomycin)	1 mg	24	25	20	26
2	(L ₁ Cd)	5 mg	5	4	6	7
3	(L ₂ Hg)	5 mg	5	7	4	8
4	[Cd(L ₁ , L ₂) Hg]	5 mg	16	18	15	20

3.6 Anti-fungal

Antifungal activity against *Candida Albicans* *A. Niger* (Agar healthy plate diffusion Method)
Diffusion method for the determination of zone of inhibition.

Antifungal activity Stock solution for antifungal activity: For the antifungal study, sample concentrations of 5 mg was stored in a refrigerator till further use. The antifungal activities of the sample were evaluated using an agar well diffusion assay. The assay was carried out according to the method of (Hufford et al., 1975). Sabouraud dextrose agar (Hi media) was used to grow fungus. Media with acidic pH (pH 5.5 to 5.6) containing a relatively high concentration of glucose (40%) is prepared by mixing (SDA) Sabouraud dextrose and distilled water and autoclaved at 121 $^{\circ}$ C for 15 minutes.

Twenty-five ml of molten (45 $^{\circ}$ C) SDA medium was aseptically transferred into each 100mm $\times$ 15mm sterile Petri dish. For counting spore (fungi) were suspended in normal saline to make volume up to 1 ml and then counted with help of haemocytometer. Once the agar was hardened, 6 mm wells were bored using a sterile cork borer. Then 0.1 ml (100 μ l) from the stock solution of the sample having a final concentration of 5 mg was placed in each well, and the plates were incubated for 72 hours at 29 $^{\circ}$ C. The antifungal activity was measured as the diameter (mm) of the clear zone of growth inhibition. (Umadevi et al., 2003).^[13,14,15]

Antifungal Activity of heterobimetallic complex against *Candida Albicans* and *A. Niger*

Sr. No	SAMPLES	CONCERN TRACTION (mg/ml)	ZONE IN DIAMETER (mm) against <i>Candida albicans</i>	ZONE IN DIAMETER(mm) against <i>A. Niger</i>
1	STD	1 mg	20	22
2	(L ₁ Cd)	5 mg	6	9
3	(L ₂ Hg)	5 mg	2	5
4	[Cd(L ₁ ,L ₂)Hg]	5 mg	15	16

4. CONCLUSION

A stable bimetallic complex of Cd(II) & Hg(II) was successfully prepared. This prepared heterobimetallic complex [Cd(L₁,L₂)Hg] is characterized through spectral studies. Bimetallic complex exhibits antibacterial activities against *S.aureus*, *B.subtilis*, *S.typhi* and *E.coli*. Comparison of the inhibition values of the monometallic complexes and the heterobimetallic complex indicate that the prepared heterobimetallic complex [Cd(L₁,L₂)Hg] exhibits higher antimicrobial activity.

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