



SYNTHESIS AND BIOLOGICAL SCREENING OF 3D TRANSITION METAL COMPLEXES WITH SCHIFF BASE DERIVED FROM 4-CHLORO BENZALDEHYDE WITH 2 AMINO 4-PHENYL THIAZOLE

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ABSTRACT

Metal complexes of Co (II), Ni (II), Mn (II) and Zn (II) having general formulae $[ML_2X_2]$ was prepared. Where L_2 = Schiff base derived from 4-chloro benzylidene with 2-amino 4-phenyl thiazole. The characterization of ligand as well as their metal complexes has been made on the basis of elemental analysis, magnetic susceptibility measurement, Infrared spectra and magnetic moment data. The anti-microbial screening was done on *E.Coli*, *S.aureus*, *B.subtilis*, *S.typhi* *A.niger* and percentage inhibition was found after 168 hours.

KEY WORDS: Co (II), Ni (II), Mn (II) and Zn (II) complexes, Schiff base

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INTRODUCTION

Thiazoline and their derivatives have created an interest among the scientist due to their wide range of activity¹. They are mainly used as analgesic, nematocides, bactericides, fungicides etc. In the present day therapy these thiazolines are also popular for their radioprotective activities. The versatility of thiazole and their derivative is demonstrated by the fact that some of these compounds possess antimalarial², anthelmintic³, antifungal^{4,5}, antimalarial⁶, antiplasmodic⁷ and antitubercular⁸ activities. These compounds are also used as local anaesthetic⁹ anti radiation drugs¹⁰ anti viral and anti neoplastic agent¹¹ and above all vulcanization accelerators¹² in the rubber industry.

EXPERIMENTAL

The entire reagent used were of AR grade otherwise purified before used,

Preparation of ligand

The ligand was prepared by the condensation of 4-chloro benzaldehyde with 2-amino 4-phenyl thiazole using liquid bromine as condensing agent. First 4-chloro benzaldehyde 0.02 mole was added to 0.02 mole of 2-amino 4-phenyl thiazole 50 ml. absolute ethanol. Mixture was refluxed for four hours in a round bottom flask using water condenser fitted with anhydrous CaCl₂ guard tube at the top of the condenser. After the reaction approximately 50% of the alcohols were distilled off and the

reaction mixture was cooled by immersing the flask in ice. The precipitate obtained was filtered, washed three times by minimum amount of absolute ethanol (ice cooled) and dried in electric oven at 60°C-65 °C

Preparation of the complexes

Approximately 0.02 mole of the ligand (10% in excess than the required amount) dissolved in 25 ml of absolute ethanol and 0.01 mole of the respective chlorides of the metal [Mn(II), Co(II), Ni(II) and Zn(II)] dissolved in 25 ml of absolute ethanol and refluxed on water bath for an hour. The reaction mixture was concentrated approximately half of its total volume and then cooled in ice. Different colored precipitates for different metal ions are obtained. The precipitate were filtered washed with ice cold absolute ethanol and dried in electric oven at 85-95 °C. Elemental analysis and spectral data are shown in table.

RESULTS AND DISCUSSION

Yield (Calculated/observed) = 9.06/7.86 gm

Percentage = 87%

Melting point = 189 °C

Analytical data suggested 1:1 stoichiometry for the binary complexes. The complexes were soluble in common organic solvents i.e. DMSO (Dimethyl silane organic solvent) and DMF. (Dimethyl furane)

TABLE 1
COLOUR, ANALYTICAL, CONDUCTANCE AND MAGNETIC
MOMENT DATA OF METAL COMPLEXES

Complex	Color of the complex	Elemental analysis						Ω cm /mol	μ_{eff} (BM)
		% calcd./ (found)							
		M	C	H	N	S	Cl		
[Mn (C ₁₆ H ₁₂ N ₂ SCl) ₂]Cl ₂	Yellow	7.59 (7.53)	53.12 (53.10)	3.04 (3.00)	7.74 (7.10)	8.85 (8.79)	19.6 (19.1)	61.4	5.80
[Co (C ₁₆ H ₁₂ N ₂ SCl) ₂]Cl ₂	Light pink	8.10 (8.9)	52.80 (52.00)	3.02 (3.00)	7.7 (7.1)	8.8 (8.0)	18.75 (18.70)	59.2	3.36
[Ni(C ₁₆ H ₁₂ N ₂ SCl) ₂]Cl ₂	Light Green	8.1 (8.0)	52.82 (52.70)	3.01 (2.9)	7.7 (7.3)	8.8 (8.1)	19.5 (19.1)	62.3	3.36
[Zn (C ₁₆ H ₁₂ N ₂ SCl) ₂]Cl ₂	White	8.91 (8.8)	52.35 (52.30)	2.99 (2.91)	7.6 (7.2)	8.7 (8.1)	19.36 (19.31)	58.2	3.27

M=Metal C= carbon H= Hydrogen N= Nitrogen S= sulphur Cl= chlorine

The magnetic moment values of complexes are found to be 3.27 – 5.80 B.M. The spectra of these complexes exhibit three bands at 10900, 16100 and 22400 cm⁻¹ corresponding to the transition 3A_{2g}(F) → 3T_{2g}(F), 3A_{2g}(F) → 3T_{1g}(F) and 3A_{2g}(F) → 3T_{2g}(P) respectively, which corresponds to octahedral geometry⁷. Magnetic moment value of the present Mn(II) complexes was 5.80BM indicating that 6A_{1g} as ground state for d⁵ configuration in high spin octahedral stereochemistry. The electronic spectra of Mn(II) complexes exhibit three bands at 15650-15500, 18630-18400 and 23650-23400 cm⁻¹ which can be assigned to the transition 6A_{1g} → 4T_{1g}, 6A_{1g} → 4T_{2g} and 6A_{1g} → 4E_g, 4A_{1g}(G) respectively. From a careful comparison of the infrared spectra of metal complexes with those of ligands, it is inferred that a band at 1200cm⁻¹ due to C=N bonding disappears during chelate formation. This indicates complex formation between the metal cations and the ligand leading to the generation of a new entity. In the chelate the band

observed around 500cm⁻¹ correspond to M-O vibration suggests that phenolic groups are involved in bond formation with metal ions. The M-N stretching frequency in the Schiff base complex is obtained at higher wave number because of the character of M-N due to M-N π interaction. Nakamoto⁷⁻⁸ has shown that M-N stretching frequency undergo coupling with other stretching, vibration resulting in a number of bands. The bands around 730 and 610 cm⁻¹ may correspond to the coupled ν (M-N). From these results it is concluded that the primary ligand is being utilized with various species showing absence of CHO group and generation of new entities. Further weight loss in the complexes at 140-180°C corresponds to a coordinated water molecule.

Bacterial screening

The anti microbial activity of the ligands and synthesized ternary complexes were evaluated by the paper disc plate methods. The MTCC (Microbial type culture collection) culture of *E.coli*, *S.aureus*, *B. Subtilis*, *S.typhi* and *A.niger* were taken for the antimicrobial screening. The

result of the antibacterial screening in terms of zone of inhibition is shown in table 2 and controlled used as mentioned in table 3. The entire synthesized compounds along with the parent compound were screened for their antibacterial activities. DMSO and chloroform were taken as controlled standard. From the antibacterial screening it is observed that Ni-complexes was found to be more active against

B.subtilis, *S.Typhii* and *A.niger* as gram positive bacteria only whereas with gram negative bacteria no significant activity has been observed. Against *A.niger* all compounds were found active amongst the synthesized complexes. Only Mn-complexes show active against gram negative bacteria whereas other found inactive.

TABLE 2
ZONE INHIBITION (mm)

Complexes	solvent	<i>E.Coli</i> (614)	<i>S.aureus</i> (96)	<i>B.subtilis</i> (441)	<i>S.typhi</i> (531)	<i>A.niger</i> (281)
[Mn (C ₁₆ H ₁₂ N ₂ SCl ₂) ₂]Cl ₂	Chloroform	-	-	-	-	0.2
[Co (C ₁₆ H ₁₂ N ₂ SCl ₂) ₂]Cl ₂	Chloroform	-	-	40	-	10.0
[Ni(C ₁₆ H ₁₂ N ₂ SCl ₂) ₂]Cl ₂	Chloroform	-	-	32	19	15.0
[Zn (C ₁₆ H ₁₂ N ₂ SCl ₂) ₂]Cl ₂	DMSO	-	-	-	-	37.0

TABLE 3
CONTROL (mm)

solvent	<i>E.Coli</i>	<i>S.aureus</i>	<i>B.subtilis</i>	<i>S.typhi</i>	<i>A.niger</i>
DMSO	-	14	-	-	11
Chloroform	-	10	-	-	-

From the above results it has been observed that amongst the synthesized complexes were found active against *S.aureus*. Thus it can be

concluded that it is worthwhile to per sue further investigation by modifying the structure as well as concentration ratio.

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