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X- RAY DIFFRACTION STUDIES OF SOME COORDINATION POLYMERS OF ADIPYL BIS P CHLOROPHENYL UREA WITH Mn (II) Co (II) Cu (II) and Zn (II) TRANSITION METAL IONS

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ABSTRACT

Coordination polymers of Mn(II), Co(II),Cu(II) and Zn(II) have been prepared with the ligand derived from adipyl bis p chlorophenyl urea. These coordination polymers are coloured, crystalline solid and insoluble in water and in almost all organic solvents. The crystal structure and lattice parameters of these newly synthesized coordination polymers have been studied by X-ray diffraction studies. On the basis of X- ray diffraction data an orthorhombic crystal system has been proposed for the coordination polymers. The X-ray diffraction data was also used to index the compounds and for determination of various parameters.

Keywords: X-ray diffraction studies, Coordination polymers, Adipyl bis p chlorophenyl urea.

INTRODUCTION

Many coordination polymers have been synthesized and characterized by X- ray diffraction method[1,2] and tested for their commercial applications[3-5]. Crystalline orthorhombic nature of chelate polymers of hydoxamic acid with first row transition metal ions was determined by X- ray diffraction technique[6]. Literature survey reveals that coordination complexes of transition metal ions are crystalline with octahedral, tetrahedral or square planar geometry[7]. The present work discusses the synthesis and X- ray diffraction studies of coordination polymers of manganese (II), Cobalt (II), Copper (II) and Zink (II) transition metal ions with adipyl bis p chlorophenyl urea ligands. An attempt has been made to evaluate the lattice parameters.

EXPERIMENTAL

Materials

All the chemicals used were of Analytical grade. Solvents were purified by usual method before used.

Instruments

The X- ray diffraction studies have been performed at RSIC, RTM Nagpur University Nagpur on Philips PW 1710 automated X-ray powdered diffractometer. The experimental conditions employed in reading the pattern were as under. The operating target voltage was 35 KV, the tube current was 20 mA and scanning speed was 0.5 degree 2θ per minute. The X- ray from copper target was filtered with nickel and a monochromatic K α line of wavelength 1.54056 A^o was obtained. Filtration reduces noise due to white radiation and increases resolution also.

Synthesis of coordination polymers

The coordination polymers of adipyl bis p chlorophenyl urea with transition metal ions Mn(II), Co(II), Cu(II) and Zn(II) have been synthesized by dissolving (0.1 m mol) of ligand and (0.1 m mol) of metal acetate separately in a minimum quantity of hot DMF. The hot solution was then mixed and refluxed on an oil bath for 5 to 6 hours. The temperature was maintained at 140^0C.The colour products thus obtained were filtered, washed first with hot DMF and then with ethanol and acetone, and finally dried in oven. The coordination polymers formed were insoluble in almost all organic solvents. The purity of coordination polymers was ascertained by repeated washing with hot DMF and ethanol.

RESULTS AND DISCUSSION

The X –ray diffraction of Mn (II), Co (II), Cu (II) and Zn (II) coordination polymers indicates crystalline nature of these complexes. All the reflection has been indexed for h k l values using reported methods[8]. Bragg's equation ($n\lambda = 2d \sin\theta$) was used to obtain 'd' values of reflection. All the coordination polymers are found to be orthorhombic crystal. These values of sin and 2θ for each peak have been calculated with the help of the cell parameters and corresponding h, k, l values. The lattice constants a, b, c for each unit cell has been found out and is given in Table 1-4. The diffractogram of coordination polymers have been shown in figs. 1-4.

Table -1: X-ray diffraction data of Mn(II) ADPCPU Coordination Polymer

d-observed	d-calculated	I/Io (%)	h	k	l
8.375	8.374	2.5	0	20	3
2.611	2.611	14.5	0	15	23
1.935	1.937	13.8	20	19	0
1.727	1.727	8.9	14	0	25

Type of crystal system – Orthorhombic, Unit Cell Volume = 6582.356 $(A^0)^3$
Lattice parameters: a = 12.250 A^0, b = 25.175 A^0, c = 21.344 A^0

Table – 2: X-ray diffraction data of Co(II) ADPCPU Coordination Polymer

d-observed	d-calculated	I/Io (%)	h	k	l
4.119	4.120	88.5	0	0	6
3.757	3.759	7.8	0	11	20
3.099	3.092	7.0	0	0	8
2.393	2.393	5.6	12	20	0

Type of crystal system – Orthorhombic, Unit Cell Volume = 8272.221$(A^0)^3$
Lattice parameters: a = 12.045 A^0, b = 27.762 A0,c = 24.738A^o

Table-3: X-ray diffraction data of Cu(II) ADPCPU Coordination Polymer

d-observed	d-calculated	I/Io (%)	h	k	l
9.054	9.059	55.6	3	24	13
4.653	4.653	7.1	15	0	7
2.159	2.158	4.3	20	0	19
2.002	2.002	4.5	15	0	29

Type of crystal system –Orthorhombic, Unit Cell Volume = 14113.447$(A^0)^3$
Lattice parameters: a = 17.398 A^0, b = 33.112 A^0, c =24.499A^0

Table-4: X-ray diffraction data of Zn(II) ADPCPU Coordination Polymer

d-observed	d-calculated	I/Io (%)	h	k	l
8.224	8.224	100	0	0	3
3.769	3.769	8.5	7	0	20
2.398	2.398	6.0	11	42	0
1.126	1.126	12.8	0	53	4

Type of crystal system –Orthorhombic, Unit Cell Volume = 14458.06$(A^0)^3$
Lattice parameters: a = 17.745 A^0 , b = 33.023 A^0, c = 24.672A^0

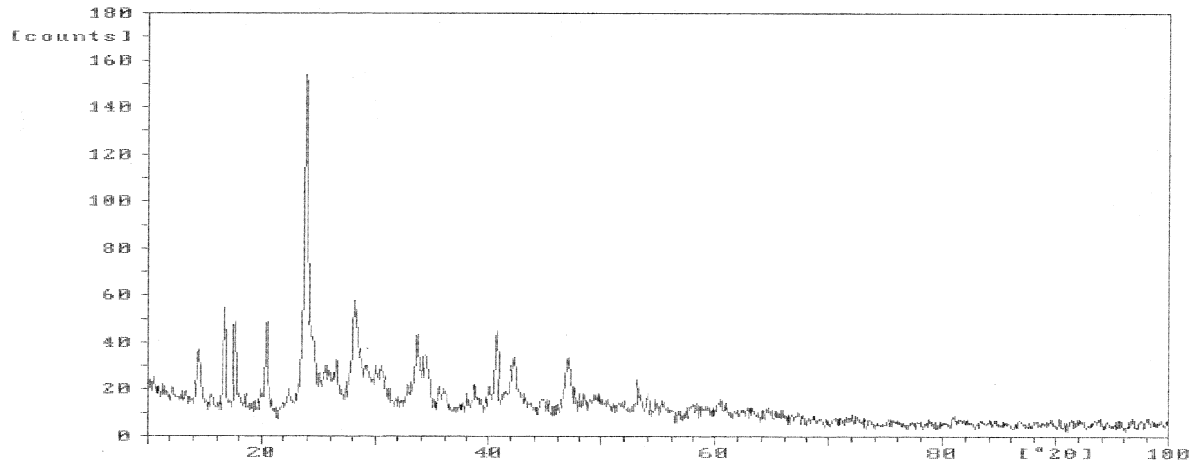

Fig.-1: X-ray diffractogram of Mn(II)ADPCPU coordination polymer

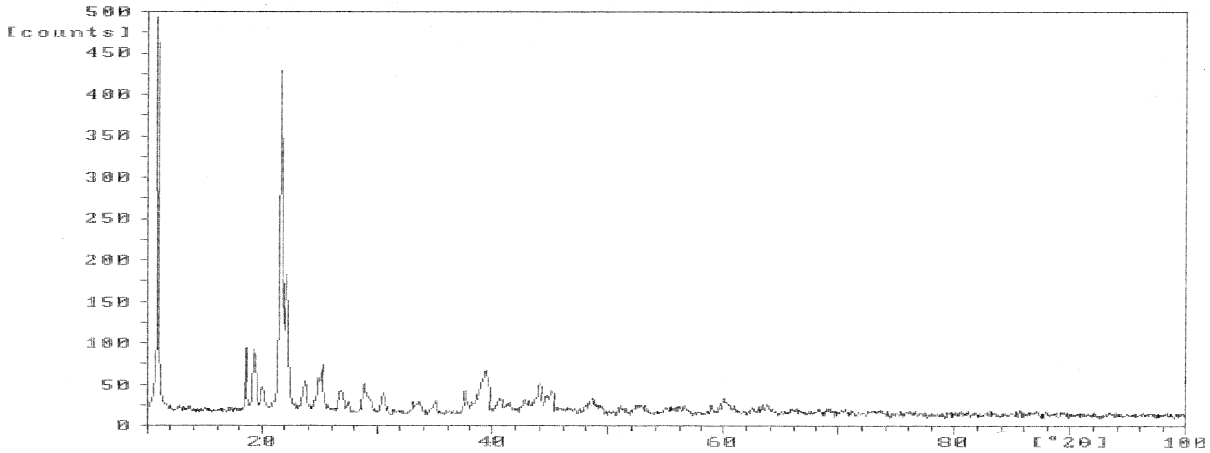

Fig.-2: X-ray diffractogram of Co(II)ADPCPU coordination polymer

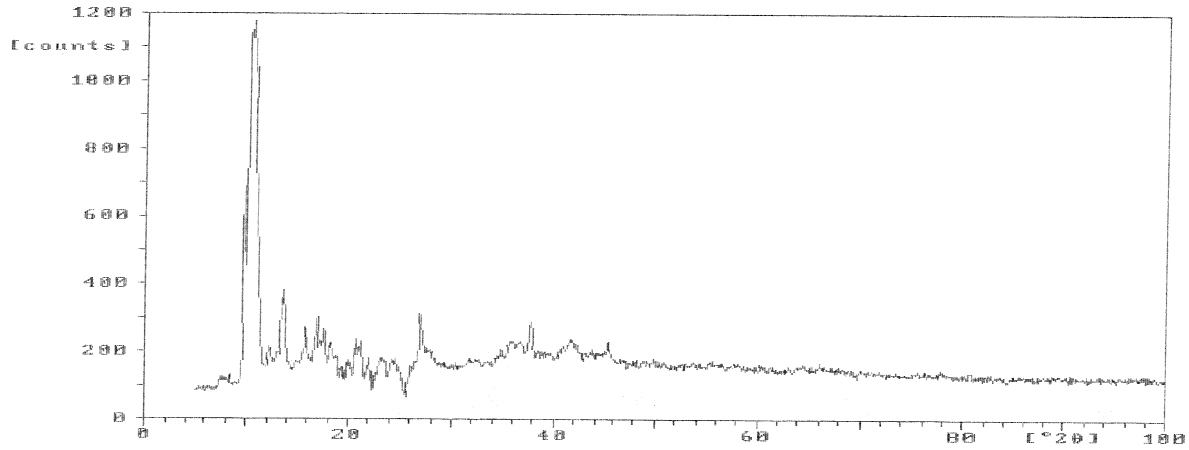

Fig.-3: X-ray diffractogram of Cu(II)ADPCPU coordination polymer

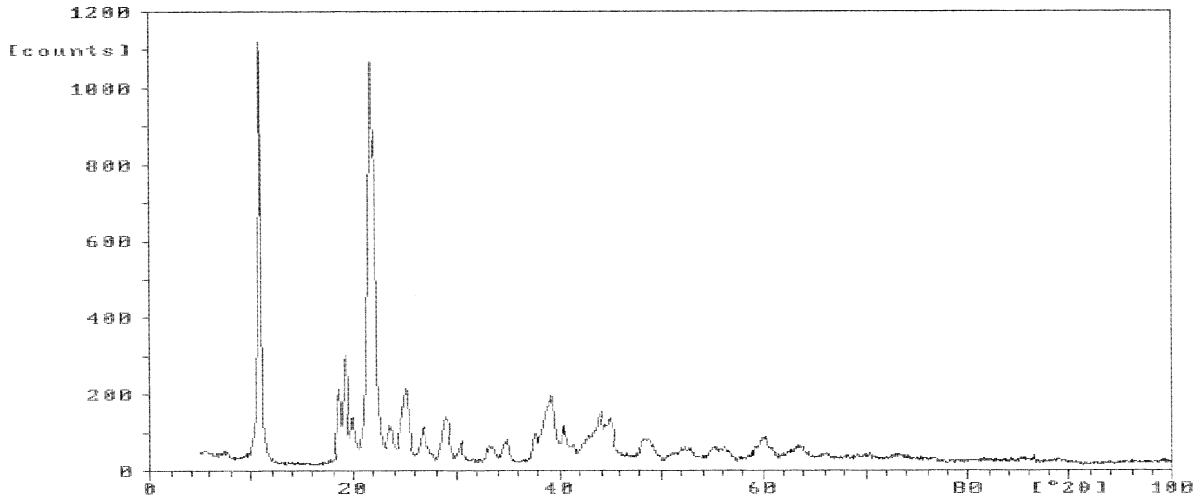

Fig.-4: X-ray diffractogram of Zn(II)ADPCPU coordination polymer

CONCLUSION

On the basis of X-ray diffraction studies it has been found that all the coordination polymers of adipyl bis p chlorophenyl urea are crystalline in nature and have orthorhombic crystallographic systems.

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