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Synthesis, Characterization, and Antibacterial Activity of the Schiff Base Derived from P-Toluic Hydrazide and o-Vanilin (OVPTH Ligand) and its Mn (II), Co (II), Ni (II) and Cu (II) Complexes

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ABSTRACT

Metal complexes of a novel Schiff base derived from condensation of p-toluic hydrazide and o-Vanilin are reported and characterized based on elemental analyses, FT-IR, ¹H-NMR, UV-Visible, VSM, Molar conductance, ESR Spectroscopy, Powdwe XRD, micro analytical data and also found to be an anti bacterial activity. The magnetic properties of these Mn (II), Co (II), Ni (II) and Cu (II) complexes are 5.20 B.M. 4.93 B.M., 3.2 B.M and 1.63 B.M respectively. This result clearly indicates that the metal complexes of this ligand show octahedral geometry. The IR spectrums of these Schiff base metal complexes shows bands at 1603 cm⁻¹, 1597 cm⁻¹, 1598 cm⁻¹, and 1591 cm⁻¹ respectively, which is assigned to (C=N) stretching vibrations, a fundamental feature of azomethine group. The anti bacterial results also indicate that the metal complexes are better antibacterial agents as compared to the Schiff base. All the metal chelates are found to be nonelectrolytes.

Key words: Schiff Base, p-Toluic Hydrazide, o-Vanilin, Anti Bacterial Activity, Characterization.

1. INTRODUCTION

There is a continuing interest in transition metal complexes of Schiff bases because of the presence of both nitrogen and oxygen donor atoms in the backbones of these ligands, some of these complexes have been exhibit interesting physical and chemical properties and potentially useful biological activities. Schiff base have also been used for analytical purposes in the determination of metal ions, and some Schiff base derivatives [1-2] have been used in the extraction of metal solvents. The applications of such complexes depend to a large extent on their molecular structure. Schiff base ligands are able to coordinate many different metals, and to stabilize them in various oxidation states [2-4]. The Schiff base complexes have been used in catalytic reactions and as models for biological systems. During the past two decades, considerable attention has been paid to the chemistry of the metal complexes of Schiff bases containing nitrogen and other donors. This may be attributed to their stability, biological activity [5-6] and potential applications in many fields such as oxidation catalysis, and electrochemistry. Schiff base ligands have an affinity for transition metals such as Cu. Mn. Co. Ni and Fe. Some of these complexes have been studied in great deal for their various structures, steric effects and their coordination [7] chemistry.

*Corresponding Author: Email:kondareddyseku@gmail.com The present paper deals with a new series of metal complexes of Mn (II), Co (II), Ni (II) and Cu (II) with Schiff base ligand (OVPTH) derived from P-Toluic hydrazide and o-Vanilin. These complexes were characterized by elemental analysis, IR, NMR, UV, TG-DTA, and conductivity measurements to determine the mode of bonding, geometry and biological activities of the metal complexes were also studied.

2. EXPERIMENTAL

2.1 Synthesis of p-toluic hydrazide and o-vanilin Schiff base (OVPTH)

Mixture of p-toluic hydrazide (0.01moles) and o-Vanilin (0.01moles) were dissolved in 50 ml methanol and added few drops of Tri ethyl amine. The whole mixture was transferred in to 250 ml round bottom flask. The mixture was refluxed about one and half hour on water bath. When the reaction mixture was allowed to cool light yellow crystals were obtained. The compound was recrystalized from methanol. The %of yield was 85% and melting point of the compound was 180-182 °C. The reagent p-toluic hydrazide and o-Vanilin Schiff base was dissolved in 40ml of 50% methanol and Mn (II), Co (II), Ni (II) and Cu (II) metal ions dissolved in 10ml of distilled water, this compounds mixture was stirred for 6 hours. A



colored precipitate of complex was obtained with good yield. These products were washed several times with hot water and cold methanol to remove unreacted ligand and metal slats respectively. Finally, all these complexes were dried in vacuo over calcium chloride desiccator.

3. RESULTS AND DISCUSSION *3.1. IR & NMR Spectral Studies*

IR & NMR data gives further useful information on the structure OVPTH ligand and its metal complexes. IR & NMR spectrum of the free ligand was compared with the spectrum of the metal complexes. The structurally significant IR & NMR Spectral data of free OVPTH ligand and its metal complexes have been reported in table 1&2. The IR & NMR spectras are shown in Fig 1-3.

In NMR spectrum a signal observed at 11.07ppm due to N-H proton in the ligand is shifted to δ 11.25, 11.31ppm respectively for OVPTH-Co and OVPTH-Cu metal complexes. A broad band exhibited at 3330 cm⁻¹ in the IR spectrum of the ligand is due to N-H stretching vibration. On complexation [8-9] this band shifted to 3312, 3297. $3310 \text{ and } 3303 \text{cm}^{-1}$ for Mn (II), Co (II), Ni (II) and Cu (II) complexes respectively. The IR spectrum of the ligand has shown a sharp band at 1711 cm⁻¹ due to C=O stretching vibration. On complexation this band shifted to 1676, 1678, 1665 and 1670 cm⁻¹ for Mn (II), Co (II), Ni (II) and Cu (II) Complexes respectively. These results clearly indicate the formation of complexes. The IR spectra of Mn (II), Co (II), Ni (II) and Cu (II) complexes exhibited broad bands at 3453 cm⁻¹, 3460 cm⁻¹, 3420 cm⁻¹ and 3422 cm⁻¹ respectively which can be assigned to the OH stretching vibration of the coordinated water molecules [10-11]. These results indicate that the ligand coordinates with the metal ion through the azomethine nitrogen and the oxygen of the deprotonated hydroxyl group.

3.2. UV Spectral studies & Magnetic susceptibility measurements of OVPTH complexes

By UV spectral studies the transition for the ligand occurred at 303 nm. But on complexation with the different metal ions like Mn (II), Co (II), Ni (II) and Cu (II) new bands appeared at 390 nm, 376 373 399 nm and nm respectively nm, corresponding to the transitional charge transfer from the ligand to the different metal ions. Bands occurred in the region of 370-395 nm for all complexes are assigned to charge transfer transition $(L \rightarrow M)$. Based on the results octahedral structure is proposed [12-13] for Mn^{+2} , Co^{+2} , Ni^{+2} and Cu^{+2}

complexes. The magnetic moments of the present OVPTH Mn (II), Co (II), Ni (II) and Cu (II) Complexes [14] are 5.20, 4.93, 3.2 and 1.63BM. The magnetic moments of this ranges are suggest octahedral geometry for Mn (II), Co (II), Ni (II) and Cu (II) complexes.

3.3. Thermal behavior of Mn (II), Co (II), Ni (II) and Cu (II) Metal complexes of OVPTH

The Thermo gravimetric studies of all the complexes were carried out in air at a heating rate of 10 °C per minute. The thermo analytical data is summarized in Table.3. The thermal decomposition of the complexes proceeds in three stages. The Mn (II), Co (II), Ni (II) and Cu (II) complexes are thermally stable up to 135.32, 130.49, 110.59 and 140 °C respectively. The first of decomposition corresponding stage to endothermic dehydration of complexes by the loss of two water molecules occurs in the temperature range 135-280 °C, 132-233 °C, 110-257 °C and 140-276 °C respectively [15-16]. The intermediates formed are stable up to 310, 313, 284 and 309 °C. The second decomposition with exothermic peak by the loss of ligand moiety occurs in the temperature range 310-553 °C, 313-482 °C, 284-499 °C and 309-592 °C. The solid residues above 611, 520, 560 and 716 °C were identified as Mn, Co, Ni and Cu metal oxides respectively. In all the complexes, the final products are metal oxides.

3.4. ESR Spectral studies of OVPTH-Cu complex

Anisotropic spectra obtained for all complexes in DMF at LNT and representative ESR spectra of RAPPTH-Cu (II) ion complex is presented in Fig.5. In this low temperature spectrum, four peaks of small intensity have been identified which [17-18] are considered to originate from g₁ component. The G value of present complex is greater than four and suggests that there are no interactions between Copper-Copper centers in DMF medium. The ESR parameters g_{l} , $g_{\perp} A_{l}^{*}$ and A_{\perp}^{*} of the complexes and the energies of d-d transitions are used to evaluate the orbital reduction parameters (K₁, K_{\perp}), the bonding parameters (α^2), the dipolar interaction (P). The observed $K_1 < K_{\perp}$ indicates the presence of out of plane π -bonding. The α^2 value for the present chelate is 0.574. It indicates that the complex has covalent character [19]. This shows an appreciable covalency in the inplane σ bonding. Girdano and Bereman suggested the identification of bonding groups from the values of dipolar term P. The reduction of P values from the free ion value (0.036 cm⁻¹) might be attributable to the strong covalent bonding. The value of P obtained for the present complex is 0.017 cm⁻¹ and remain consistent with bonding of Copper ions to oxygen and nitrogen donor atoms respectively. The shape of ESR lines, ESR data together with the electronic spectral data

Compound	OH Water	OH Phenolic	C=N	N-H	C=O	М-О	M-N
OVPTH	-	3215	1645	3330	1711	-	-
OVPTH-Mn	3453	-	1603	3312	1676	600	462
OVPTH-Co	3460	-	1597	3297	1678	597	460
OVPTH-Ni	3420	-	1598	3301	1665	598	465
OVPTH-Cu	3422	-	1591	3303	1670	623	472

Table 1. Important IR Spectral bands of OVPTH ligand and its metal complexes.

Table 2. ¹H NMR spectral data of OVPTH ligand and its metal complexes.

		0				
Compound	H-C=N	Ar-H	CH ₃	OCH ₃	Ar-OH	N-H
OVPTH	8.69	6.80-7.86	2.4	3.81	12.04	11.07
OVPTH-Co	8.93	7.01-8.03	2.46	4.02	-	11.25
OVPTH-Cu	8.90	6.82-7.91	2.5	3.82	-	11.31

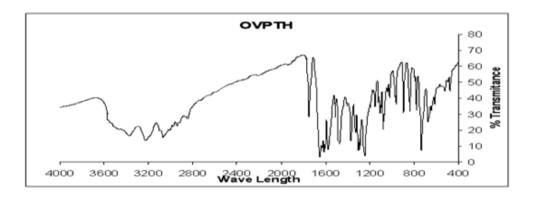


Figure 1. IR spectrum of OVPTH ligand.

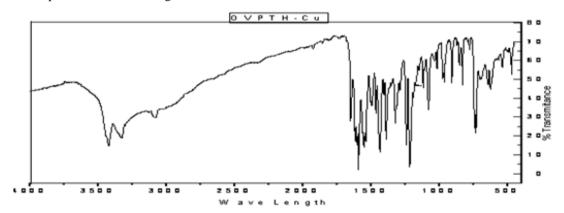


Figure 2. IR spectrum of OVPTH-Cu (II) metal complex.

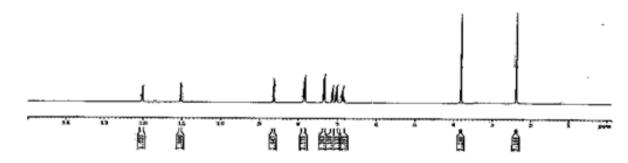


Figure 3. H¹-NMR spectrum of OVPTH ligand.

Complex X=H ₂ O	Molecular Weight in grams	Weight of the complex taken in mgs	e Temperature lex Stage range inºC in		Probable assignment	Mass loss (%)	Total mass loss (%)
$\begin{array}{c} Mn \ L_2 \\ 2X \\ L=C_{16} \\ H_{16} \ O_3N_2 \end{array}$	659.586	7.50	1 2 3	135.32 to 280.15 310.53 to 553.69 Above 611.25	Loss of 2H ₂ O molecules Loss of 2L molecules Corresponds to MnO	5.462 83.241 11.106	88.703
$\begin{array}{c} Co \ L_2 \ 2X \\ L=C_{16} \\ H_{16} \ O_3N_2 \end{array}$	663.576	8.00	1 2 3	130.49 to 233.12 313.89 to 482.14 Above 520.34	Loss of 2H ₂ O molecules Loss of 2L molecules Corresponds to CoO	5.429 79.014 14.321	84.443
$Ni L_2 2X \\ L=C_{16} \\ H_{16} O_3 N_2$	663.285	7.50	1 2 3	110.59 to 257.27 284.97 to 499.47 Above 560.19	Loss of 2H ₂ O molecules Loss of 2L molecules Corresponds to NiO	5.427 88.124 5.698	93.557
$\begin{array}{c} Cu \ L_2 \ 2X \\ L=C_{16} \\ H_{16} \ O_3N_2 \end{array}$	668.102	8.00	1 2 3	140 to 276.54 309.06 to 592.12 Above 716.18	Loss of 2H ₂ O molecules Loss of 2L molecules Corresponds to CuO	5.389 82.210 12.301	87.5

Table 3. Thermal Analytical Data of the OVPTH Ligand metal complexes.

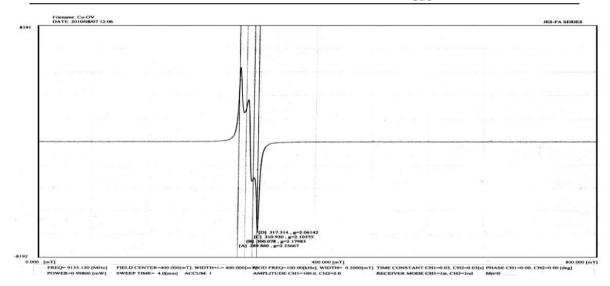


Figure 5. ESR spectrum of OVPTH-Cu (II) metal complex.

suggest an octahedral geometry for OVPTH-Cu complex.

3.5. Molar Conductivity

The molar conductance data of complexes in DMF at 10^{-3} are presented in Table 5. The molar conductance values are 4.40, 5.40, 7.00 and 5.20 ohm⁻¹ cm² mol⁻¹ respectively for OVPTH-Mn (II), OVPTH-Co (II), OVPTH-Ni (II) and OVPTH-Cu (II). The complexes are found to be non electrolyte in DMF solution.

3.6. Powder XRD study of OVPTH-Cu Complex

The powder X-ray diffraction data obtained for OVPTH-Cu complex with difractograms using DROL-2 powder diffractometer. Radiation filled by metal foil. The difractograms (15 diffractions) reflects Fig. 6. between 5-75 (2 θ) values for Cu complex. Where θ is Brages angle all the main peaks are indicted and calculated values of Miller indices (h k l) along with observed d-specified and reveled intensities are specified in the Fig 3.8a. All the peaks have been indexed 2 θ values compared in graph. Comparison values revels that there is good

Table	4.	ESR	spectral	data	of	OVPTH-Cu
	(

complex.	
Parameters	OVPTH-Cu
g	2.21825
g⊥	2.05258
gave	2.10780
G	4.15081
A∥ [*]	0.01862
$A_{\perp}^{"*}$	0.00434
A_{ave}^{*}	0.0091
d-d	16432
K	0.614
$\mathbf{K}_{\perp}^{"}$	0.753
\mathbf{P}^*	0.017
α^2	0.574

agreement between values of 20 and d-values. The powder x-ray diffraction data showed identical features with very poor crystalinity [20]. The patterns are qualitative and dispersive in intensity of Cu complex. The XRD patterns are used to explain qualitatively the degree of crystalinity. Xray diffraction data of OVPTH-Cu complex are presented in table.5.

3.7. Anti bacterial activity

The biological activity of the Schiff base ligand and their metal complexes and streptomycin (as a standard compound) were tested against bacteria because bacteria can achieve resistance to antibiotics through biochemical and morphological modifications. The organisms used in the present investigations included Staphylococcus aureus and Bacillus subtillis (as gram positive bacteria) and Pseudomonas aereuguinosa and Escherichia coli (as gram negative bacteria). The diffusion agar technique was used to evaluate the antibacterial activity of synthesized ligand and its metal complexes [21-22]. The results of the bacterial screening of the synthesized compounds are recorded in Table 7. The data obtained reflect that the Schiff base ligand shows moderate activity in comparison with Staphylococcus aureus, Bacillus

Table.5. X-ray Diffraction data of OVPTH-Cu complex.

subtillis and less active in comparison with *Pseudomonas aereuguinosa, Escherichia coli.* Antibacterial activity of all the complexes at low concentration towards gram positive and negative bacteria is not detected or low. The activity of the Schiff base ligand and its metal complexes increases as the concentration increases because it is well known [23-24] fact that concentration plays a vital role in increasing the degree of inhibition. The antibacterial activity results indicate that most of the metal chelates exhibited good to moderate antibacterial activity when compared to Ligand. Among the synthesized metal complexes OVPTH-Cu showed high activity.

4. CONCLUSIONS

The design and synthesis of a new family of bidentate Schiff base ligands for use in octahedral complexes have been successfully demonistrated. In this paper, the coordination chemistry of a Schiff base ligand obtained from the reaction of p-toulic hydrazide and o-vanilin is described Mn (II), Co (II), Ni (II), and Cu (II) complexes have been characterized by spectral and analytical data. The IR&electronic transition data lead to the conclusion that the Mn (II), Co (II), Ni (II), and Cu (II) metal complexes are octahedral in nature and hence the structure of OVPTH Schiff base metal complexes are given in Fig. 7. In all the complexes, the ligand acts as bidentate. The results of in-vitro biocidal activities of the ligand and its metal complexes clearly show antibacterial activity against the tested organisms. On the basis of chelation theory, metal complexes have more biological activity than free ligand. All the metal chelates are found to be nonelectrolytes. The present work points to the future work that OVPTH can be exploited as precursor for the synthesis of new complexes and it would be fruitful to obtain complexes with promising antibacterial activity. Further coordination chemistry needs to explore in these chemically rich systems.

S.No.	d expt	dCalc	2θ expt	2θ Calc	h k l
1	12.7403	12.7396	6.9235	6.9212	311
2	10.7611	10.7425	8.2492	8.2361	320
3	9.4974	9.4862	9.1502	9.1211	321
4	8.8618	8.8439	9.9831	9.9736	330
5	6.2993	6.2872	13.9864	13.9647	442
6	5.6436	5.6326	15.6804	15.6719	542
7	5.0422	5.0215	17.4826	17.3929	642
8	4.1829	4.0949	21.1935	21.1736	753
9	4.0017	3.9976	22.3570	22.3467	761
10	3.8259	3.8158	23.5902	23.4997	763
11	3.3973	3.3859	26.3826	26.3615	874
12	3.1667	3.1548	28.1968	28.1920	965
13	3.0507	3.0217	29.3102	29.3052	982
14	2.9033	2.8926	30.7962	30.6989	1155
15	2.7399	2.7147	32.8149	32.8059	1090

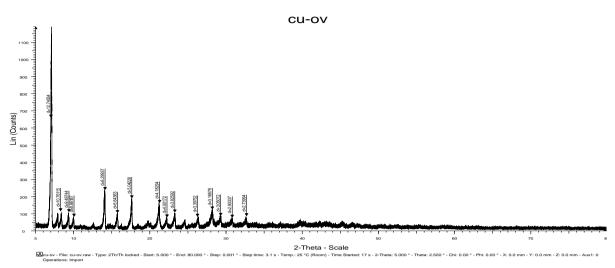


Figure 6. XRD spectrum of OVPTH-Cu (II) metal complex

Molecul	Molecul			Meltin	Elemental analysis								
ar Formul	ar Weight	Colou		g Point in ⁰ C	Carbon %		Hydrogen %		Nitrogen %		Metal %		
a X=H ₂ O	,, eight				Calc.	Foun d	Cal c.	Foun d	Calc.	Found	Cal c.	Foun d	
$\begin{array}{c} L = \!$	285.33	Light yello w	85	180- 182	67.29	66.78	5.60	5.35	9.81	9.52	-	-	
[Mn. L _{2.} 2X]	659.586	Light Yello w	72	310- 340	58.21	57.52	5.15	4.91	8.49	8.23	8.84	8.54	
[Co.L ₂ .2 X]	663.576	Orang e yello w	68	290- 310	57.86	57.34	5.12	4.75	8.44	8.12	8.88	8.02	
[Ni. L _{2.} 2X]	663.285	Green	86	305- 325	57.89	57.23	5.13	4.93	8.44	7.91	8.33	7.92	
[Cu.L ₂ .2 X]	668.102	Green	75	300- 330	57.49	57.21	5.09	4.89	8.38	8.06	9.49	9.13	

Table 6. Analytical data of the OVPTH ligand and its metal complexes

Table 6. Antibacterial Activity of OVPTH Schiff base Ligand and its metal complexes. Compound Cram positive

Compound	Gram positive						Gramnegative						
	Staphylococcus aureus		Bacillus subtillis			Pseudomonas aereuguinosa			Escherichia coli				
L=C ₁₆ H ₁₆ O ₃ N _{2.} (OVPTH)	5	2.5	1	5	2.5	1	5	2.5	1	5	2.5	1	
[Mn L ₂]2X. (OVPTH-Mn)	++	+	nd	++	+ +	+	+	+	nd	++	+	+	
[Co L2]2X.(OVPTH-Co)	++	++	+	++	++	nd	++	++	+	++	+	+	
[Ni L ₂]2X.(OVPTH-Ni)	++	++	nd	++	+	+	++	++	+	++	nd	nd	
[Cu L _{2.}]2X. (OVPTH-Cu)	++	+ +	+	++	+	nd	++	++	nd	++	+	+	
Streptomycin(standard)	++	++	+	++ +	++	+	++	++	+	++	++	+	
Sucpromyciii(standard)	++	++	++	+++	++	++	++	++	++	++	++	+ +	

Inhibition values = 0.1 - 0.5 cm beyond control = + (less active); inhibition values = 0.6 - 1.0 cm beyond control = ++ (moderate active); inhibition values = 1.1 - 1.5 cm beyond control = +++ (high active). Concentration in mg/ml. nd:non detected

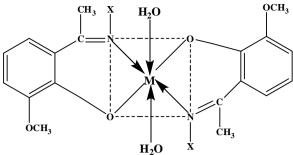


Figure.7. OVPTH metal complexes $(M=Cu^{+2}, Co^{+2}, Ni^{+2}, Mn^{+2})$.

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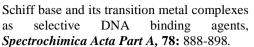
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