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HETEROCYCLIC SCHIFF BASE Cu(II) METAL COMPLEXES AND THEIR X-RAY DIFFRACTION STUDY

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ABSTRACT

A series of metal complexes synthesized by the reaction of Schiff bases (L_1- L_6) with the Cu metal acetates. The ligands used for the synthesis of metal complexes are **L_1**(2-{[1-(2,4-dihydroxyphenyl)ethylidene]amino}-1,3-benzothiazole-6-sulfonamide), **L_2**(2-{[1-(2-hydroxyphenyl)ethylidene]amino}-1,3-benzothiazole-6-sulfonamide), **L_3**(2-[(1H-pyrrol-2-ylmethylidene)amino]-1,3-benzothiazole-6-sulfonamide),**L_4**(2-[(pyridin-2-ylmethylidene) amino]-1,3-benzothiazole-6-sulfonamide), **L_5**(2-{[1-(thiophen-2-yl) ethylidene]amino}-1,3-benzothiazole-6-sulfonamide and **L_6**(2-[(2-hydroxy benzylidene) amino]-1,3-benzothiazole-6-sulfonamide). The data obtained was preceding using XRD data analysis program. From the experimental measurements, various parameters, Crystal System have been estimated. The XRD analysis revealed that all complexes show good intense and sharp peaks, indicating high crystallinity of complexes.

KEYWORDS: Benzothiazole**,** Schiff bases, Cu metal complexes, spectral analysis.

INTRODUCTION

Ketones and amines are very reactive organic compounds due to presence of C=O and NH_2 groups respectively. The nucleophilic addition reactions of these compounds results in an important class of compounds known as Schiff bases having the general structure –R-C=N-. They are formed by the condensation of a primary amine with an active carbonyl compound.

Now a days the transition metal complexes of the Schiff bases are the study of interest for many young scientist because of the wide variety of possible structure for the ligands, depending upon the carbonyl compounds and amines used and also due to their varied industrial and biological applications.[1-5]

The varios biological applications of the Schiff bases are anticancer[6], antidiabetic[7], antimicrobial[8], antitubercular.[9] Many transition metal complexes show fluorescence properties[10] used in DNA binding studies[11], diuretic studies.[12] Keeping all the varied applications of Schiff base metal complexes in mind we synthesized a series of heterocyclic Schiff bases and all the synthesized Schiff bases are analyzed using different physical and analytical tools such as IR, UV, ^{1}H-NMR, Mass Spectra etc.

In this paper we discuss about the crystal nature of the complexes i.e. X-ray diffraction study of Cu (II) metal complexes of Ligands L_1 to L_6 is done to find out the nature of the crystal.

MATERIAL AND METHOD

Synthesis of Schiff Bases

Schiff bases were synthesized by taking equimolar ethanolic solutions of heterocyclic amine and hydroxyketone/ aldehyde in 50 ml ethanol and refluxing for 3-4 hours. The reaction progress was monitored by TLC. After confirming the completion of the reaction by TLC, the reaction mixture was poured on crushed ice or cold water and the solid separated was then filtered, washed with distilled water and dried, recrystallised from ethanol.

Synthesis of Ligands L_1- L6

Equimolar ethanolic solutions of the heterocyclic amine i.e., 2-amino-6 sulfamyl benzothiazole and **(L_1)** resacetophenone, **(L_2)** 2-hydroxy acetophenone**,** **L_3** (pyrrole 2- aldehyde) **L_4** (pyridine 2- aldehyde), **(L_5)** 2-acetyl thiophene, and **L_6** (salicyldehyde) was refluxed on water bath for 3-4 hours. Then the reaction mixture was poured on ice cold water/crushed ice and then the separated solid was collected by filtration, washing and drying, recrystallized from ethanol. The melting points were recorded.

Synthesis of Metal Complexes

For the synthesis of Cu(II) metal complexes we have adopted the method of Rao et.al.[13]

For the synthesis of all Cu (II), the metal acetates were used. Ethanolic solutions of Schiff bases and respective metal acetate solutions were refluxed in the stoichiometric ratio. The precipitated solid complexes filtered, washed to remove excess base and then dried over fused $CaCl_2$ in vacuum desiccators.

RESULT AND DISCUSSION

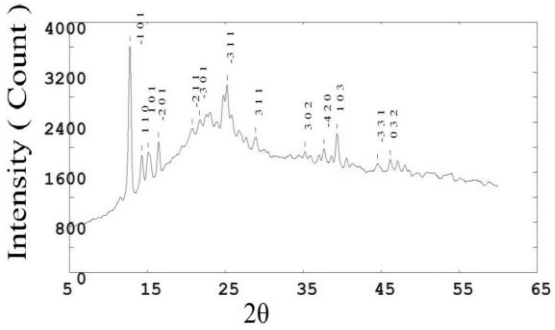
XRD Spectral Analysis of Cu (II) Complexes

Powder XRD patterns of the complexes were recorded on Brukar D8 advance spectrophotometer at department of physics, Shivaji Mahavidyalaya, Omerga. In the 2θ range roughly from 5^o to 60^o and indexed with the help of computer programme. 3D pattern of the structures assigned to the complexes were resolved from POWDERX software which has produced the following results.

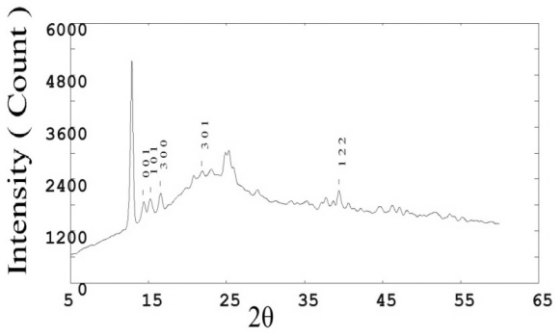
Table I: X-ray Diffraction analysis Data.

Sr. No.	Complex	Lattice Parameter	Lattice Type	Crystal System
1	CuL_1	13.26	P	Monoclinic
2	CuL_2	13.94	P	Monoclinic
3	CuL_3	18.78	P	Orthorhombic
4	CuL_4	16.12	P	Orthorhombic
5	CuL_5	12.34	P	Orthorhombic
6	CuL_6	21.46	P	Orthorhombic

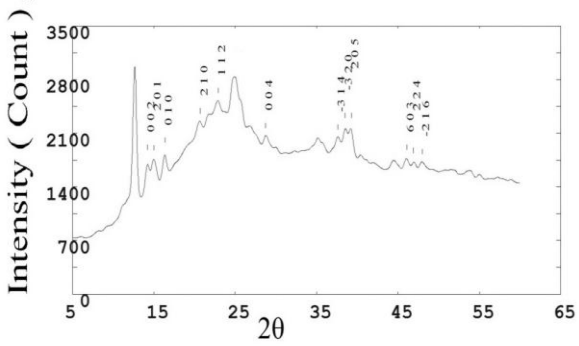
Sample Name: CuL_1



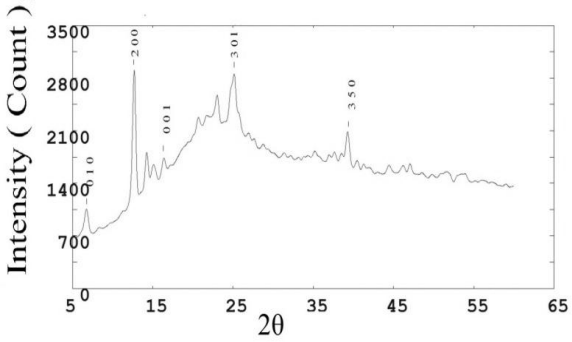
Sample Name: CuL_4



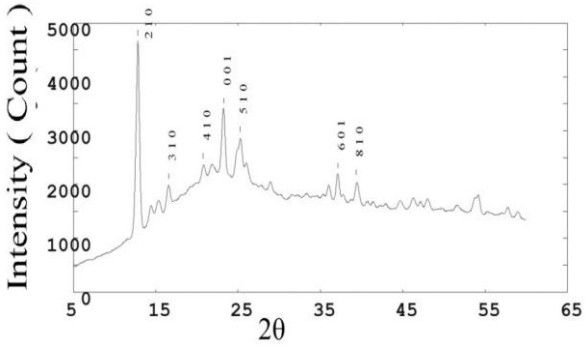
Sample Name: CuL_2



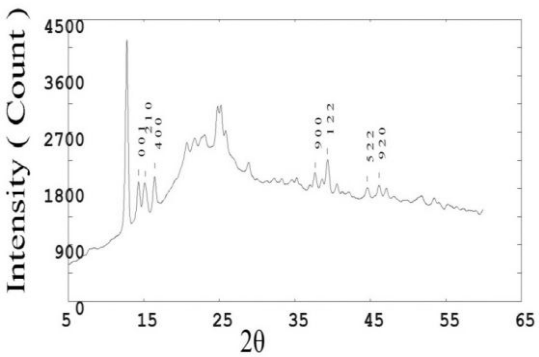
Sample Name: CuL_5



Sample Name: CuL_3



Sample Name: CuL_6



X-ray diffractograms of the metal complexes under investigation show good intense peaks indicating high crystallinity. The results obtained from computational data gave lattice parameter values as $a \neq b \neq c$ and $\alpha = \beta = \gamma$ which suggests the **Orthorhombic Crystal Structure** of P type lattice for copper complex of ligand L_3, L_4, L_5 and L_6.

Lattice parameter values as $a \neq b \neq c$ and $\alpha = \gamma \neq \beta$ which suggest **Monoclinic Crystal Structure** of P type lattice for copper complex of ligand L_1, L_2.

The x-ray diffractograms of Cu (II) complexes with ligand L_1, L_2, L_3, L_4, L_5, L_6, was scanned in the range 5^0 to 60^0at wavelength 1.540598 (A^0). The diffractograms and associated data depict the 2θ value for each peak, relative intensity and inter planar spacing (d-values).

The diffractogram of Cu (II) complex of L_1 had twenty reflections with maxima at $2\theta = 12.78^0$ corresponding to d value $6.921A^0$ having intensity 2359 cps. Diffractogram of Cu (II) complex of L_2 had sixteen reflections with maxima at $2\theta = 12.66^0$ corresponding to d value $6.98641A^0$ having intensity 1869 cps. Diffractogram of Cu (II) complex of L_3 had twenty four reflections with maxima at $2\theta = 12.86^0$ corresponding to d value $6.8782A^0$ having intensity 3000 cps. Diffractogram of Cu (II) complex of L_4 had eighteen reflections with maxima at $2\theta = 12.860^0$ corresponding to d value $6.8782A^0$ having intensity 3433 cps. Diffractogram of Cu (II) complex of L_5 had twenty reflections with maxima at $2\theta = 12.700^0$ corresponding to d value $6.9645A^0$ having intensity 1948 cps and diffractogram of Cu (II) complex of L_6 had twenty reflections with maxima at $2\theta = 12.78^0$ corresponding to d value $6.921A^0$ having intensity 2794 cps.

CONCLUSION

We have synthesized the heterocyclic Schiff bases using pyrrole, pyridine, thiophene 2-aldehyde, O-hydroxy aldehyde / ketone and their transition metal complexes of Cu (II) metal ions. The synthesized Schiff bases are showed bidentate nature and gave stable transition metal complexes. The XRD analysis revealed that all complexes show good intense and sharp peaks, indicating high crystallinity of complexes.

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