X-ray diffraction and X-ray K absorption near edge studies of copper (II) complexes with amino acids

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Abstract. Synthesis of copper (II) complexes $[CuL_1L_2X].nH_2O$, where n=1, 2,3 $(X=Cl,Br,NO_3)$ (L_1 is 2,2'-bipyridine and L_2 is L-tyrosine) by the chemical root method. The XRD data for the samples have been recorded. EXAFS spectra have also been recorded at the K-edge of Cu using the dispersive beam line BL-8 at 2.5 Gev Indus-2 Synchrotron radiation source at RRCAT, Indore, India. XRD and EXAFS data have been analysed using the computer software. X-ray diffraction studies of all complexes indicate their crystalline nature. Lattice parameter, bond length, particle size have been determined from XRD data.

1. Introduction
X-ray structural studies have revealed that serine phosphorylation of the active and less active forms of an allosteric enzyme muscle phosphorylase induces a conformational transition due to hydrogen bonds involving the phosphate moiety and histidine and arginine residues [1, 2]. In its complex with a substrate, glycyl-L-tyrosine, it fixes the substrate by electrostatic interactions between the carboxylate group of the substrate and the arginine guanidinium group of the enzyme [3]. Ternary Cu(II) complexes containing aromatic heterocycles such as phen and aromatic amino acids like phenylalanine(phe) and L-tyrosine(Tyr) have been shown to be stabilized by stacking interaction [4]. This paper describes the synthesis and characterization of three Cu(II) complexes$[CuL_1L_2X].3H_2O$ where L_1 is 2,2'-bipyridine and L_2 is L-tyrosine [5].

2. Experimental
The required chemicals were of analytical reagent grade and were purchased from Merck, all manipulation were performed using materials as received the content of the metallic ion was elemental analyzer. Copper (II) complexes have been prepared by chemical root method. Elemental analyses were performed after drying the complexes at 600C. All the sample were dissolve in 0.2M HCl (25ml) by heating and after cooling the solution of L-Tyr in 1 M NaOH was added the mixture was concentrated and kept at room temperature.The seprated crystal were collected and crystallized from water.
The powder (debye-scherrer) method is the standard method for obtaining crystal lattice spacing. This experiment can give lattice parameters to about four-figure accuracy. The powdermethod assumes that all orientation is present in the sample. In practice, this would be a time consuming operation to reorient the crystal, measure the angle quad determine the d-spacing for all atomic plane. A faster way is to use a method called the powder method.

3. Results and discussions

Figure 1 Shows the X-ray diffraction pattern of [Cu(bpy)(L-Tyr)Cl].3H2O, Cu(bpy)(L-Tyr)Br].3H2O, Cu(bpy)(L-Tyr)SO4]3H2O. The diffraction pattern of complexes recorded between 2θ ranging from 10^0 to 80^0. The interplanar distance d (Å) are calculated using Brag's law. The crystalline size for each composition are calculated width of the using Scherrer Formula [6-9]. The phase confirmed by XRD studies. The particle size and lattice parameter are shown in the table 1 . Crystal structure has found cubic. The table 2 presents the results for the K- absorption (E_K) and the energy of the principal absorption maximum (E_A) of copper metal and its complexes. EXAFS data shown in figure 2. The bond length of copper complexes evaluated with the help of Athena software [10].

Table 1: XRD for Copper (II) complexes with amino acid

S. No.	Sample name	2θ	h k l	Particle size (nm)	Lattice parameter (Å)
1	[Cu(bpy)(L-Tyr)Cl].3H$_2$O	12.50	1 1 1	9.18	12.01
2	[Cu(bpy)(L-Tyr)Br].3H$_2$O	12.07	1 1 1	10.56	12.40
3	**[**Cu(bpy)(L-Tyr)SO$_4$].3H$_2$O	20.95	2 2 2	10.59	14.82

Table 2: XANES parameters and bond length for the K absorption edge of copper in the complexes

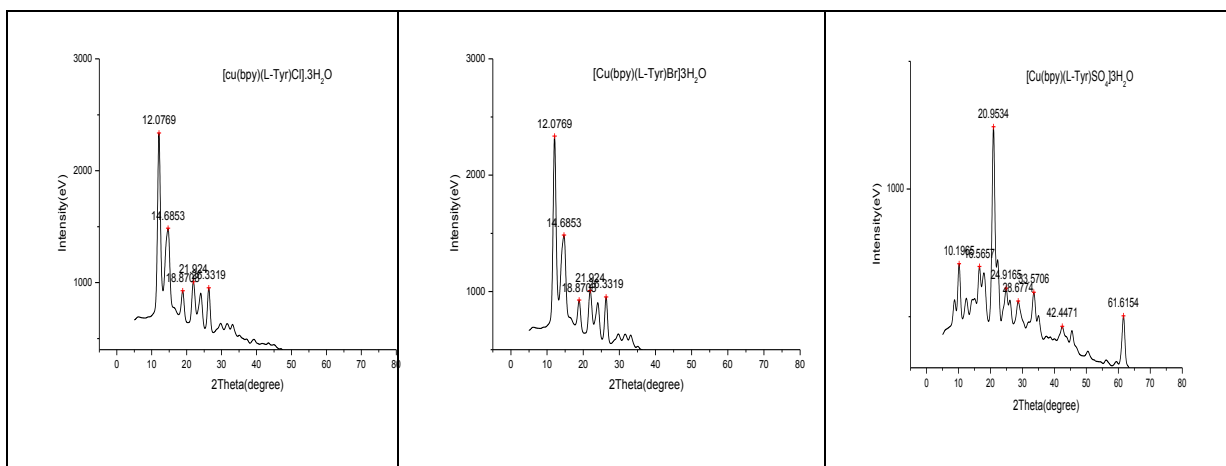| S.No.	Sample name	E_K-edge (eV)	E_A (eV)	Bond length
1	[Cu(bpy)(L-Tyr)Cl].3H$_2$O	8984.6	8997.8	0.9
2	[Cu(bpy)(L-Tyr)Br].3H$_2$O	8988.6	8998.4	1.4
3	**[**Cu(bpy)(L-Tyr)SO$_4$].3H$_2$O	8989.0	8997.5	1.3

Figure 1. XRD Pattern for Cu complexes with amino acids
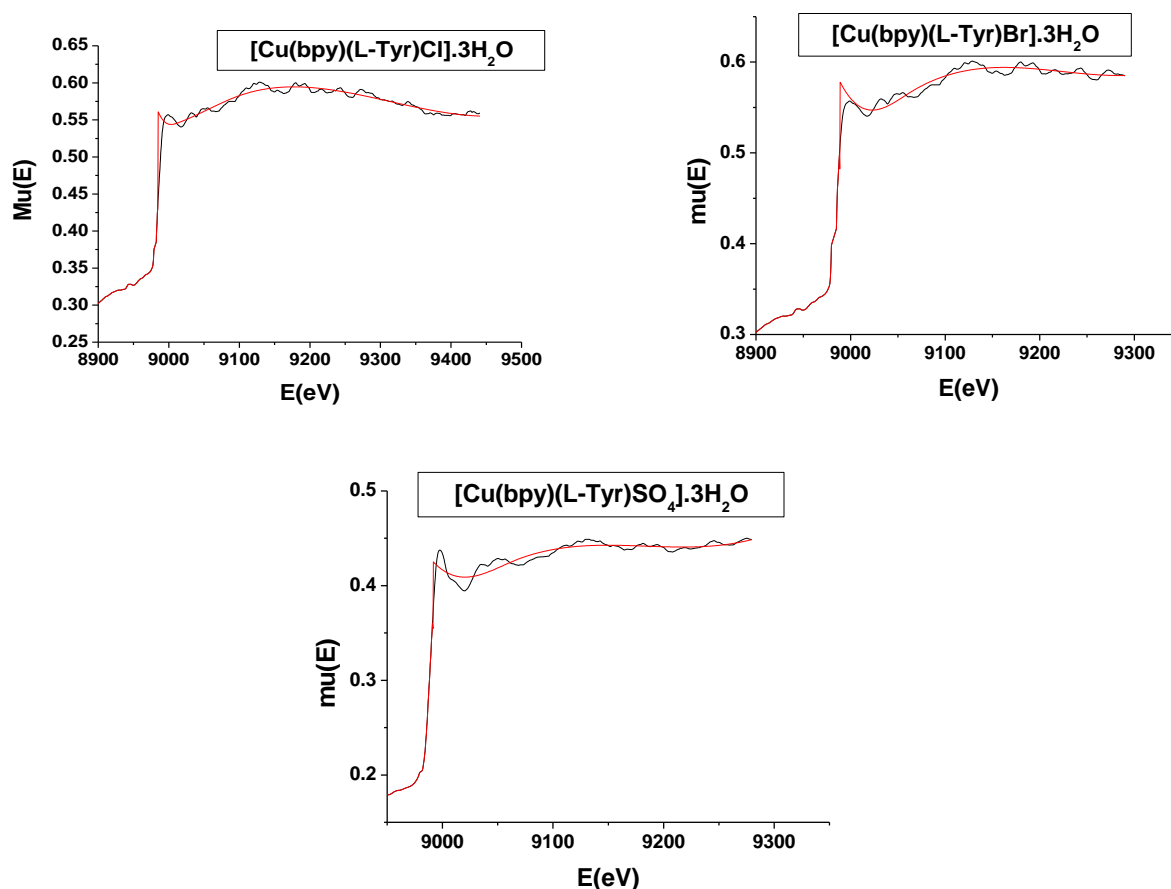
Figure 2. EXAFS spectra [(μ(E) Vs E] for copper complexes

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