

Preparation and antimicrobial studies of some metal complexes of 2-(imine-2/3-hydroxybenzene)-3-hydroxyiminobutane

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Abstract:

Various Schiff bases and their metal complexes have wide applications in various fields such as pharmaceuticals, analytical, clinical, biological etc. due to their biological activity such as antibacterial, antifungal, anticancer, antidiuretic, antioxidant etc. Reaction between 2,3-Butanedione 3-monoxime, 2/3-hydroxyaniline and Mn(II) and Fe(II) salt gives complexes of the type $[ML_2(H_2O)]$. The ligand and the complexes were characterized by elemental analysis, magnetic susceptibility measurements and spectral (I.R., U.V.-Vis.) analysis. I.R. and U.V-Vis. spectra show bidentate nature of the ligand. The spectral and magnetic study suggests octahedral geometry for all metal complexes. Spectroscopic study indicates coordination occurs through oxime oxygen after deprotonation and nitrogen of azomethine group. The ligand and metal complex were screened for their antimicrobial activity against microorganisms *S. aureus* (gm positive), *P. aeruginosa* (gm negative), *Aspergillus niger* and *Candida albicans*. Both the ligand shows moderate activity against all microorganisms as compared to the standard drugs. Both the ligands and their Mn(II) complexes behave as good antimicrobial agent as compared to Fe(II) complexes..

Keywords: Bidentate Ligand, Magnetic susceptibility measurements, Octahedral Complex, antifungal and antibacterial activity.

1. Introduction:

Metal complexes with various donor groups had attracted by many workers [1-3] due to their biological, pharmacological, clinical and analytical importance. Thiophene derivatives exhibit an array of biological activity such as antibacterial and antifungal activity, antioxidant and molecular docking study [4-7]. Piperonylamine Schiff bases metal complexes are most potent antimicrobial agents as compared to its Schiff bases [8]. Macrocyclic Schiff base metal complexes derived from 1,4-dicarbonylphenyl dihydrazide and pentane-2,4-dione also acts as growth inhibiting agents against some bacteria and fungi.[9]. There is enormous interest presently in the field of coordination chemistry of '3d' transition metals with Schiff bases. They have also been used as biological models [10], oxygen carriers and antifertility agents on male albino rats [11]. Some studies of metal complexes of diacetylmonoxime and their related ligands have been reported [12-13]. We have already reported the antibacterial and antifungal properties of 2-nitroaniline-2,3-Butanedione-3-monoxime and 3-nitroaniline-2,3-Butanedione-3-monoxime and their metal complexes [14]. In this paper we reported the study of 2-(imine 2-hydroxybenzene)-3-hydroxyiminobutane L^1 , 2-(imine 2-hydroxybenzene)-3-hydroxyiminobutane L^2 and their Mn(II)

and Fe(II) complexes. The main interest in this ligand originates in its imine group containing -N-O- donar atom and to study the biological activity of ligand and its metal complex.

2. Materials and methods:

All the chemicals used are of analytical grade such as 2,3-Butanedione 3-monoxime, 2/3-hydroxy aniline and metal chlorides etc. Ligand prepared according to the literature method [14-6]. The solvents Ethanol, Petroleum ether, Ethyl acetate were use after distillation.

Synthesis of Schiff base ligands:

Scheme 1: The alcoholic solutions of 2/3-hydroxyaniline and 2,3-butanedione 3-monoxime were mix together in 1:1 molar concentration. After refluxing for about 3-4 hrs., solid crude product were form, after cooling, product were recrystallize and dried in desiccator over anhydrous CaCl₂ [4-5].

Synthesis of metal complexes:

Scheme 2: A suitable ligand and metal chloride in 1:2 molar concentrations were reflux for 4-5hrs. Solid colored products were form on cooling. After recrystallization, the complexes dried over anhydrous CaCl₂. The physical and analytical data listed in table 1.

Antibacterial activity and Antifungal activity:

The synthesized ligand and complexes tested for in vitro growth inhibitory activity against gram-positive bacteria *Staphylococcus aureus* and gram-negative bacteria *Pseudomonas aeruginosa*. Nutrient agar plates made uniformly surface inoculated from the culture of the tested microorganisms. Well was prepared on the agar medium at the center by using cork borer. Normal saline water was use to prepare the inoculum of the bacteria to be used for the antibacterial study [10].

The synthesized ligand and complexes also screened for in vitro growth inhibitory activity against *Aspergillus niger* and *Candida albicans*. Sabouraud dextrose agar plates made uniformly and ditch made on the agar medium at the center. Two separate striking lines of two different fungi made on both the sides of ditch [6, 10]. Mueller Hint Agar Media (HI Media) used to subculture various strains of microorganisms. The lowest concentration of the compound which shows positive antibacterial and antifungal activity was subjected to dilution method for quantitative measurement of inhibitory (micro static) activity. The lowest concentration, which completely inhibited visible microbial growth, was record as the Minimum inhibitory concentration (MIC, µg/ml) [5, 9]. The stock solution of test compound (0.04mg/ml) was prepare in DMF and subsequent dilutions of stock solution made in the same solvent. The activity of DMF was check previously. Under aseptic conditions, the diluted test solutions with different concentrations added to the disc and ditch placed on the numbered plates. Then the plates were incubating at room temperature for 24 hr. During this period, the test solution diffused and the growth of the inoculated microorganisms affected. Antibacterial activity was indicating by the presence of clear inhibition zone around the well and antifungal activity indicated by the presence of inhibition zone nearer to the ditch.

3. Results and discussion:

The complex is stable in air but decomposed at high temperature. Both the compounds are easily soluble in dimethylformamide (DMF) and dimethylsulphoxide (DMSO). The physical and elemental analysis of ligand and metal complex are listed in Table 1.

Table 1: Physical and Elemental analysis

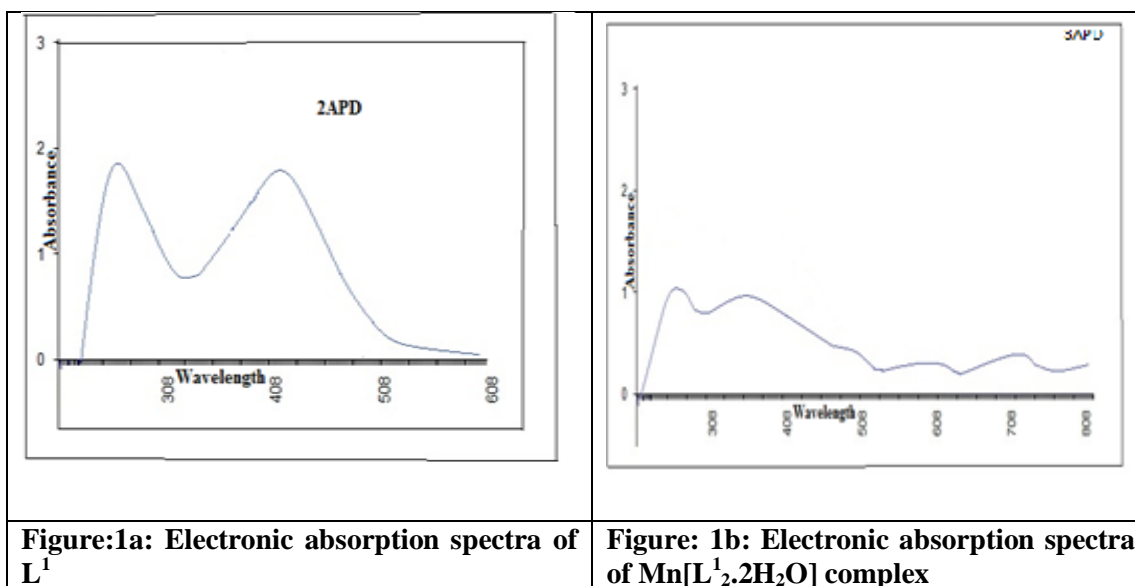
Name of the compounds	% Yield Color	Melting point	μ_{eff} BM	Elemental Analysis found (calculated) %			
				C %	H %	N%	Mn%
L ¹ [C ₁₀ H ₁₂ N ₂ O ₂] 192MW	90% Faint Violet	368K	-	57.88 (60.00)	4.827 (5.45)	10.56 (12.72)	-
Mn[L ¹ ₂ .2H ₂ O] Mn[C ₂₀ H ₂₆ N ₂ O ₆]	69% Dark red brown	<570K	6.590	50.73 (50.74)	5.52 (5.53)	11.82 (11.83)	11.59 (11.60)
Fe[L ¹ ₂ .2H ₂ O] Fe[C ₂₀ H ₂₆ N ₂ O ₆]	68% Reddish brown	<570	4.635	50.63 (50.64)	5.54 (5.52)	11.80 (11.81)	11.75 (11.77)
L ² [C ₁₀ H ₁₂ N ₂ O ₂] 191.1MW	95% Brown	375K	-	51.89 (54.31)	3.6806 (4.974)	18.29 (19.0)	-
Mn[L ² ₂ .2H ₂ O] Mn[C ₂₀ H ₂₆ N ₂ O ₆]	64% Dark red brown	<573K	6.400	50.74 (50.74)	5.51 (5.53)	11.80 (11.83)	11.61 (11.60)
Fe[L ² ₂ .2H ₂ O] Fe[C ₂₀ H ₂₆ N ₂ O ₆]	65% Reddish brown	<570K	4.632	50.64 (50.64)	5.54 (5.52)	11.78 (11.81)	11.74 (11.77)

Electronic Spectra and Magnetic moment:-

Table 2: Electronic spectral data of complexes.

Name of the compounds	$\pi \rightarrow \pi^*$ and $n \rightarrow \pi^*$ transitions and charge transitions (nm)	d-d transitions
L ¹	260, 430	-
Mn[L ¹ ₂ .2H ₂ O]	250, 360	502, 615, 700
Fe[L ¹ ₂ .2H ₂ O]	280, 374	588, 495
L ²	264, 425	-
Mn[L ² ₂ .2H ₂ O]	275, 378	506, 624, 710
Fe[L ² ₂ .2H ₂ O]	279, 375	585, 490

The electronic spectrum of ligand and complexes helps to indicate the geometry. The important electronic spectral bands recorded in table 2. The spectra of strong absorption band in the range 260- 275nm in both ligand and metal complex remain more or less at the same frequency [17] which are $\pi \rightarrow \pi^*$ charge transfer transitions. The azomethine group which is assigned as $n \rightarrow \pi^*$ transitions observed at 430nm and 425 nm in the ligand shifted to lower frequencies in metal complex indicating that the imine nitrogen atom is involved in coordination to the metal ion [18]. The low intensity absorption bands associated with d-d transition observed only in metal complexes supports the coordinated geometry of the metal complexes [19]. Figure:1: a and b indicate the electronic spectra of L^1 and $Mn[L^1_2 \cdot 2H_2O]$ complex. The room temperature magnetic moment values also help to indicate the octahedral geometry of metal complexes. The magnetic moment value of Mn^{II} at 6.4 BM is same as theoretical spin only value ($\mu_{eff} = 6.4$ to 6.5 BM) for Mn^{2+} (d^5 system) and 4.635BM is near to theoretical spin only value ($\mu_{eff} = 5.0$ to 5.6 BM) for high spin d^6 system of Fe^{2+} . In octahedral $Fe(II)$ complexes 5D ground term leads to significant orbital contribution and effective magnetic moments often lie between the limits of 5.0-5.6 BM. Actual value depends upon the magnitude of orbital angular momentum.



Infrared Spectra:-

Table 3: I.R. bands of ligand and metal complexes.

Compound	$\nu(C=N^*)$	$\nu(C=N)$	$\nu(N-O)$	$\nu(O-H)$ of (phen-OH)	Coord.- H_2O	$\nu(M-N)$	$\nu(M-O)$
L^1	1630	1565	998	3360	-	-	-
$Mn[L^1_2 \cdot 2H_2O]$	1625	1563	1101	3360	3460	350	435
$Fe[L^1_2 \cdot 2H_2O]$	1625	1563	1105	3360	3460	355	440
L_2	1625	1531	998	3296	-	-	-

Mn[L ² .2H ₂ O]	1634	1496	1113	3297	3442	455	521
Fe[L ² .2H ₂ O]	1609	1496	1110	3345	3445	458	520

The I.R. spectra (Table 3) shows, complexes behave as bidentate coordinating ligand via the azomethine nitrogen (C=N*) and oxygen of oxime (-NOH) group by replacement of hydrogen ion forming six-membered ring around metal ion [13]. In the ligand azomethine (C=N*) group is at high wavenumber but in metal complex it shifts to lower wavenumber, this indicates the imine nitrogen atom involved in coordination to the metal ion [17, 21] while oxime (C=N) remain more or less at the same position. The N-O band in ligand observed at 998cm⁻¹ but in metal complexes it shifts to higher wavenumber, this indicates the oxygen atom by replacing oxime proton involved in coordination to the metal ion [13, 19]. Phenolic (-OH) in ligands and metal complex observed in rang 3200-3445cm⁻¹ means (-OH) of hydroxyl group does not involved in coordination or ligand formation. A broad band observed in all the complexes in rang ~3450 cm⁻¹ due to (-OH) of the coordinated H₂O. This is supported by the appearance of an additional band in rang 950-900 cm⁻¹ for (-OH) rocking deformation and 800-750 cm⁻¹ for (-OH) wagging mode of coordination which is not observed in the ligand spectrum [19, 20]. Thus, H₂O is coordinated in metal complexes. New bands appear in the range 435 -520 cm⁻¹ and 350 to 450cm⁻¹ assignable to the ν(M-O) and ν(M-N) resp. in metal complexes only [22, 23]. Figure 2: a and b indicates IR spectra of L² and its Mn^{II} complex.

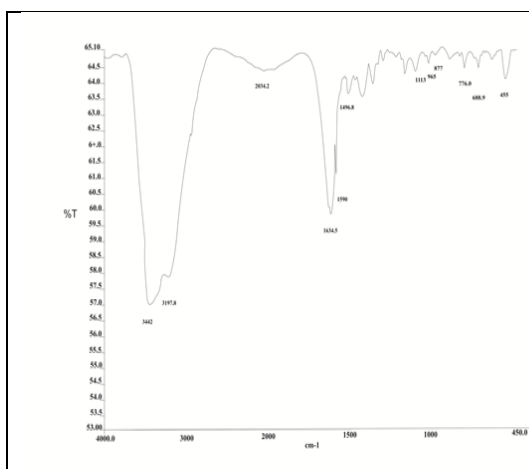


Figure 2a: I.R spectra of L².

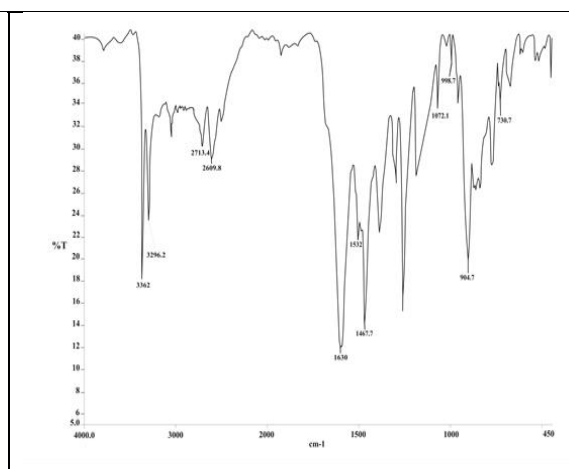


Figure 2b: I.R spectra of Fe[L².2H₂O] complex.

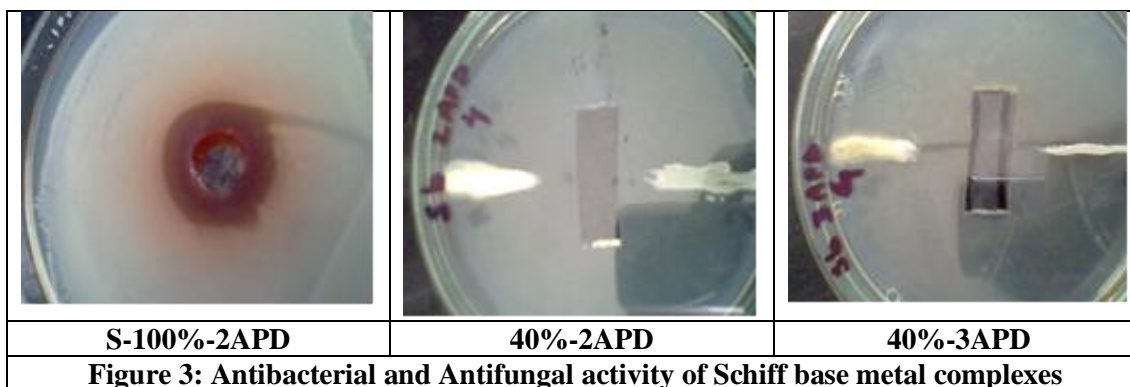
Antibacterial and Antifungal activity:-

Table 4: Antibacterial activity:

Name of compo	<i>Pseudomonas aeruginosa</i> (mm)					<i>Staphylococcus aureus</i> (mm)				
	20	40	60	80	100	20	40	60	80	100
L ¹	-	+	+++(-	+++)	+++)	++	++	+++)	+++)	+++)
Mn[L ¹ .2H ₂ O]	-	-	-	-	+(18)	-	-	-	-	-

Fe[L ¹ .2.2 H ₂ O]	-	-	-	-	-	-	-	-	-	-
L ²	-	-	-	-	+(16)	-	-	-	-	+(14)
Mn[L ² . 2H ₂ O]	-	-	+	++	++	-	-	+(16)	++	++
			(15)	(20)	(23)				(20)	(23)
Fe[L ² .2.2 H ₂ O]	-	-	-	-	-	-	-	-	-	-
Ciprofl	++	+++	+++	+++	+++	++	+++	+++	+++	+++
-No activity, + 10 to 19mm (less), ++20 to 29mm (moderate), +++ above 30mm (highly active)										
Name of compo	<u><i>Aspergillus niger</i></u>					<u><i>Candida albicans</i></u>				
	20	40	60	80	100	20	40	60	80	100
L ¹	+	++	++	++	++	+	+	+	+	+
Mn[L ¹ .2. 2H ₂ O]	+	++	++	++	++	+	+	+	+	+
Fe[L ¹ .2.2 H ₂ O]	-	++	++	++	++	-	-	-	-	-
L ²	-	+	+	++	++	-	-	-	-	+
Mn[L ² . 2H ₂ O]	-	+	+	++	++	-	-	+	+	++
Fe[L ² .2.2 H ₂ O]	-	++	++	++	++	-	-	-	-	-
Flucona zole	+++	+++	+++	+++	+++	+++	+++	+++	+++	+++
+ less activity, ++ moderate activity, +++ higher activity.										

The inhibitory zones are shown in Table 4. It has been observe that the L1 ligand show moderate to high antibacterial and antifungal activity than all metal complexes but less activity as compared to standard drugs. The Mn[L².2H₂O] complex shows moderate activity with inhibitory zone up to 35mm against *Staphylococcus aureus* and *Pseudomonas aeruginosa* at higher concentrations. There is less activity for L² ligand and its Mn-complex shows moderate activity against *Staphylococcus aureus* and *Pseudomonas aeruginosa* at higher concentrations. L¹ and L² and all metal complexes shows moderate to high activity *Aspergillus niger* but only L¹, Mn[L¹.2H₂O] and Mn[L².2H₂O] shows less activity against *Candida albicans*. Figure: 3 indicate the antibacterial and antifungal activity of schiff bases.



It has been suggested that the complexes having antimicrobial activity may act either by killing the microbes or by inhibiting multiplication of the microbes by blocking their active sites of enzymes [24,25]. This is also probably due to the greater lipophilic nature of the complexes. The lipid membrane that surrounds the cell favors the passage of only lipid soluble material, which controls the microbial activity. On chelation, the metal ion will be reduced largely due to the overlap of the ligand orbital and partial sharing of positive charge of the metal ion with donor groups. Further, it increases the delocalization of π -electron over the completely chelate ring and enhances the lipophilicity of the complex [26]. This lipophilicity enhances the penetration of the complex into lipid membrane and blocks the metal binding sites on enzymes of microorganisms.

4. Conclusion:

The ligands 2-(2-hydroxybenzene)-3-hydroxyiminobutane (L^1), 2-(3-hydroxybenzene)-3-hydroxyiminobutane and their Mn(II) and Fe(II) complexes had synthesized and characterized. Metal complexes show octahedral geometry by involvement of azomethine nitrogen and oxime oxygen. The ligand and metal complex were screened for their antimicrobial activity against microorganisms *S. aureus* (gm positive), *P. aeruginosa* (gm negative), *Aspergillus niger* and *Candida albicans*. Both the ligands show moderate activity against all microorganisms as compared to the standard drugs. Both the ligands and their Mn(II) complexes behave as good antimicrobial agent as compared to Fe(II) complexes..

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