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XRD Analysis of Copper (II) Mixed Ligand Complexes with 3-hydroxypicolinamide and Determination of Crystallite Size and Lattice Parameters

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Abstract: **XRD analysis is used to identify the crystalline phases present in a material and thereby reveal chemical composition information. X-ray diffraction is useful for evaluating minerals, polymers, corrosion products, and unknown materials. In most cases, samples are analyzed by powder diffraction using samples prepared as finely ground powders. XRD analysis is performed by directing an x-ray beam at a sample and measuring the scattered intensity as a function of the outgoing direction, also called a diffraction pattern. The diffraction pattern characterize the crystal structure. X-Ray Diffraction analysis is used to determine crystalline structure parameters. The XRD measurements of all the samples have been carried out and characterized by 2.2 kw sealed Bruker D8 Advance X-ray diffractometer available at UGC DAE CSR, Indore, India. The data of all these samples have been processed by Origin computer software. The crystallite size and lattice parameter of the copper (II) mixed ligand complexes with 2-amino-3-hydroxypicolinamide have been reported.**

Keywords: **Diffraction, copper, crystallite size, lattice parameter.**

I. INTRODUCTION

Transition metal ions are playing an important role in biological processes in the human body [1, 2]. For example, nickel (II), copper (II) and zinc (II) ions are the most abundant transition metals in humans. They are found either at the active sites or as structural components of a good number of enzymes [3, 4]. The study of the coordination chemistry of biologically important metal ions with mixed ligands has been one of the recent developments in the field of bioinorganic chemistry. The powder XRD analytical tool is used to determine the types crystal system, phase of crystal, space group, crystalline defects, crystallite size, lattice parameter, unit cell volume etc. This technique takes place when only the Bragg's condition is fully satisfied. The crystallit size and lattice parameters of the Copper (II) mixed ligand complexes with 3-hydroxypicolinamide ligands have been estimated by the obtained XRD data [5-10].

II. EXPERIMENTAL

The XRD patterns of all Copper(II) mixed ligand complexes with 3-hydroxypicolinamide in the powder form have been recorded at room temperature with the collimated Cu Kα monochromatic X-rays of wavelength 1.54Å having voltage 40kV and maximum current 100mA and the angular range of the diffraction pattern 2θ in the range between 5° to 60° and the 0.05 step size and the time 1sec per step have been kept during the XRD process [11,12]. The XRD measurements of all the samples have been carried out and characterized by 2.2 kw sealed Bruker D8 Advance X-ray diffractometer

available at UGC DAE CSR, Indore, India. The data of all these samples have been processed by Origin computer software.

TABLE I: Copper (II)Mixed ligand Complexes with 3-hydroxypicolinamide and their Molecular Weight

S. No.	Copper (II) Mixed ligand Complexes	Abbreviation of Copper Complexes	Molecular Formula of Copper Complexes	Molecular Weight (in amu)
01	Cu(II) 3-hydroxypicolinamide -4-(phenyl(phenylimino) methyl) benzene-1,3-diol	[Cu(HPA)(PPIPMBD)]	$C_{25}H_{19}O_4N_3Cu.2H_2O$	524.5
02	Cu(II) 3-hydroxypicolinamide -4-(phenyl(p-tolylimino) methyl) benzene-1,3-diol	[Cu(HPA)(PTIPMBD)]	$C_{26}H_{21}O_4N_3Cu.2H_2O$	538.5
03	Cu(II) 3-hydroxypicolinamide -4-((4-chlorophenylimino) (phenyl) methyl) benzene-1,3-diol	[Cu(HPA)(CPIPMBD)]	$C_{25}H_{18}O_4N_3ClCu.2H_2O$	559.0
04	Cu(II) 3-hydroxypicolinamide -4-((4-nitrophenylimino) (phenyl) methyl) benzene-1,3-diol	[Cu(HPA)(NPIPMBD)]	$C_{25}H_{18}O_6N_4Cu.2H_2O$	569.5
05	Cu(II) 3-hydroxypicolinamide -4-((4-methoxyphenylimino) (phenyl) methyl) benzene-1,3-diol	[Cu(HPA)(MPIPMBD)]	$C_{26}H_{21}O_5N_3Cu.2H_2O$	554.5

III. RESULT AND DISCUSSION

All the Copper samples with molecular formula and molecular weight are shown in TABLE I. The samples are characterized by the monochromatic X-ray diffraction with the help of Cu Kα radiation at room temperature. The obtained XRD patterns of the complexes are shown in fig. 1. The XRD patterns have been matched and indexed with JCPDS software. The XRD studies of Copper (II) mixed ligand complexes regarding Schiff base ligand with 3-hydroxypicolinamide interpret by their cubic nature. The crystalline size of all the complexes have been calculated by the Debye Scherrer's relation.

$$t = k\lambda / \beta \cos\theta_\beta \quad (3.1)$$

Where t is the crystal size, λ is the wavelength of the incident radiation, θ_β is the Bragg's angle, k is a constant whose value is usually 0.91 and β is FWHM (in radians) [13-17]. The crystalline size according to every diffraction can be measured by the full width at half maximum of the diffracted peak. The lattice parameter (in Å) has been estimated by the following formula

$$a^2 = \lambda^2(h^2 + k^2 + l^2)/4\sin^2\theta \quad (3.2)$$

Where a is thickness of the particle, λ is the wavelength of the monochromatic X-ray and h, k, l are miller indices and θ is the Bragg's angle.

The crystallite size of Copper (II) mixed ligand complexes with series 3-hydroxypicolinamide is obtained in between 52.05nm to 68.87nm and the lattice parameter in between 6.32Å to 10.41Å which show according to the results that all the samples are found crystalline in nature. These results are reported in TABLE II.

IV. CONCLUSION

The crystallite size (in nm) and lattice parameter (in Å) have been determined by the study of XRD pattern of all Copper (II) mixed ligand complexes of Schiff base with 3-hydroxypicolinamide. The study of crystallite size, lattice parameter and the appearing of sharp intense peaks show that all samples are polycrystalline in nature.

Table II: Crystallite size and the lattice parameter of Copper (II) Mixed ligand Complexes with 3-hydroxypicolinamide

S. No.	Copper Complexes	Crystallite Size (in nm)	Lattice Parameter (in Å)
01	[Cu(HPA)(PPIPMBD)]	53.59	8.66
02	[Cu(HPA)(PTIPMBD)]	57.75	8.65
03	[Cu(HPA)(CPIPMBD)]	54.32	11.43
04	[Cu(HPA)(NPIPMBD)]	68.87	11.66
05	[Cu(HPA)(MPIPMBD)]	52.05	10.64

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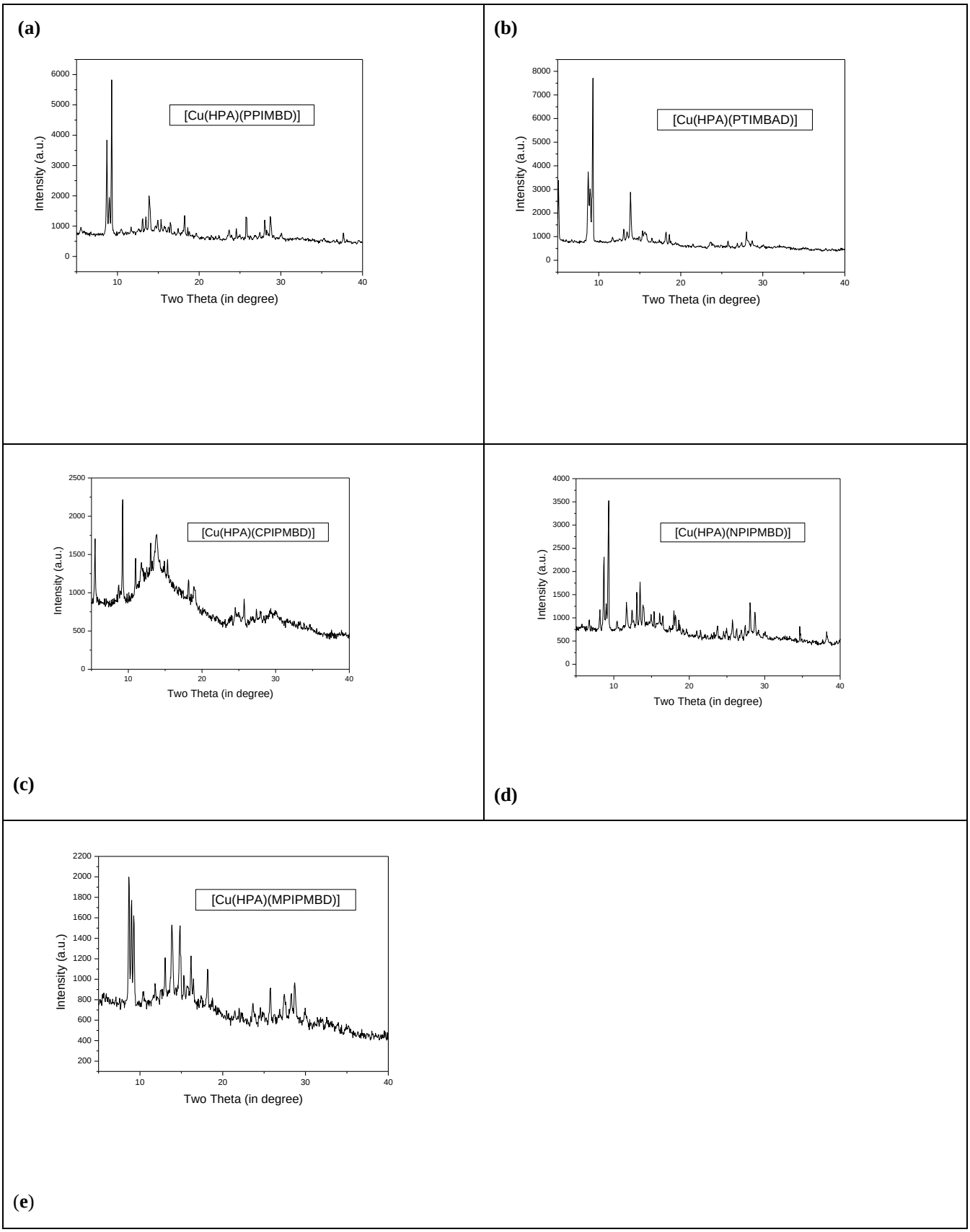

Fig. 1 XRD patterns for Copper(II) mixed ligand complexes (a) [Cu(HPA)(PPIMPBD)], (b) [Cu(HPA)(PTIMPBD)], (c) [Cu(HPA)(CPIMPBD)], (d) [Cu(HPA)(NPIMPBD)] and (e) [Cu(HPA)(MPIMPBD)].