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Research Article

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Synthesis, characterization and antimicrobial evaluation of benzoinoxime transition metal complexes

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ABSTRACT

The Transition Schiff base complexes derived from Benzoinoxime and transition metal halides has been prepared and characterized. Several physical tools in particular, elemental analysis, infrared and ^{1}H NMR spectroscopy were used to investigate the chemical structure of the prepared compounds. The elemental analysis data shows 1:2 [M: L] ratio. The infrared spectral data of the complex display the complexation behaviour of the Schiff base towards metal ions. The free Schiff base and the complexes have been tested for their antibacterial and antifungal activities against several bacteria and fungi, the obtained result showed enhancement in activity on coordination of metals with ligands.

Keywords: Benzoinoxime, Transition metal complexes of Benzoinoxime, spectral and antimicrobial study.

INTRODUCTION

The field of Schiff base complexes is fast developing because of the wide variety of possible structures for the ligands, depending on the aldehyde and amine used. Many Schiff bases and their complexes have been widely studied because of their industrial and biological applications.[1, 2] Some Schiff bases were tested for fungicidal activity, which is related to their chemical structure[3]. Schiff bases are important class of ligands and have got wide applications in various fields[4, 5].in this present paper we have synthesised Co(II), Ni(II), Cu(II) and Zn(II) metal complexes of the Benzoineoximes And the structure was elucidated using different physical and analytical tools.

EXPERIMENTAL SECTION

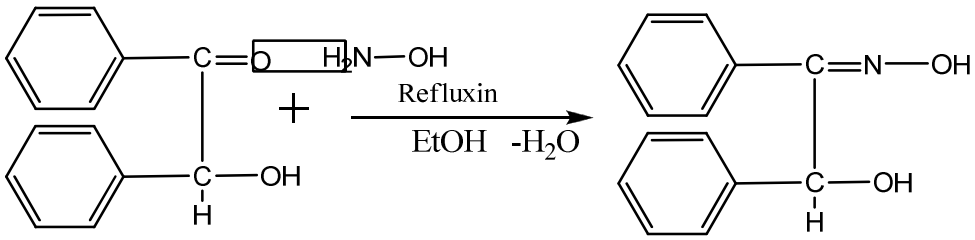
All the chemicals used for experimental work were of AR-grade. The purity of chemicals was checked by melting point, and thin layer chromatography. The purification of the chemicals and solvents were done by distillation as suggested in literature[6]

Synthesis of ligand: Benzoinoxime:
Benzoinoxime was prepared by standard procedure[7] in which 10 gm (0.047 mol) of benzoin and 20 gm (25 ml) of rectified spirit together with an aqueous solution of 8.0 gm (0.087 mol) of hydroxylamine was taken together in a 250 ml round bottom flask. Before using hydroxylamine hydrochloride was neutralized with 4.4 gm (0.091 mol) of sodium hydroxide.

The mixture was refluxed for 60 mins. Then water was added to precipitate benzoinoxime. It was cooled in ice bath. The solid was filtered with solution at pump and it was washed with water finally the product was recrystallised by using ethanol. The purity of the product was checked by TLC and Melting point.

Melting point of Benzoinoxime is 151^0C

Scheme: I



Preparation of simple complexes of Benzoinoxime:

The following complexes of Ni-Benzoinoxime, Cu-Benzoinoxime, Co-Benzoinoxime and Zn- Benzoinoxime has been synthesized. The above metal complexes were prepared by mixing the solution of the primary ligand-PL (Benzoinoxime) (0.06 mole)] in 80 ml of boiling ethanol with 0.03 mole of metal (II) chloride solution in 20 ml of distilled water. The mixture was magnetically stirred for about one hour. Then colored precipitate (except Zn) were formed which was filtered out and washed with hot water or methanol then dried over vacuum. The purity was checked by using TLC.

Table I: Analytical data of Ni (II), Cu (II).Co (II) and Zn (II) complexes with B.O.

Compopund	Mol. Form.	Colour	M.P. ^{0}C	Found Calculated (%)					Mol.Wt
				C	H	O	N	Metal	
Benzoinoxime	$C_{14}H_{13}NO_2$	White	151	74.00 (73.86)	5.72 (5.68)	14.09 (14.00)	6.16 (6.10)	---	227
B.O.Cu(II)	$[C_{28}H_{24}O_4N_2(Cu)]$	Green	165	65.17 (65.08)	4.65 (4.50)	12.41 (12.00)	5.43 (5.40)	12.31	515.5
B.O.Co(II)	$[C_{28}H_{24}O_4N_2(CO)]$	Faint pink	267	65.88 (65.75)	4.70 (4.50)	12.54 (12.45)	5.49 (5.42)	11.37	510
B.O.Ni (II)	$[C_{28}H_{24}O_4N_2(Ni)]$	Yellowish brown	261	65.62 (65.51)	4.68 (4.50)	12.05 (11.90)	5.46 (5.25)	11.71	512
B.O.Zn(II)	$[C_{28}H_{24}O_4N_2(Zn)]$	Colourless	242	64.99 (64.90)	4.64 (4.52)	12.37 (12.21)	5.41 (5.22)	12.57	517

RESULTS AND DISCUSSION

Electronic Absorption Spectral Study of Ligand (UV-Visible spectra)

The solutions of known concentration ($1x10^{-4}$ M) of ligand (oxime) was prepared in chloroform (A.R.) and their UV /Visible (electronic absorption) spectra was recorded on UV /Visible spectrophotometer model UV-1601, SHIMADZU Japan, in the range 190-400 nm. The instrument was calibrated using solution of 0.04 gm. potassium chromate in 0.05M potassium hydroxide solutions. The ligand solution spectrum was recorded by filling reference cell with pure solvent.

The observed υ max values of primary ligand benzoinoxime yields three bands in the range of 360 nm to 450 nm due to $n\rightarrow\pi^*$ and $\pi\rightarrow\pi^*$ transitions[8].

IR Spectral Study of ligand (Benzoinoxime)

The inorganic complexes derived from organic chelating groups have a tendency to absorb in the IR region 400-660 cm^{-1} which is of greatest practical value in the study of metal complexes[9]. The infrared spectra of ligand (oxime) was recorded on a Perkin Elmer Spectrum 1b No.-75430 Nujol spectrophotometer over the range 4000 cm^{-1} to 450 cm^{-1} using KBr pellet technique.

Primary ligand shows the IR stretch in the region of 1500-1650 cm^{-1} for azomethine (>C=N group) which is in agreement with literature values[10-11]. Similarly it exhibits a band at 3200-3300 cm^{-1} for (=N-OH) oxime group which is agreement to the literature survey[12-15], it is shifted to lower region after complexation with the metal. The three peaks in the region of 1400-1600 cm^{-1} shows presence of aromatic >C=C< strech[13]. The aromatic C-OH stretch is found at 3450 cm^{-1} in case of benzoinoxime [16], which is shifted to lower frequency after complexation

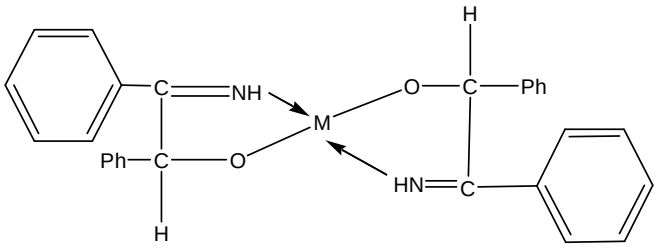
^{1}H NMR Spectral Study of Ligand:

7.28 (s, 5H) Aromatic proton attach to –CHOH group, 7.45(m, 3H) Aromatic proton attach to oxime group, 7.85(d, 2H) Aromatic proton attach to oxime group, 4.3 (s, 1H) Aliphatic proton, 3.87 (s, 1H) Aliphatic OH, 2.3 (s, 1H) oxime OH.

Characterization of metal Benzoinoxime complexes:
All the metal chelate (oxime) prepared are stable to air and moisture. These are insoluble in water and in different polar and non-polar organic solvents at room temperature. Some complexes are easily soluble and some are sparingly soluble in DMSO and in DMF.

The synthesized metal complexes are characterized by elemental analysis, electronic and infrared absorption spectroscopy.

The elemental analysis data shows 1:2 metals to ligand ratio. In the ^{1}HNMR spectra broad peaks are observed for the complexes due to complex pattern of splitting. The infrared spectral data of the complex display the complexation behaviour of the Schiff base towards metal ions.



M= Cu(II), Ni(II), Co(II), Zn(II)
Fig.1

ANTIMICROBIAL STUDY

a) Antibacterial activity by ditch-plate method

Nutrient agar of 20ml. was placed in a flat bottomed Petri dish. When solidified, 4 ml of second nutrient sol., Seed with test bacteria was poured evenly onto the first layer (at 48^{0}c). As soon as the second layer was solidified, in six sterile stainless steel cylinders were added an equal amount of a standard penicillin solution of concentrations 2.0, 1.5, 1.0, 0.5 and 0.25 mg/ml. Sample of the test solutions were deposited analogously on the other Petri dishes. The dishes were incubated at 37^{0}c for 16-18 hours. During this time the penicillin diffuses out of the cylinder into the surrounding agar and suppresses the growth of the test organism. Thus the cylinder was surrounded by clear zone, free of bacteria. The diameter of each zone provides an index of activity of the penicillin preparation.

b) Antifungal activity by paper disc diffusion method

Whatman No. 1 filter paper disc of 5mm diameter were sterilized by autoclaving for 15 min. at 121^{0}C. The sterile disks were impregnated with different compounds. Agar plates were surface inoculated uniformly from the both culture of the tested microorganisms. The impregnated disks were placed on the medium suitably spaced apart and plates were incubated at 28^{0}C for 72 hours. The inhibition zones caused by various compounds on the microorganisms were examined.

Table II- Antimicrobial Activity

Sr.No.	Ligand /Complex	Bacterial Strain		Fungal Strain	
		E.Coli	*S.Aureus*	*A. Niger*	*A. Flavus*
1	PL	8	9	8	9
2	Cu(II) PL	2	8	3	4
3	Ni(II) PL	2	4	3	4
4	Co(II) PL	0	0	0	0
5	Zn(II) PL	0	0	0	0

CONCLUSION

All these complexes are insoluble in water but moderately soluble in DMSO, DMF, dioxane and chloroform. From the above discussion and on the basis of results of elemental analysis, electronic spectral data, IR study, it may be concluded that the metal to ligand ratio is 1:2.

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