SYNTHESIS OF (E)-4-METHYL-2-((PHENETHYLIMINO (PHENYL)METHYL)PHENOL AND ITS TRANSITION METAL COMPLEXES, CHARACTERIZATION AND ELECTRICAL CONDUCTIVITY STUDY OF COMPLEXES

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ABSTRACT

A novel Schiff base (E)-4-methyl-2-((phenethylimino(phenyl)methyl)phenol synthesized by the condensation of 2-hydroxy-5-methylbenzophenone with 2-phenylethylamine. A series of metal complexes of Mn(II), Co(II), Ni(II), Cu(II), Zn(II) and Cd(II) have been prepared with the newly synthesized Schiff base ligand. The synthesized ligand was characterized by elemental analysis, FT-IR and ¹H NMR spectra. The complexes have been characterized by different physicochemical techniques. The d.c. electrical conductivity of the synthesized complexes has been measured in compressed pellet form over a wide range of temperatures. The complexes were found to be semiconducting.

Keywords: Schiff Base, FTIR, Diffuse Reflectance, Electrical Conductivity, Transition Metal Complexes, Metal Chelates.

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INTRODUCTION

The Schiff bases have a very important role in coordination chemistry. Schiff base metal complexes have attracted great interest in the research field due to novel structural features, interesting thermal, magnetic and spectral properties, biological, industrial and catalytic importance.¹⁻⁸ Many complexes of Schiff base were found to be a very important precursor of semiconducting materials.^{9,10} In the recent past it was reported that the metal complexes of N, O-chelating Schiff base ligands have unusual thermal and electrical stabilities.^{11,12} In our previous publication synthesis and characterization of (E)-4-methyl-2-((phenethylimino)(phenyl)methyl) phenol and its Mn(II), Co(II), Ni(II), Cu(II), Zn(II) and Cd(II) complexes have been reported.¹³ The spectral analysis and d.c. electrical conductivity study of these metal complexes has been reported in the present paper.

EXPERIMENTAL

Material and Methods

The solvents and chemicals used in the synthesis were of AR grade and if required, further purified by standard methods.^{14,15}

General Procedure

(a) Preparation of Schiff Base (HMBPE)

Schiff base (E)-4-methyl-2-((phenethylimino)(phenyl)methyl)phenol was prepared by condensation reaction between 2-phenylethylamine and 2-hydroxy-5-methylbenzophenone in ethanol as a solvent.^{16,17}

Fig.-1: Proposed Structure of Ligand (HMBPE)

(b) Preparation of Transition Metal Complexes of HMBPE

A solution of metal(II) acetate was mixed with a solution of ligand HMBPE in DMF in 1:2 (M:L) ratio.¹⁸ The reaction mixtures were refluxed for 5-6 hours on a sand bath using a water condenser. The colored products obtained were filtered and washed several times with petroleum ether.¹⁹ Finally, the products were dried by using desiccators over anhydrous AlCl_3 , then in an electric oven at 60–70°C.²⁰

Detection Method

^1H NMR spectrum of HMBPE was recorded in CDCl_3 at SAIF, CDRI, Lucknow. FT-IR spectra of all synthesized compounds were recorded in the IR Affinity-1 Shimadzu instrument using KBr as a reference in the range 400–4000 cm^{-1} . Diffuse reflectance spectra (DRS) were recorded at STIC, Cochin University, Kerala on Varian Cary-5E UV-visible spectrophotometer.

Analytical Discussion

(a) Elemental Analysis of HMBPE: The elemental analysis suggested $\text{C}_{22}\text{H}_{21}\text{NO}$ empirical formula for the ligand.

(b) The FT-IR spectrum of HMBPE: FT-IR spectrum of HMBPE shows the peaks at 3664 cm^{-1} (due to phenolic O–H stretching), 1608 cm^{-1} (C=N stretching), 1325 cm^{-1} (C–O phenolic stretching).

(c) ^1H NMR Spectra of HMBPE (300 MHz, CdCl_3 , δ in ppm): ^1H NMR spectrum of ligand shows following peaks²¹:

δ 7.220 – 7.441 (5H, m, Ar-H); δ 7.212 (1H, s, Ar-H); δ 6.919 – 7.191 (5H, m, Ar-H); δ 6.876 – 6.904 (1H, d, Ar-H); δ 6.484 – 6.490 (1H, d, Ar-H); δ 5.542 (1H, s, (broad) -OH); δ 3.547 – 3.595 (2H, t, - CH_2 -); δ 2.891 – 2.990 (2H, t, - CH_2 -); δ 2.080 (3H, s, Ar- CH_3).²²⁻²⁵

RESULTS AND DISCUSSION

The compounds synthesized and characterized by FT-IR, elemental analysis, ^1H NMR and diffuse reflectance spectra. The physicochemical and spectral data reveals the formation of the complex through donor site azomethine N-atom and phenolic O-atom. The d.c. electrical conductivity study of synthesized complexes has also been carried out.

(1) Elemental Analysis of Compounds:

The physical and analytical data of compounds are shown in Table-1. Elemental analysis data confirms the stoichiometric composition of the compounds. The data suggest a 1:2 (M:L) ratio in all the complexes. It also indicates the presence of coordinated and lattice water molecules in the complexes.²⁶ All the compounds were found to be nonelectrolytes.²⁷

(2) FT-IR Spectra

FT-IR spectral data of HMBPE and its metal complexes are given in Table-2. The spectrum of free HMBPE shows broad band at 3665 cm^{-1} assigned to phenolic O–H stretching. This band was not observed in the spectra of its metal complexes. Another strong absorption band observed at 1628 cm^{-1} due

to C=N stretching which is shifted to lower frequencies in the spectra of complexes by 26-46 cm^{-1} suggesting coordination through azomethine nitrogen.²⁹

Table-1: Physical and Elemental Analysis Data of Compounds²⁸

S. No.	Complex	Colour	Solubility	Mol. Wt.	Elemental Analysis % Found (Calcd.)			
					M	C	H	N
1.	HMBPE	Pale Yellow	DMF	315.4	--	83.80 (83.78)	6.66 (6.71)	4.76 (4.44)
2.	[Mn(HMBPE) ₂ (H ₂ O) ₂]	Light Brown	DMSO	719.7	7.72 (7.63)	73.33 (73.42)	6.24 (6.16)	3.81 (3.89)
3.	[Co(HMBPE) ₂ (H ₂ O) ₂].H ₂ O	Light Pink	DMSO	741.7	7.89 (7.94)	71.29 (71.24)	6.21 (6.25)	3.72 (3.78)
4.	[Ni(HMBPE) ₂ (H ₂ O) ₂].2H ₂ O	Light Green	DMSO	759.5	7.76 (7.73)	69.68 (69.58)	6.43 (6.37)	7.68 (7.73)
5.	[Cu(HMBPE) ₂].H ₂ O	Olive Green	DMSO	710.3	8.87 (8.95)	74.46 (74.39)	5.88 (5.96)	3.98 (3.94)
6.	[Zn(HMBPE) ₂ (H ₂ O) ₂].H ₂ O	Light Gray	DMSO	748.2	8.79 (8.74)	70.57 (70.63)	6.26 (6.20)	3.65 (3.74)
7.	[Cd(HMBPE) ₂]	Light Yellow	DMSO	741.2	15.11 (15.17)	71.27 (71.30)	5.38 (5.44)	3.69 (3.78)

The medium intensity band observed at 1325 cm^{-1} due to phenolic C-O stretching is shifted to lower frequencies in the complexes indicates coordination through deprotonated phenolic oxygen atom.³⁰⁻³² The appearance of new bands in complexes in the range of 529-584 cm^{-1} and 468-496 cm^{-1} has been assigned M-O and M-N stretching.^{33,34} The bands in the range of 3367-3445 cm^{-1} in complexes are due to $\nu(\text{H}_2\text{O})$ suggesting hydrated complexes.³⁵ The medium intensity sharp bands the range 1507-1531 cm^{-1} and 806-865 cm^{-1} suggesting coordinated water molecules in some complexes.^{36,37}

Table-2: FT-IR Spectral Data of Compounds (cm^{-1})

S. No.	Compound	$\nu(\text{O-H})$	$\nu(\text{C=N})$	$\nu(\text{C-O})$	$\nu(\text{M-O})$	$\nu(\text{M-N})$	$\nu(\text{H}_2\text{O})$
1.	HMBPE	3664	1628	1325	--	--	--
2.	[Mn(HMBPE) ₂ (H ₂ O) ₂]	--	1602	1296	532	479	3418, 1531, 832
3.	[Co(HMBPE) ₂ (H ₂ O) ₂].H ₂ O	--	1582	1305	569	496	3395, 1513, 865
4.	[Ni(HMBPE) ₂ (H ₂ O) ₂].2H ₂ O	--	1597	1285	548	485	3367, 1507, 806
5.	[Cu(HMBPE) ₂].H ₂ O	--	1593	1272	556	468	3435
6.	[Zn(HMBPE) ₂ (H ₂ O) ₂].H ₂ O	--	1589	1319	529	481	3445, 1530, 845
7.	[Cd(HMBPE) ₂]	--	1586	1316	584	472	--

(3) Diffuse Reflectance Spectra and d.c. Electrical Conductivity

The diffuse reflectance spectrum of HMBPE complexes of Mn(II), Co(II) and Ni(II) shows three bands.³⁸ These three bands are consistent with octahedral geometry.³⁹⁻⁴³ The absorption bands of Cu(II) complex corresponds to square planar geometry.^{44,45} The Zn(II) and Cd(II) complexes of HMBPE show no d-d transitions. Thus, their geometry cannot be predicted based on diffuse reflectance spectra.⁴⁶ Hence, based on the physicochemical data Zn(II) complex assigned octahedral and Cd(II) complex assigned tetrahedral geometry.⁴⁷ The electrical conductivity and diffuse reflectance data are given in Table-3.

The d.c. electrical conductivity measurements of the complexes were carried out in compressed pellet form at temperature range 303-393 K. The conducting behavior of complexes as a function of temperature was studied using two probe technique.⁴⁸

Table-3: Electrical Conductivity at 373 K and Diffuse Reflectance Data of the Complexes of HMBPE

S. No.	Metal Complexes	d.c. Electrical Conductivity		Diffuse Reflectance	
		Σ ($\Omega^{-1}\text{cm}^{-1}$)	E _a (eV)	Abs. Band (cm^{-1})	Assignments
1.	[Mn(HMBPE) ₂ (H ₂ O) ₂]	3.15×10^{-9}	0.139	17578 23035 25053	${}^6A_{1g} \rightarrow {}^4T_{1g}({}^4G)$ ${}^6A_{1g} \rightarrow {}^4T_{2g}({}^4G)$ ${}^6A_{1g} \rightarrow {}^4E_g$
2.	[Co(HMBPE) ₂ (H ₂ O) ₂].H ₂ O	9.23×10^{-10}	0.248	15936 19120 28248	${}^4T_{1g}(F) \rightarrow {}^4T_{2g}(F)$ ${}^4T_{1g}(F) \rightarrow {}^4A_{2g}(F)$ ${}^4T_{1g}(F) \rightarrow {}^4T_1(P)$
3.	[Ni(HMBPE) ₂ (H ₂ O) ₂].2H ₂ O	2.83×10^{-8}	0.106	10131 15337 28169	${}^3A_{2g}(F) \rightarrow {}^3T_{2g}(F)$ ${}^3A_{2g}(F) \rightarrow {}^3T_{1g}(F)$ ${}^3A_{2g}(F) \rightarrow {}^3T_{1g}(P)$
4.	[Cu(HMBPE) ₂]. H ₂ O	2.26×10^{-10}	0.311	14025 19120 37594	${}^2B_{1g} \rightarrow {}^2A_{1g}$ ${}^2B_{1g} \rightarrow {}^2E_g$ C. T.
5.	[Zn(HMBPE) ₂ (H ₂ O) ₂].H ₂ O	3.17×10^{-8}	0.195	26315 36231	C.T. $\pi \rightarrow \pi^*$
6.	[Cd(HMBPE) ₂]	1.93×10^{-7}	0.279	28248 30211 38910	C.T. $n \rightarrow \pi^*$ $\pi \rightarrow \pi^*$

Solid-state d.c. electrical conductivity of HMBPE metal complexes was found in range of 9.23×10^{-10} to $1.93 \times 10^{-7} \Omega^{-1}\text{cm}^{-1}$ at 373 K and decreases in order Cd(II) > Zn(II) > Ni(II) > Mn(II) > Co(II) > Cu(II).^{49,50} It has been found that the electrical conductivity of HMBPE complexes of Mn(II), Co(II), Cu(II) and Zn(II) increases with increasing temperature and decreases with cooling in temperature range under study, indicating the semiconducting behaviour.⁵¹ The d.c. electrical conductivity (σ) has been found to vary according to Arrhenius equation:⁵²⁻⁵⁴

$$\sigma = \sigma_0 \exp (-E_a/kT)$$

Where,

σ_0 - Conductivity constant,

k - Boltzmann's constant,

E_a - Activation energy,

T - Absolute temperature.

The plot of log σ vs. 1000/T of complexes was found to be linear over the temperature range 303-393 K (Fig.-2). The electrical conductance of Ni(II) complex was found to decrease with an increase in temperature up to temperature 343 K and above this, it increases with temperature. This indicates the initial behavior was like a typical metallic conductor while the latter behavior was the characteristics of semiconductor.⁵⁵ The plot of Cd(II) complex shows two different regions. In the low-temperature range, a slow increase in electrical conductivity and high-temperature range rapid increase with temperature has been observed. The activation energy (E_a) of HMBPE metal complexes obtained from plots was found in the range 0.106-0.311 eV and decrease in order Cu(II) > Cd(II) > Co(II) > Zn(II) > Mn(II) > Ni(II)^{56,57}. Activation energy is the direct measure of the band gap of semiconductors, lowers the activation energy, the lower will be the band gap.⁵⁸

CONCLUSION

The metal complexes of Schiff base ligand HMBPE have been synthesized and characterized by different analytical and spectroscopic methods. Solid-state d.c. the electrical conductivity of HMBPE metal complexes has been measured at different temperatures in compressed pellet form.⁵⁹ The study shows semiconducting behavior of HMBPE metal complexes in the range of temperature 303-393 K. The activation energy of electrical conduction of the complexes was found in the range 0.106-0.311 eV in the order of Cu(II) > Cd(II) > Co(II) > Zn(II) > Mn(II) > Ni(II).

Fig.-2: Variation of Electrical Conductivity of HMBPE Complexes with Temperature

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