



SYNTHESIS AND CHARACTERIZATION OF '3D' METAL COMPLEXES WITH 2-(FURAN -2- FORMYL IMINO) SULPHANILAMIDO THIAZOLE

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ABSTRACT

The metal complexes of Mn (II), Ni (II), Co (II), Cu (II), and Zn (II) with 2-furan -2-formyl imino sulphanilamido thiazole are synthesized and characterized. The structure of the newly synthesized complexes and the ligands were established by using U.V., I.R., Magnetic moment study and elemental analysis. The ligand and the complexes were also screened for their fungicidal activity against different fungi Viz. *Phoma-exigua*, *Macrophomina-phaseoli* and *Collechtsotrichum – capsici*.

KEY WORDS

FFST, Fungicidal activity, Metal complexes.

INTRODUCTION

The complexing behavior of thiazole with their metal complexes are well known¹⁻⁵. Among the organic chelating ligands azomethines are gaining importance day by day. Because a large no. of such compounds and their metal chelates are well known which possess appreciable antibacterial⁶, fungicidal⁷, antituberculous⁸ and anticancerous activity⁹. Various such compounds have been proved of great importance in the field of medicine, industry, biochemistry and research. Azomethines derived from sulphadiazine and salicylaldehyde have been prepared and used as chelating agent, bactericidal and fungicidal agents¹⁰⁻¹². Keeping the above fact in mind our present paper involves synthesis and fungicidal activity of 2-furan -2-formyl imino sulphanilamido thiazole and their metal complexes.

EXPERIMENTAL

All the chemicals used were of B.D.H. (A.R.) grade, Benzene sulphonyl chloride, 2-Amino thiazole and furfural was purchased from the Sisco laboratories Mumbai.

Synthesis of the ligand 2-(Furan -2-formyl imino) Sulphanilamido thiazole

The ligand was prepared in following two steps

- First of all benzene sulphonyl chloride 0.02 mole was added to 0.02 mole of 2-Amino thiazole in 50 ml. of 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
- The product obtained in the above reaction was dissolved in 50 ml. of absolute ethanol and added to a solution of 0.04 mole of furfural in 50 ml. of absolute ethanol with constant stirring. Mixture



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was refluxed for four hour in a round bottom flask using water condenser fitted with anhydrous CaCl_2 guard tube at the top of the condenser. After the reaction approximately 50% of the alcohol was distilled off and the reaction mixture was cooled by immersing the flask in ice. A precipitate was obtained and filtered, washed three times by minimum amount of absolute ethanol (ice cold) and dried in electric oven at 60-65°C.

Yield (calculated)= 9.06 gm.

Found = 7.86 gm

Percentage = 87%

Melting point = 170°C.

Preparation of Metal complexes

Approximately 0.002 mole of the ligand dissolved in 25 ml. of absolute ethanol and 0.02 mol of metal halides of Mn(II), Co(II) Ni(II),

Cu(II) and Zn(II) dissolved in 25 ml. of absolute ethanol were mixed together and refluxed for an hour. The reaction mixture was concentrated approximately half of its volume and then cooled in ice. Different colored precipitate of the complexes are formed. The precipitate were filtered washed with ice cold absolute ethanol and dried in an electric oven at 85-90°C. Elemental analysis and the color of the complexes are shown in table 1. The I. R. spectra of the ligand and metal complexes were analyzed using IR 20 spectrophotometer using KBr pellets. Electronic spectra were calculated using Cary 2390 spectrophotometer. The magnetic moment was measured by using Guoy method using mercury Tetra iso thiocyanato Co(II) as clibrant.

TABLE 1

ELEMENTAL ANALYSIS DATA OF 2-(FURAN-2- FORMYL IMINO) SULPHANILAMIDO THIAZOLE WIYH THE CHLORIDES OF DIVALENT Mn, Co, Ni, Cu & Zn

| Compound (mol.formulae) | Elemental analysis % found/(calculated) | | | | | | |
|--|---|------------------|----------------|----------------|------------------|------------------|------------------|
| | Color | C | H | N | S | Cl | M |
| [MnL ₂ Cl ₂] ₂ | Buff | 36.60 (36.62) | 2.39 (2.38) | 9.15 (9.16) | 13.94 (13.98) | 15.47 (15.50) | 11.97 (11.94) |
| [CoL ₂ Cl ₂] ₂ | Light pink | 36.29 (36.31) | 2.37 (2.38) | 9.07 (9.10) | 13.82 (13.86) | 15.33 (15.32) | 12.72 (12.80) |
| [NiL ₂ Cl ₂] ₂ | Green | 36.31 (36.32) | 2.37 (2.38) | 9.07 (9.06) | 13.83 (13.84) | 15.34 (15.36) | 12.68 (12.66) |
| [CuL ₂ Cl ₂] ₂ | Light green | 35.93 (35.88) | 2.35 (2.36) | 8.98 (8.96) | 13.68 (13.65) | 15.18 (15.20) | 13.59 (13.61) |
| [ZnL ₂ Cl ₂] ₂ | Dirty white | 35.79 (35.81) | 2.34 (2.33) | 8.94 (8.96) | 13.63 (13.64) | 15.12 (15.13) | 13.93 (13.96) |





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RESULT AND DISCUSSION

On comparing the IR spectra of the ligand with that of its complexes, a negative side shift ranging from 15-25 cm^{-1} in stretching frequencies corresponding to the furan ring oxygen and the azomethine (C=N) groups have been observed. This clearly shows the participation of furfural oxygen and azomethine nitrogen in the coordination. In fact, the shift in the position of the absorption band upon coordination depends upon its conjugation with the adjacent heterocyclic group and on the capacity of the metal ion to the ligand π orbitals. In high transfers of the electron density, either the band disappears or shifts, as has been observed in the furan ring

oxygen and azomethine $\nu(\text{C}=\text{N})$ stretching frequencies indicate the transfer of electron density to a significant extent. This fact also gets support by the considerable metal-ligand overlapping as calculated by ν_2/ν_1 values from ν_2 & ν_1 electronic spectral frequencies.

In chloro complexes, medium intensity absorption bands corresponding to the metal to ligand coordination have been observed in the far IR region at 540-580, 350-430 and 270-330 cm^{-1} which are assignable to M-O, M-N, and M-Cl stretching vibrations respectively. The IR spectra are given in table 2.

TABLE 2
I.R. spectra of metal chloride and their complexes

| Ligands | Mncomplex | CoComplex | NiComplex | Cucomplex | ZnComplex | Assignment |
|---------|-----------|-----------|-----------|-----------|-----------|--|
| 1100(s) | 1070 | 1080 | 1070 | 1065 | 1080 | Furan ring oxygen stretching vibration |
| 1245 | 1220 | 1215 | 1215 | 1220 | 1210 | |
| 1605 | 1575 | 1565 | 1570 | 1580 | 1575 | $\nu\text{C}=\text{N}$ azomethine stretching vibration |
| 1120 | 1120 | 1125 | 1120 | 1125 | 1125 | ν sulphone group vibration |
| 1290 | 1290 | 1292 | 1290 | 1288 | 1290 | ν sulphone group |
| | 565 | 575 | 545 | 560 | 580 | $\nu\text{M}-\text{O}$ stretching |
| | 380 | 430 | 420 | 350 | 365 | $\nu\text{M}-\text{N}$ stretching |
| | 285 | 295 | 330 | 305 | 270 | $\nu\text{M}-\text{Cl}$ stretching |

The fungicidal screening of ligand and metal complexes are recorded in table 3. The activity was determined by using the growth method. The fungitoxicity also varies from fungus to fungus as well as on their concentration. The ligand is found to be more toxic in comparison to its metal complex.



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TABLE 3
FUNGICIDAL SCREENING OF (FFST) AND ITS METAL COMPLEXES

| COPLEX | Average percentage inhibition after 168 hour | | | | | | | | |
|--|--|-------|-------|------------------------------|-------|-------|----------------------------------|-------|-------|
| | <i>Phoma exigua</i> | | | <i>Macrophomina phaseoli</i> | | | <i>Collectrotrichum capsicii</i> | | |
| | Concentration in ppm | | | | | | | | |
| | 100 | 50 | 20 | 100 | 50 | 20 | 100 | 50 | 20 |
| FFST | 55.60 | 46.40 | 38.20 | 45.60 | 40.00 | 36.20 | 48.00 | 42.28 | 37.90 |
| [MnL ₂ Cl ₂] ₂ | 38.79 | 34.40 | 30.00 | 27.95 | 22.79 | 18.80 | 35.48 | 30.00 | 25.56 |
| [CoL ₂ Cl ₂] ₂ | 40.68 | 35.50 | 31.35 | 30.48 | 25.68 | 20.90 | 38.59 | 32.65 | 30.54 |
| [NiL ₂ Cl ₂] ₂ | 35.67 | 30.05 | 28.55 | 26.87 | 21.48 | 17.92 | 30.55 | 27.67 | 22.44 |
| [CuL ₂ Cl ₂] ₂ | 33.98 | 28.55 | 24.67 | 23.90 | 18.52 | 15.42 | 29.40 | 25.68 | 20.45 |
| [ZnL ₂ Cl ₂] ₂ | 32.10 | 27.54 | 24.58 | 22.40 | 17.51 | 13.79 | 28.90 | 24.94 | 20.00 |

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