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Synthesis and Spectroscopic Characterization-XRD of Mixed Ligand Cu(II) Complexes of 8-Hydroxyquinoline and *o*-hydroxybenzylidene-1-phenyl-2,3-dimethyl-4-amino-3-pyrazolin-5-on

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Abstract : In this paper new mixed ligand Cu(II) complexes containing Schiff base and 8-hydroxyquinoline with the general formula [Cu(L)(Q)] where L= o-hydroxybenzylidene-1-phenyl-2,3-dimethyl-4-amino-3-pyrazolin-5-on and Q=8-hydroxyquinoline were synthesized. The mixed ligand was prepared by chemical root procedure. The resulting product was found to be solid which have been characterized using XRD spectroscopy. The data obtained has been preceding using XRD data analysis program Origin 6.0 Professional. From the experimental measurements, various parameters- lattice parameter and particle size have been estimated. Particle size is found to be in the range of 11.39 – 32.78 nanometer. The XRD analysis revealed the crystalline nature of all the complexes**.**

Keywords: XRD, 8-Hydroxyquinoline Complexes, Schiff base Complexes, o-Hydroxybenzylidene-1-phenyl-2,3-dimethyl-4-amino-3-pyrazolin-5-on Complexes, Mixed ligand Complexes .

1.Introduction

Schiff bases are an important class of ligands in coordination chemistry and find extensive application in different fields [1] Schiff bases derived from the salicylaldehydes are well known as polydentate ligands coordinating in neutral forms [2] The interaction of these donors ligands and metal ions give complexes of different geometries and these complexes are potentially biologically active [3]. Thus, in recent years metal complexes of schiff bases have attracted considerable attention due to their remarkable antifungal, antibacterial, antitumor and anticancer activity [4]. In particular, Merck company has successfully developed an antibacterial drug cilastatin using chiral copper (II). Several research papers have been synthesized and characterized on transition metal complexes of Schiff base derived from salicylaldehyde [5]. A search through literature reveals that there is no work has been done on the XRD of transition metal complexes of the Schiff base and 8- hydroxyquinoline [6]. Keeping this in view, the present paper describes the results of an XRD analysis of mixed Schiff base complexes.

2. Material

All chemicals used in this study were obtained from Merck Company. All chemicals were of analytical grade and they were used without purification.

3. Experimental

The sample is irradiated with a beam of monochromatic x-rays over a variable incident angle range. The X-ray were produced using a sealed tube and the wavelength of X-ray as 0.154nm (Cu K-alpa). The X-ray were detected using a fast counting detector based on silicon strip technology (Bruker LynxEye detector). Interaction with atoms in the sample results in diffracted x-rays when the Bragg equation is satisfied. The X-ray were detected using a fast counting detector based on silicon strip technology (Bruker LynxEye detector).X-ray diffraction pattern have been recorded by Bruker D8 advance diffractometer at IUC, Indore. All chemicals are prepared by chemical root method.

(a)Preparation of Schiff Base (L)

The Schiff base ligand was prepared by the condensation of the p-amino-2, 3-dimethyl-1-phenyl-3-pyrozoline-5-on 0.5 g, 2.47 mmol with the 0.26 ml, 2.47 mmol salicylaldehyde in methanol 15ml. The resulting mixture was then refluxed for 1h. The yellow precipitate formed was filtered and recrystalized from absolute ethanol to give yellow needles [2].

(b)General Method for Preparation of the Complexes:

An ethanolic KOH solution of ligand L 0.64-1.06 g 2.08-3.44 mmole and an ethanolic KOH solution of ligand Q 0.30-0.50 g 2.06- 3.44 mmole were added respectively to an aqeous solution of the metal salts 0.5 g. The reaction mixture

was continuously stirred. The required product was shortly precipitated at room temperature. The precipitates were filtered off and washed with 1:1 ethanol: water and crystallized from ethanol and dried at 60°C.

4. Results and Discussion:

XRD pattern is shown in Fig 1. The sample were characterized at room temperature by X-ray diffraction using Cu Kα radiation. The diffraction pattern of complexes are recorded between 2θ ranging from 10° to 80°. The crystalline size of the samples is estimated using the Scherrer's formula. According to Scherrer's equation , the particle size is given by $\mathbf{t = 0.9\ \lambda/B\ cos\theta}$, where t is the crystal thickness (in nm), B is half width (in radians),θ is the Bragg angle and λ is the wavelength. The particle size corresponding to each diffraction maxima are determined from the measurement of the half width of the diffraction peak. Lattice parameter for simple cubic crystal structure is determined by $\mathbf{a^2= \lambda^2(h^2+k^2+l^2)\ /4sin^2\theta}$. The value of Lattice parameter and the particle size are shown in Table.1 for all the four complexes. The particle size was found to be within in the range 11.39 – 32.78 nm.

Table-1 for Lattice parameter and particle size

Complexes	**Lattice parameter (Å)**	**Particle size (nm)**
$[CuSO_4(L)(Q)]$	4.93	15.23
[CuCl(L)(Q)]	6.03	32.78
$[CuCH_3COO(L)(Q)$	5.22	15.32
$[CuNO_3(L)(Q)]$	8.41	11.39

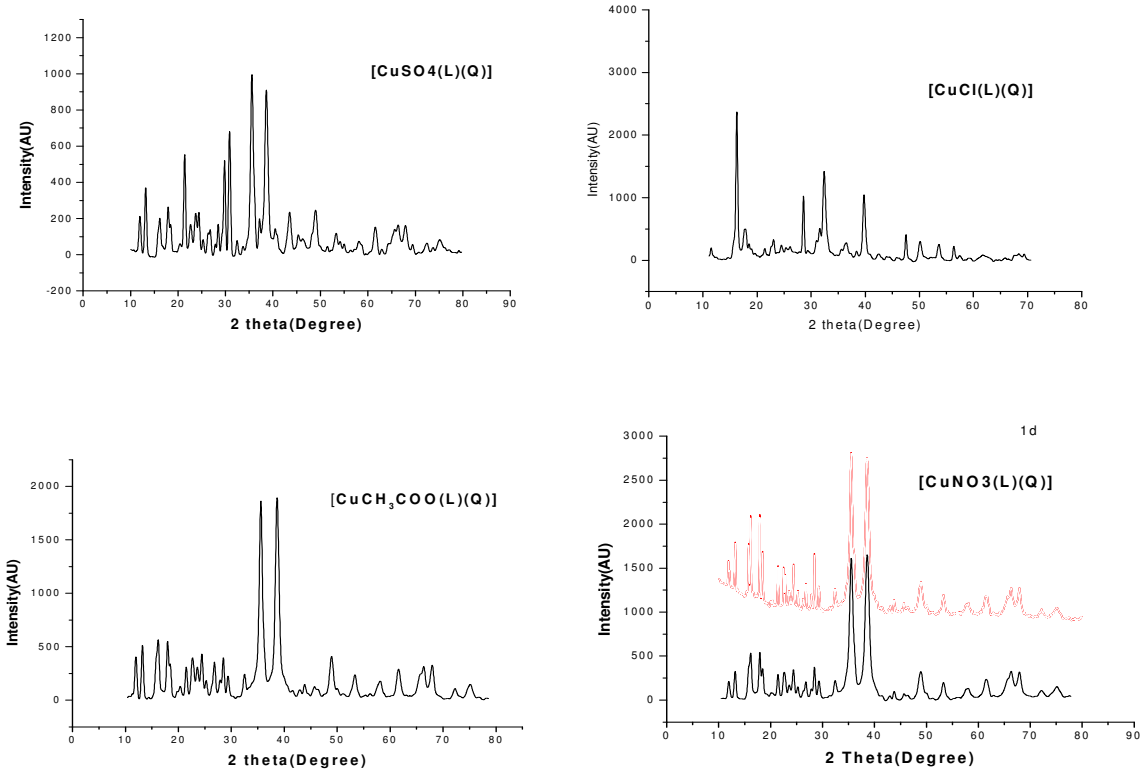

Fig 1. XRD spectra of [Cu(L)(Q)] complexes

5. Conclusion

The XRD pattern is indicative of their crystalline in nature which is confirmed by the main peaks positioned .The X-ray analysis reveals that the sample is cubic in phase as seen from the presence of extra peaks in XRD pattern. All the peaks match with the soft ware JCPDF.

6. Acknowledgment:

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7. References

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