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Study on X-Ray Diffraction of some Mn (II), Fe (III), Co(II), Ni(II), Cu(II), Zn(II), Complexes on the basis of Mixed ligands

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Abstract

Comparative studies on the x-ray diffraction parameter of some transition metal complexes such as Mn(II), Fe(III), Co(II), Ni(II), Cu(II) and Zn(II), has been synthesized by 4-methoxyplenylmaine and salicylaldehyde, o-vanillin having equiamolar ratio of 1:1:1(metal : L^1 : L^2) in the same solvent. These complexes were different physico-chemical properties such as different color, different melting points and different crystal systems. All the complexes of XRD studies indicate that monoclinic crystal structure has been proposed for the mixed ligands metal complexes. The XRD data were also being used for the determination of various parameter, unit cell volume and miller indices values (h, k, l). The XRD measurement is to be determine the dimensions and shape of unit cell and to identify the detailed structure of the molecule such as tetrahedral, octahedral or square planer geometry.

Keywords: Mixed Schiff ligand, x-ray diffraction studies, 4-methoxyplenylmaine, salicylaldehyde, o-vanillin.

Introduction

The wavelengths are important part of crystal system of complexes to determine the peak position, miller (h k l) value, unit cell parameters and 2θ value with a radiation source of CuKα by used as x-ray diffractometer range[1-2]. The objectivity of complexes to identify the detailed structure of the molecule is x-ray diffraction. To achieve this objective, we must be able to express mathematically the nature of the measured interference pattern in terms of the position of the various atoms within the crystal[3] To identify crystalline materials they are using XRD instrumental technique[4]. In the present work, literature survey reveals that transition metal complexes generally crystalline as well as amorphous in nature with tetrahedral, octahedral or square planer geometry[5]. In this work the powder x-ray diffraction of Mn(II), Fe(III), Co(II), Ni(II), Cu(II), Zn(II) complexes were mixed ligands scanned in the range of 5°-65° at wavelength 1.540598Å. The diffract gram and associated data depict the 2θ value each peak, relative intensity and inter-planar spacing (d-values). The position of the various atoms within the crystal to measured mathematical expression[6].

Material and Methods

Experimental: All chemicals and solvents used were of A.R. grade. The transition metal (II) nitrates obtained from Rare Earth Ltd. (India). Were used without further purification. salicylaldehyde, o-vanillin and p-anisidine was obtained from Alfa Acer Chemicals and solvents were obtained from Aldrich Chemical Company. The powders XRD were recorded on Perkin Elmer TA/SDT-2960 and Philips 3701, respectively at the range of 5°-65°at wavelength 1.540598Å.

Synthesis of Schiff base ligand: The synthesis of Schiff base ligand were prepared by reported method [7]. The synthesis of L^1 in 50 ml solution of ethanol contain 0.001mol of salicylaldehyde (0.122g), and 0.001 mol of o-vanilline (0.152 g) were continuously stirred for 4 hours at room temperature. The gray color was obtained. The ligands were collected, filtered by using Buckner funnel, washed with ethanol and dried in the desiccator. Purity of the ligands was checked by thin layer chromatography in silica gel plates. The product was purified and recrystallized with a hot ethanol. Obtained yield were 80%.

The synthesis of L^2 in 50 ml solution of ethanol contain 0.001mol of o-vanilline (0.152 g) and 0.001mol of 4-methoxyphenylamine (0.123 g) were continuous stirred for 4 hours at room temperature. The orange color was obtained. The ligands were collected, filtered by using Buckner funnel, washed with ethanol and dried in the desiccators. Purity of the ligand was checked by thin layer chromatography in silica gel plates. The product was purified and recrystallized with a hot ethanol. Obtained yield were 78%.

Synthesis of metal complexes: Synthesis of some transition metal (II) complexes, the mixture of (0.01mol) ligands 2-(E)-[4-methoxybenzylideneamine]phenol (L^1) and 2-(E)-[4-methoxy phenylimino methyl] -6-methoxy phenol (L^2) dissolve in 25 ml hot ethanol solution simultaneously and other metal (II) nitrate (0.01mol) 25 ml of hot ethanol was added under constant stirring for 3 hours at room temperature. The precipitated complexes were filtered off washed with diethyl ether, ethanol and dried vacuum under anhydrous calcium chloride ($CaCl_2$). Decomposition points of transition (II) complexes were above 200°C.

Results and Discussion

An x-ray diffractogram of some transition mixed ligand metal (II) complex indicates that they are crystalline as well as amorphous in nature. To calculate the h, k, l values of reflection by using reported methods[8]. All the mixed ligand metal complexes except the amorphous complexes found to be crystal system were monoclinic. These values of sin 2d for each peak have been calculated with the help of cell parameters and corresponding h, k, l, values. The lattice constants a, b and c for each unit cell have been found out and are given in table-1. The XRD patterns the major peak, which showed relative intensity greater than 10% indexed by computer program[9]. The above indexing method also yields miller indices (h, k, l), unit cell parameters and unit cell volume. To calculate the experimental density values of the complexes by using specific gravity method[10]. In a figure-1- 6 show in that the relative intensity vs 2θ values.

They'd' values of reflections were obtained using Bragg's equation.

$n\lambda = 2d \sin\theta$

To calculate the unit cell volume of Mn(II), Fe(III), Co(III), Ni(III), Cu(III) and Zn (II) complexes for monoclinic system of crystal by the following equation was used.

$V = abc \sin \beta$

Table-1
Lattice constant, Unit cell Volume, Crystal system, Inter-planar spacing of metal

Unit cell of complexes	Lattice Constant			Unit cell Volume	Edge length	Inter axial angle	Crystal system	Reflections	2θ	d-value (Å)
	a (Å)	b (Å)	c (Å)	V (Å^3)						
Mn (II)	14.24	9.51	9.32	733.47	a ≠ b ≠ c	α = β = 90 ≠ y˜	monoclinic	9	26.11	3.40
Fe(III)	12.53	9.23	7.12	478.98	a ≠ b ≠ c	α = β = 90 ≠ y˜	monoclinic	14	8.724	10.12
Co (II)	12.54	10.35	7.42	559.64	a ≠ b ≠ c	α = β = 90 ≠ y˜	monoclinic	11	12.13	7.28
Ni(II)	14.23	9.23	7.25	553.67	a ≠ b ≠ c	α = β = 90 ≠ y˜	monoclinic	11	16.94	5.68
Cu(II)	14.47	11.54	9.25	898.23	a ≠ b ≠ c	α = β = 90 ≠ y˜	monoclinic	15	13.63	6.48
Zn(II)	11.25	9.25	6.32	382.58	a ≠ b ≠ c	α = β = 90 ≠ y˜	monoclinic	12	14.60	6.05

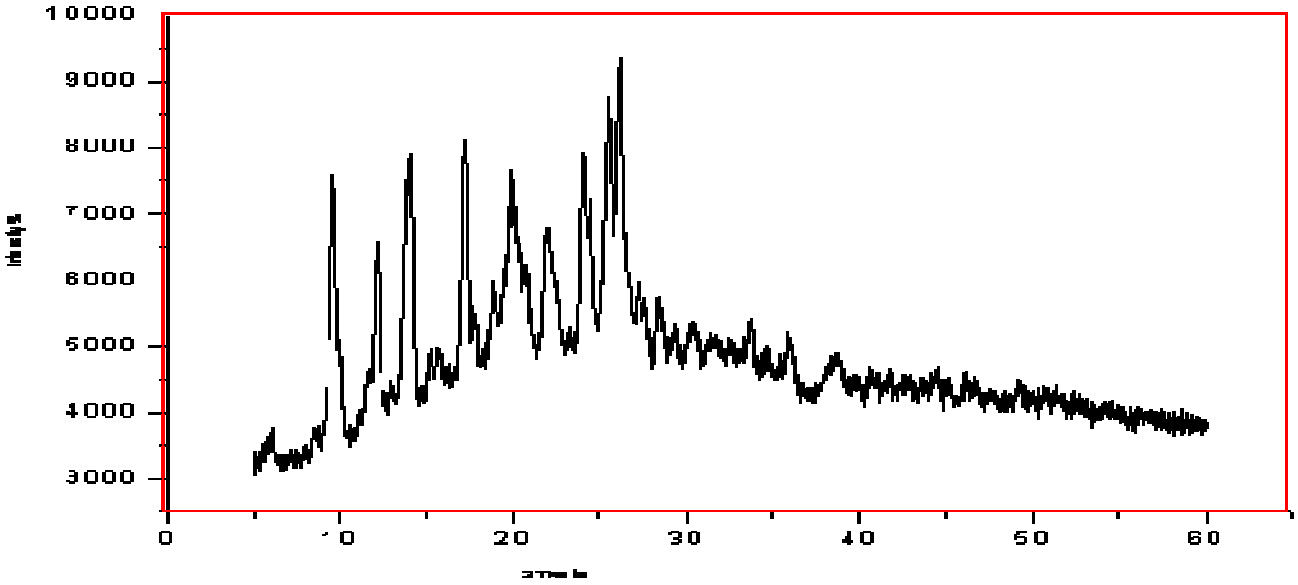

Figure-1
X-ray diffraction data of [MnL^1- L^2 (H$_2$O) $_2$] complex

Table-2
Millar indices and interplanar distances of $Mn^{(II)}$ complex

h	k	l	2θ (Obs)	2θ (Cal)	d (Obs)	d (Cal)	Relative intensity
-1	0	0	6.56	7.16	13.46	12.33	0.56
-1	0	0	7.17	7.16	12.30	12.33	0.54
-1	0	0	7.54	7.16	11.71	12.33	0.53
-1	0	0	7.94	7.16	11.12	12.33	0.68
0	0	1	9.62	9.47	9.18	9.32	22.27

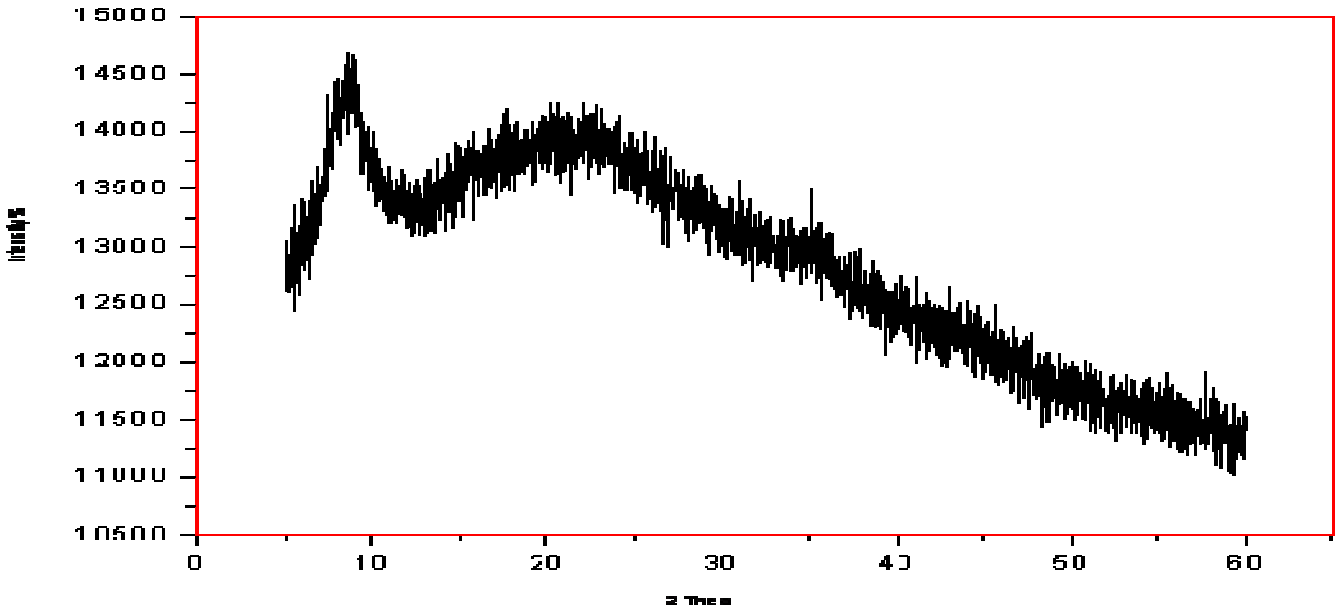

Figure-2
X-ray diffraction data of [FeL^1- L^2 $(H_2O)_2$] Complex

Table-3
Millar indices and interplanar distances of $Fe^{(III)}$ complex

h	k	l	2θ (Obs)	2θ (Cal)	d (Obs)	d (Cal)	Relative intensity
-1	0	0	7.89	8.13	11.18	10.85	4.53
-1	0	0	8.52	8.13	10.36	10.85	5.42
-1	0	0	8.72	8.13	10.12	10.85	5.46
-1	1	0	10.09	9.84	8.75	8.97	1.60
0	1	0	10.50	10.94	8.41	8.07	0.52
0	1	0	10.76	10.94	8.21	8.07	0.40

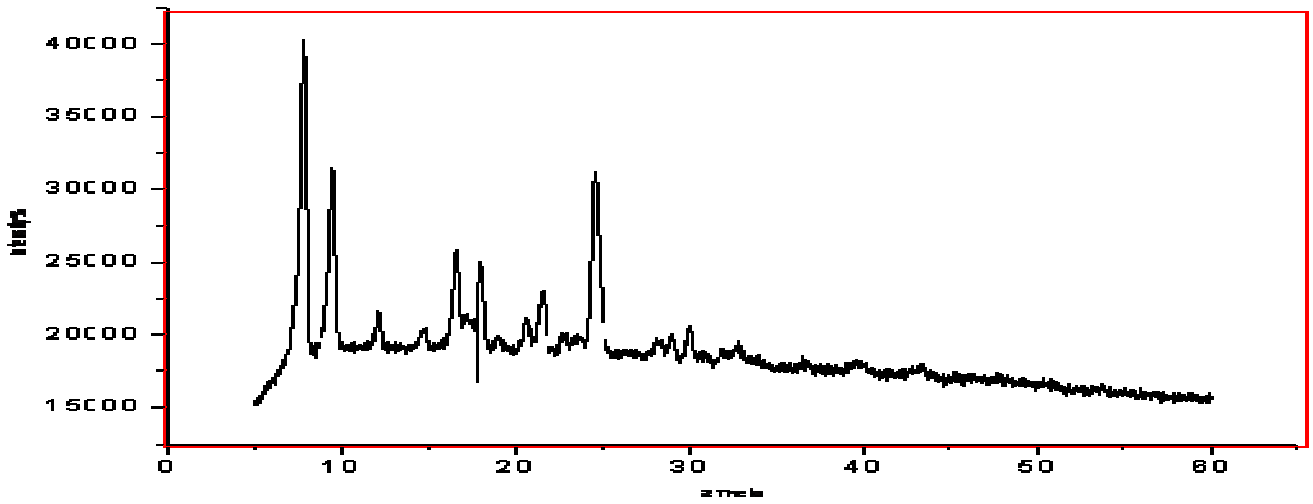

Figure-3
X-ray diffraction data of [CoL^1-L^2 $(H_2O)_2$] Complex

Table-4
Millar indices and interplanar distances of Co (II) complex

h	k	1	2θ (Obs)	2θ (Cal)	d (Obs)	d (Cal)	Relative intensity
-1	0	0	7.81	8.13	11.30	10.86	133.33
0	1	0	9.51	9.85	9.28	8.96	73.08
0	0	1	12.13	11.91	7.28	7.42	15.52
-1	0	1	14.66	14.44	6.03	6.12	7.25
-2	0	0	16.61	16.30	5.33	5.43	38.2
-1	2	0	17.21	17.19	5.14	5.15	13.00

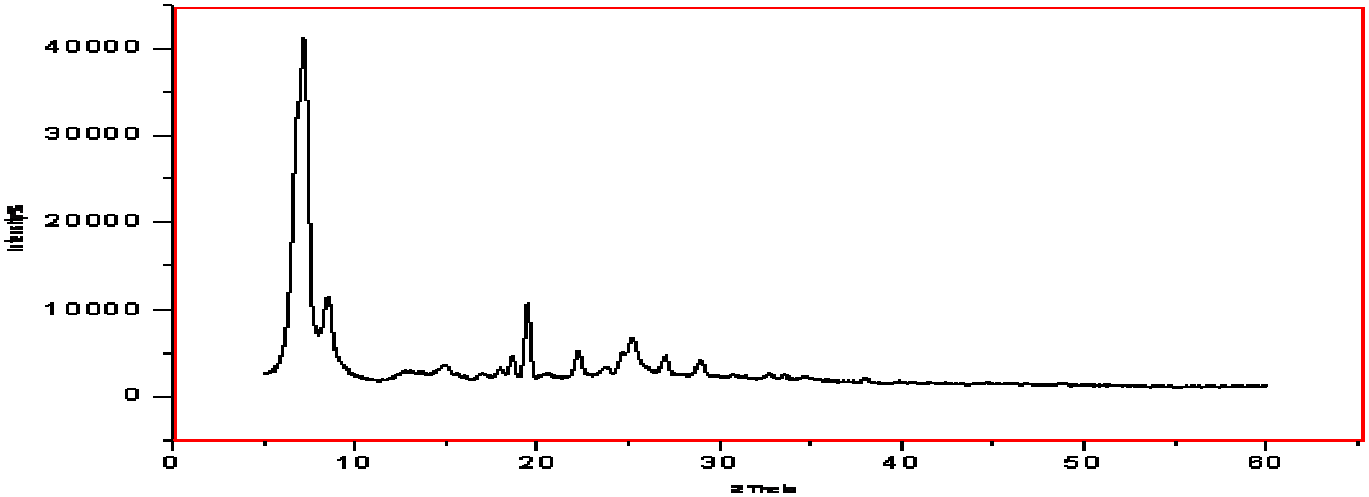

Figure-4
X-ray diffraction data of [NiL^1-L^2 $(H_2O)_2$] Complex

Table-5
Millar indices and interplanar distances of Ni (II) complex

h	k	l	2θ (Obs)	2θ (Cal)	d (Obs)	d (Cal)	Relative intensity
-1	0	0	7.213	7.16	12.24	12.32	226.31
-2	1	0	12.76	13.02	6.92	6.79	58.94
-1	0	1	13.63	14.15	6.48	6.25	47.25
-2	0	0	14.88	14.35	5.94	6.16	93.15
-1	1	1	15.56	15.60	5.69	5.67	40.26

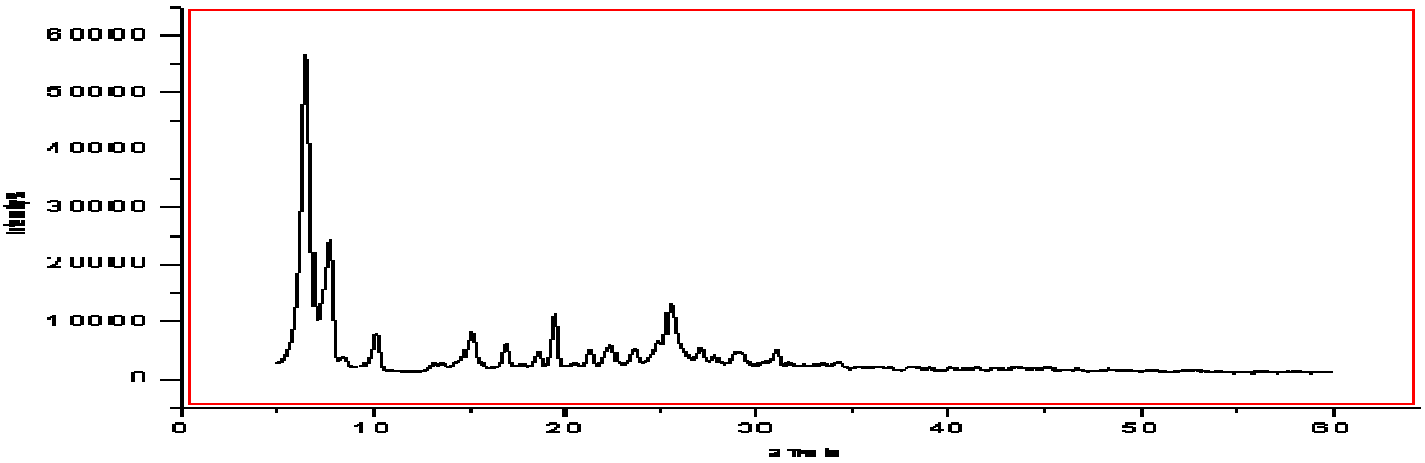

Figure-5
X-ray diffraction data of [CuL^1- L^2 $(H_2O)_2$] Complex

Table-6
Millar indices and interplanar distances of Cu (II) complex

h	k	l	2θ (Obs)	2θ (Cal)	d (Obs)	d (Cal)	Relative intensity
-1	0	0	6.51	7.04	13.55	12.53	322.18
-1	1	0	7.70	8.09	11.47	10.91	126.26
0	1	0	8.46	8.83	10.43	9.99	78.54
0	0	1	10.14	9.54	8.71	9.25	36.58
0	1	1	13.17	13.02	6.71	6.79	72.74
1	1	0	13.63	13.80	6.4	6.40	74.35

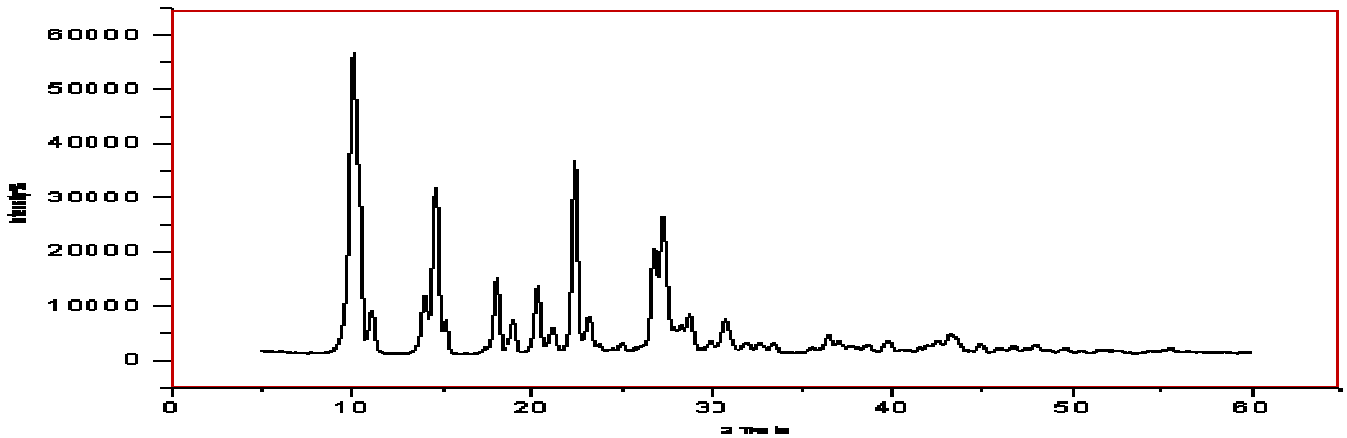

Figure-6
X-ray diffraction data of [ZnL^1-L^2 (H_2O)$_2$] Complex

Table-7
Millar indices and interplanar distances of Zn $^{(II)}$ complex

h	k	l	2θ (Obs)	2θ (Cal)	d (Obs)	d (Cal)	Relative intensity
-1	1	0	10.09	10.18	8.75	8.67	330.91
0	1	0	11.09	11.02	7.96	8.01	450.35
0	0	1	14.00	13.99	6.31	6.32	620.46
0	0	1	14.60	13.99	6.05	6.32	179.54
-2	1	0	15.12	15.85	5.85	5.58	34.80
0	1	1	17.96	17.84	4.93	4.96	79.11

Conclusion

All synthesized metal complexes like Mn(II), Fe(III), Co(II), Ni(II), Cu(II), Zn(II) complexes with N, O donor mixed ligand Schiff base derived from 4-methoxyphenylanaline, salicylaldehyde and o-vanilline studied by x-ray diffraction and it is found that Ni(II), Cu(II), Zn(II) complexes are amorphous in nature and Mn(II), Fe(III), Co(II) are crystalline in nature. They are monoclinic crystal system.

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