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A comprehensive study on some transition metal chelating complexes

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Abstract

In the present manuscript M^{+3} & M^{+2} transition metal ions with biologically active chelating ligand 2(*o*-hydroxyphenyl) benzthiazoline were synthesized and characterized from the studies viz., ir, uv-vis. spectra, magnetic susceptibility measurement. Physico-chemical studies suggested to be distorted octahedral and square planar coordination geometry in all cases of isolated M^{+3} & M^{+2} complexes. Compounds 1-10 assayed for their *in vitro* antimicrobial activities against four bacterial strains *S. aureus*, *B. subtilis*, *E. coli* & *P. aeruginosa*, were found to be complexes are more noxious than ligand and also have significant biological properties for modification towards future drugs development as microorganism invalidate current antimicrobial agents effectiveness through many adaption and resistance. These complexes also have been found as efficient electron transfer agent in many biological processes.

Keywords: IR, UV-Vis. spectra, ligand, complexes, antimicrobial activity

Introduction

Coordination complexes of chelating schiff base ligands with several transition metal ions have been studied in recent years. Chelating ligands are an important class of ligands in coordination chemistry which are playing an important role in the pharmaceuticals as they can affect the biological activities by forming complexes with transition metal ions. These ligands acted as antibodies, cancer treatment apart from that they have also been proven effective in the neurological disorder, Parkinson's and Alzheimer's treatment with their complexing ability containing different donor atoms is also widely reported in our lab [1-4]. With this background, the preference of coordination with bidentate ligand 2 (*o*-hydroxyphenyl) benzthiazoline around M^{+3} & M^{+2} transition metal ions are reported in the present communication.

Methodology

All chemicals and reagents for this research work used were of A.R. grade. Complexes were prepared by using 1:3 / 1:2 M-L stoichiometric ratio.

Preparation of ligand: When salicylaldehyde and *o*-aminobenzthiol were taken in 1:1 molar ratio in absolute ethanol and refluxed for about 3 hrs. A condensation product of yellow colour was obtained, which was unstable and decomposed after 2 hrs of drying. Again salicylaldehyde and *o*-aminobenzthiol in 1:1 molar ratio in absolute EtOH refluxed for about 3 hrs. And obtained mixture was heated at 60 °C on magnetic stirrer for about 12 hrs. Rather it undergoes cyclization reaction. A dark yellow coloured compound was obtained, which was dried in vacuum desiccator over anhydrous P_2O_5 , compound was found to be stable.



N(2-Mercaptophenyl) Salicylamide (unstable) → 2(*o*-hydroxyphenyl) benzthiazoline (stable)

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Preparation of Titanium (III) chloride: 200 ml of titanium (III) chloride aqueous solution (containing 15% of TiCl_3) was taken and 4-5 gm. of titanium metal sponge was added to it. This mixture was cooled over a freezing mixture and dry HCl gas was passed through the solution at low temperature. Now the titanium (III) chloride was covered by a thick layer of toluene in order to avoid the oxidation. After saturation with HCl gas the solution was warmed over water bath. Titanium metal reacted with HCl gas to form TiCl_4 which was subsequently reduced to TiCl_3 by hydrogen gas produced deep blue colour solution during the reaction. The titanium (III) chloride in the solution with high concentration was kept inside a freezing mixture for 10 hrs, a dark violet coloured crystals of $\text{TiCl}_3 \cdot 6\text{H}_2\text{O}$ were obtained at the bottom of the flask.

Preparation of complexes

[2(o-hydroxyphenyl) benzthiazoline with metal Ti^{+3} , V^{+3} , Fe^{+3} , Ni^{+2} , Zn^{+2} , Cd^{+2}]: An ethanolic solution of ligand (0.01 mole) was added to the hydrated metal chloride (0.02mole) dissolved in 50 ml of warm ethanol. From the resulting solution a small amount of impure product separated. The impure excess of dilute NH_3 when flocculent neutral complexes separated. The product was filtered washed with dilute NH_4OH solution and dried in vacuum desiccator over anhydrous P_2O_5 .

[2-(o-hydroxyphenyl) benzthiazoline with metals Mn^{+2} , Cu^{+2} , Pd^{+2}]: An ethanolic solution of ligand was treated with metal acetate (chloride in case of Pd^{+2}) in stoichiometric proportion. Then mixture was stirred for about 6 hrs, the complexes separated as granular precipitate. But in case of Cu^{+2} the complex was obtained on diluting with water. The product was filtered, washed with petroleum benzene and dried in vacuum desiccator over anhydrous P_2O_5 .

Characterization of ligand and metal complexes: The colour of the ligand and its metal complexes were noted, m.p. of the ligand and its complexes were determined by open capillary method and are uncorrected ligand and subjected to analysis for C, H, N, S, whereas metal was estimated gravimetrically in the lab [5]. IR spectra of the ligand and its complexes were recorded in FT/IR-4100 type in KBr / nujol mull phase $4000\text{--}200\text{ cm}^{-1}$, IITL Labs Pvt. Ltd. Delhi. Electronic spectra of the complexes were recorded with the help of UV-Backman Du-2 spectrophotometer. Molar conductance of the complexes in different solvents viz., nitrobenzene, DMF & DMSO in 10^{-3} M dilution were observed at 27°C by Philips conductivitybridge RP-9500 with a dip type conductivity cell. Magnetic susceptibility measurement were carried out by Gouy balance using $\text{HgCo}(\text{SCN})_4$ as standard. Mass spectra of the ligand and its complexes were recorded at ToF magnetic sector and Quadrupole-SIMS instrument at New Delhi. The molecular weight determination were carried out by Rast's method using camphor as solvent [6]. The biological activities against antimicrobial and anti-fungal activities as compared to respective standard drugs using the disc diffusion methods [7].

Result and Discussion

Ligand and complexes were subjected to elemental analysis given in Table 1, which revealed that the ligand has reacted

with metals in 1:3 /1:2 (M:L_n) stoichiometric ratio. On the basis of analysis the general formula of the complexes came out to be ML_3 ($\text{M}=\text{Ti}^{+3}$, V^{+3} , Fe^{+3}) and ML_2 ($\text{M}=\text{Mn}^{+2}$, Cu^{+2} , Ni^{+2} , Zn^{+2} , Pd^{+2} , Cd^{+2}). There is a significant difference in the melting point of all complexes formed with ligand. These vast difference of melting point indicate that the complexes may have been formed. The complexes are stable in air except those of Ti^{+3} and V^{+3} are non-hygroscopic and insoluble in water. All the complexes were found to be fairly soluble in ethanol, methanol, benzene. But those of Mn^{+2} , Cu^{+2} , Pd^{+2} are sparingly soluble in ethanol and methanol, soluble in DMF, DMSO. The DMF solution of the complexes are non-conducting indicating their non-electrolytic nature. Ni^{+2} , Pd^{+2} , Zn^{+2} and Cd^{+2} complexes are diamagnetic but Ti^{+3} , V^{+3} , Fe^{+3} , Cu^{+2} and Mn^{+2} complexes are paramagnetic at room temperature. The magnetic moment values of Ti^{+3} , V^{+3} , Fe^{+3} and Mn^{+2} , Cu^{+2} complexes (1.7-5.84) B.M. is similar to six and four coordinated high spin complexes and Cu^{+2} complexes 1.88 B.M. is similar to the magnetically dilute Cu^{+2} complexes [8]. The electronic absorption spectra of Mn^{+2} , Zn^{+2} , Pd^{+2} and Cd^{+2} complexes do not display absorption band in visible region.

The Ti^{+3} complex shows magnetic moment 1.70 B.M. for one unpaired e^{-1} . The higher value may be due to orbital contribution [9]. A single broad band has been observed at 19225 cm^{-1} for Ti^{+3} complex derived from the transition ${}^2\text{T}_{2g} \rightarrow {}^2\text{E}_g$ for an octahedral geometry [9]. This band is unsymmetrical in shape is indeed made up of two closely shaped bands. The second band appears as a hump and may be due to the presence of John-Teller distortion in the complex.

The electronic spectrum of V^{+3} complex was recorded which showed band at 15995 cm^{-1} with a shoulder at 21020 cm^{-1} . The low energy band has been assigned to ${}^3\text{T}_{1g} \rightarrow {}^3\text{T}_{2g}$ whereas the high energy band to ${}^3\text{T}_{1g} \rightarrow {}^3\text{T}_{1g}(\text{P})$ transition respectively, indicates the characteristic of octahedral geometry [10]. Which is further confirmed by the absorption spectrum of 2.85 B.M. for V^{+3} complex

The magnetic moment of the Fe^{+3} complex is 5.97 B.M. corresponding to five unpaired e^{-1} 's and high spin state Fe^{+3} ion. The bands have been observed in electronic spectrum at 11230 , 21755 & 27775 cm^{-1} , corresponding to ${}^6\text{A}_{1g} \rightarrow {}^4\text{T}_{1g}$, ${}^6\text{A}_{1g} \rightarrow {}^4\text{T}_{2g}$ and ${}^6\text{A}_{1g} \rightarrow {}^4\text{E}_g$ transitions, respectively suggested octahedral geometry [11].

The absorption spectrum of Ni^{+2} complex displays medium band at 21735 ($\epsilon_{\text{max}}=65$) assignable to ${}^1\text{A}_{1g} \rightarrow {}^1\text{B}_{1g}$ transition in planar field [9, 12] and a strong absorption below 25635 cm^{-1} , probably charge transfer type Cu^{+2} complex displayed a broad band in the region $14290\text{--}13330\text{ cm}^{-1}$ and a strong assigned $\text{E}_g \rightarrow \text{T}_{2g}$ transition and later as charge transfer [12-14]. The electronic absorption spectra of Mn^{+2} , Zn^{+2} , Pd^{+2} and Cd^{+2} complexes do not display absorption band in visible region.

The ir spectrum of ligand recorded which displays a number of bands at 3233m , 3023w , 2850 , 1660m , 1575s , 1460m , 1451s , 1420 sh , 1399m , 1352m , 1307m , 1269m , 1244s , 1225w , 1182s , 1148m , 1075s , 1024s , 920s , 878m , 852w , 735vs , 737m , 715m , 637m , 575m , 546m , 510m and 458m cm^{-1} . Due to various mode of ir vibrations of different groups present in the ligand molecule. The absence of band near $2400\text{--}2650\text{ cm}^{-1}$ indicates the absence of S-H group in the ligand molecule. A medium band at 3233 cm^{-1} is assigned to $\gamma^{(\text{N-H})}$ vibration, which is shifted to lower wave number by $80\text{--}100\text{ cm}^{-1}$ indicating the bonding of ligand

through benzthiazoline ring N-H nitrogen¹⁵. A medium broad band at 2850 cm^{-1} is assigned as H-band $\gamma^{(\text{OH})}$ vibration, which disappears in the complexes. The disappearance of the $\gamma^{(\text{OH})}$ band indicates the coordination of ligand through deprotonated phenolic OH. A medium band at 1352 cm^{-1} assigned as OH bending band^[15, 16] also disappears in almost all metal complexes. The N-H bending band of the ligand located at 1575 cm^{-1} is also shifted to

lower wave number by 30-50 cm^{-1} in the metal complexes supporting the involvement of N-H nitrogen in complexes. A medium band recorded at 1460 cm^{-1} is suggested to $\gamma^{(\text{C-O})}$ vibration. The $\gamma^{(\text{C-O})}$ band shifted to higher frequencies by 40-60 cm^{-1} in complexes. The shift of $\gamma^{(\text{C-O})}$ band suggested the increase in bond order of phenolic (C-O) group on coordination^[16, 17]. Since the ligand showed a number of M-O and M-N stretching bands could not be assigned.

Table 1: Physico-chemical analysis of the ligand and complexes

Ligand / Complexes	Mol. Formula	Colour	m.p. (°C)	Elemental Analysis						(ohm ⁻¹ cm ² mol ⁻¹)			μ^{eff} (B.M.)
				%C	%H	%N	%S	%O	%M	NB	DMF	DMSO	
2(o'-hydroxyphenyl) benzthiazoline	C ₁₃ H ₁₁ NOS	Dark Yellow	138	68.00 (68.12)	5.01 (5.24)	5.42 (6.11)	6.28 (6.98)	5.97 (6.98)					
1. Tris{2(o'-hydroxyphenyl) benzthiazolinu} Titanium	(C ₃₉ H ₃₃ N ₃ O ₃ S ₃)Ti	blue green	203	62.89 (63.68)	4.10 (4.49)	5.81 (5.71)	12.11 (13.06)	6.17 (6.53)	6.90 (6.51)	1	12	14	1.70(d ¹)
2. Tris {2(o'-hydroxyphenyl) benzthiazolinu} Vanadium	(C ₃₉ H ₃₃ N ₃ O ₃ S ₃)V	Yellow green	186	62.86 (63.41)	4.03 (4.47)	5.93 (5.96)	12.59 (13.01)	6.17 (6.50)	6.23 (6.93)	2	11	15	2.8 (d ²)
3. Tris {2(o'-hydroxyphenyl)benzthiazolinu} Iron	(C ₃₉ H ₃₃ N ₃ O ₃ S ₃)Fe	Grey	157	62.85 (63.00)	3.98 (4.44)	5.15 (5.65)	12.31 (12.92)	5.87 (6.46)	5.64 (7.51)	2	10	13	5.97(d ⁵)
4. Bis{2(o'-hydroxyphenyl) benzthiazolinu} Manganese	(C ₂₆ H ₂₂ N ₂ O ₂ S ₂)Mn	Orange yellow	211	60.02 (60.82)	3.97 (4.29)	5.25 (5.46)	12.11 (12.47)	6.01 (6.24)	10.02 (10.69)	1.9	11	16	5.84(d ⁵)
5. Bis{2(o'-hydroxyphenyl) benzthiazolinu} Nickel	(C ₂₆ H ₂₂ N ₂ O ₂ S ₂)Ni	Orange	194	59.25 (60.38)	3.89 (4.26)	5.32 (5.44)	11.98 (12.38)	5.84 (6.19)	10.67 (11.39)	1.8	14	13	Dia.
6. Bis{2(o'-hydroxyphenyl) benzthiazolinu} Copper	(C ₂₆ H ₂₂ N ₂ O ₂ S ₂)Cu	Green	163	59.05 (59.82)	3.86 (4.22)	5.42 (5.39)	12.02 (12.27)	5.78 (6.14)	12.03 (12.21)	1.6	9	12	1.88(d ¹)
7. Bis{2(o'-hydroxyphenyl) benzthiazolinu} Zinc	(C ₂₆ H ₂₂ N ₂ O ₂ S ₂)Zn	Pale yellow	178	59.10 (59.61)	3.86 (4.22)	5.12 (5.34)	11.86 (12.23)	5.89 (6.11)	12.80 (12.50)	2	10	10	Dia.
8. Bis{2(o'-hydroxyphenyl) benzthiazolinu} Palladium	(C ₂₆ H ₂₂ N ₂ O ₂ S ₂)Pd	Canery yellow	109	54.79 (55.28)	3.17 (3.89)	4.98 (4.96)	11.20 (11.33)	5.33 (5.67)	19.01 (18.85)	1.8	7	10	Dia.
9. Bis{2(o'-hydroxyphenyl)benzthiazolinu} Cadmium	(C ₂₆ H ₂₂ N ₂ O ₂ S ₂)Cd	Light yellow	126	54.07 (54.69)	3.45 (3.85)	4.81 (4.91)	10.86 (11.21)	4.98 (5.61)	19.82 (20.18)	2.2	11	13	Dia.

Values in parenthesis are calculative value

Table 2: Mass spectra data of ligand and its metal complexes

Compounds	Calculated Mass	Observed Mass	Peak
2(o'-hydroxyphenyl) benzthiazoline	229	228.69	M
TiL ₃	734.9	735	M
VL ₃	737.9	738	M
FeL ₃	742.8	744.6	M+1
MnL ₂	512.94	512.4	M
NiL ₂	516.69	517.2	M
CuL ₂	521.5	522	M
ZnL ₂	523.38	523.5	M
PdL ₂	564.42	565.01	M
CdL ₂	570.41	571	M

The mass spectra data of ligand exhibit some peaks illustrate the power fragmentation in most cases instance molecular ion peaks were recorded. The mass spectra data of the ligand and its complexes are given in table-2 which were in good agreement for expected values^[11, 18]. The mass spectra of the ligand gives some peaks at 228.69 m/z due to the loss of e⁻¹ C₁₃H₁₀NOS¹⁺ which assigned for molecular ion peak at H/L⁺ 202m/z is attributed to the presence of base peak

C₁₂H₁₂NS¹⁺ at 100% relative abundance. Ti(III), V(III), Fe(III), Mn(II), Ni(II), Cu(II), Zn(II), Pd(II) and Cd(II) complexes give some peaks at 735.2 m/z, 738 m/z, 744.6 m/z, 512.4 m/z, 517.2 m/z, 522 m/z, 523.5 m/z, 565.01 m/z and 571 m/z, respectively which assign as M⁺ peak in all cases but in the spectra of only one Fe(III) complex this peak was at 744.6 m/z which assigned to M+1 peak.

Table 3: Antimicrobial activity of the ligand and its metal complexes, Gram (+) bacteria Gram (-) bacteria (Microorganism Inhibition Zone in mm)

Compounds	<i>S. aureus</i>	<i>B.subtilis</i>	<i>E.coli</i>	<i>P. aeruginosa</i>
2(o'-hydroxyphenyl) benzthiazoline	10	11	-	-
TiL ₃	24		24	23
VL ₃	-	-	21	22
FeL ₃	23		23	22
MnL ₂	25		24	24
NiL ₂	20	20	-	-
CuL ₂	21	20	-	-
ZnL ₂	15	15	15	15
PdL ₂	26	-	27	27
CdL ₂	25	24	-	-

This M+1 peak was recorded along with the molecular ion peak due to the presence of isotope of Fe-atom in the complex [18, 19]. The mass spectra proved that the isotope pattern of detected complex corresponds to the characteristic isotopic pattern ^{54}Fe 18.5% and table-2 present the assumed composition and complexes of ^{54}Fe isotope.

Ligand and its chelates were studied by the usual cup-plate agar diffusion technique against *S. aureus*, *B. subtilis* gram (+) and *E. coli*, *P. aeruginosa* as gram (+) bacteria (1.0 w/v) solution of the compounds were prepared. A mixture of equal volumes of isopropyl alcohol and dioxan was taken and its 20% solution was used as solvent. Four holes (about 5 mm diam.) were cut in the agar medium enriched culture and 0.1 ml of 1.0% solution of the compound were put in these holes. The Petri dishes were allowed to remain in the refrigerator at 4-6 °C for about 4 hrs. To allow the diffusion of the solution [7, 20]. These Petri dishes were then transferred to an incubator maintained at 35°C and the zone of inhibition were noted and data shown in table-3 and Figure A as well. compound 1 showed a little activity towards *S. aureus* & *B. subtilis* and no activity towards *E. coli* & *P. aeruginosa*, while compound no. 8 showed activity against both the gram + & -organism. Compound no. 3 showed no activity against *S. aureus* & *B. subtilis*, while compound no. 6, 7 and 10 showed no activity against *E. coli* & *P.*

aeruginosa but were active against both the gram (+) organism [10, 20]. We can thus, in general say that antibacterial activity is enhanced on complexation of the ligand with metals. The metal complexes showed variation in activity towards different strains because of their impermeability into the strain cell. The inhibition particularly depends upon the concentration of the strains taken in culture medium of incubation conditions rate of diffusion [11, 20]. The antimicrobial activity of the synthesized compounds might be explained on the basis of the overtone concept and chelation theory. Accordingly polarity of metal atom get reduced due to sharing of the partial (+) charge of metal ion to the different donor atom of the compounds and some p-electrons delocalization inside the entire chelates. Some metal complexes do not show good activity because of their limited lipid solubility, therefore, metal ion present on center may not be able to attack the required site of cell wall to interfere with the normal cell activity. Although chelation enhances the biological activity of the complexes along with other factors like dipole moment, geometry of the complexes, concentration, size of the molecules, bond length, coordination site and hydrophobicity effect activity. Hence, it may be concluded that not only chelation but may other contribution also enhance the antimicrobial activity [13, 20].

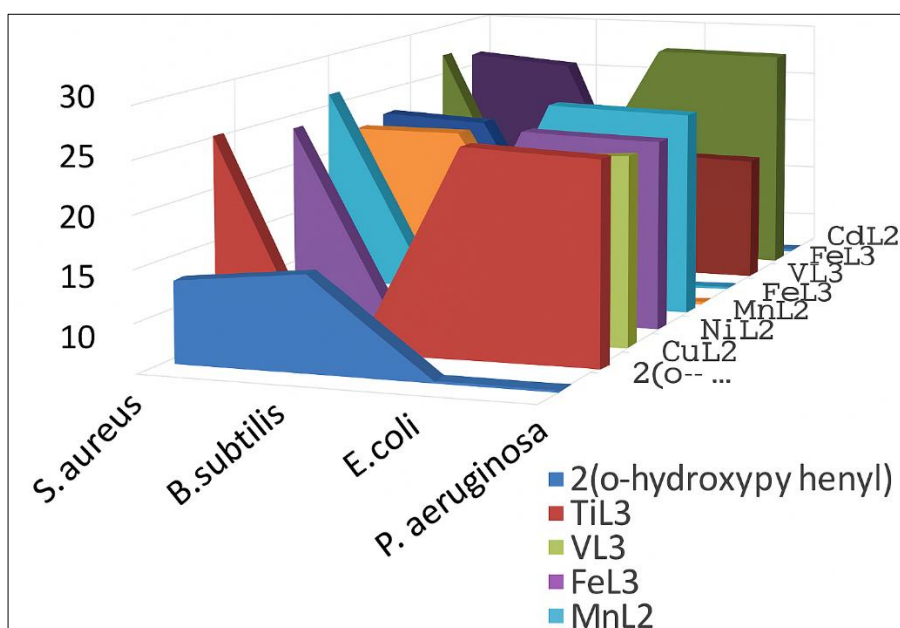


Fig 1: Showing antimicrobial studies of compounds

Conclusion

Trivalent iron metal complexes with biologically active ligand were synthesized in stoichiometric proportion, the ligand functions as anionic chelating ligand through deprotonated O, N, atoms. The ligand field parameters of the synthesized complexes have been evaluated which suggested to be distorted octahedral and square planar geometry. Antimicrobial activities of free ligand and its metal complexes were measured and data prove that an enhancement in antimicrobial activity was found in its metal complexes as compare to ligand. The molecular docking of the ligand and its complex with enzyme sterol 1, 4- α Demethylase suggested the hydrophobic binding. Furthermore in the silico stressed that the compounds may be used as orally active drugs.

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