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X-RAY STUDIES OF CHELATE POLYMERS OF ADIPYL-BIS- PHENYL HYDRAZIDE (ABPH) AND SUCCINYL-BIS- 2,4 DINITROPHENYL HYDRAZIDE (SBDNPH) WITH TRANSITION METAL IONS.

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Abstract- Chelate polymers of Mn (II), &Zn (II) withAdipyl bis- phenyl hydrazide (ABPH)andSuccinyl bis- 2,4 dinitrophenyl hydrazide (SBDNPH) have been prepared and characterized with the help of elemental analysis, magnetic, Infra red & reflectance spectra, thermo gravimetric and X-ray diffraction studies. An attempt has been made to establish crystal structure of the chelate polymers on the basis of the data obtained with the help of X-ray powder diffraction technique. It is observed that the structures of both the polychelate selected are orthorhombic in nature.

On the basis of above studies their composition have been fixed and it has been observed that these polymers are having (ML)n composition. Since these chelate polymers are highly insoluble in almost all the organic solvents, including alcohol, acetone, chloroform, carbon tetrachloride, DMF, and DMSO, and have high thermal stability, they may be used as surface-coating materials and as a thermally stable material.

Keywords- Chelate polymers, ABPH, SBDNPH, Transition metal ions, X-ray diffraction.

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Introduction

The design and synthesis of metal–organic chelate polymers have attracted great interest from chemists because of their potential applications as functional solid materials, as well as their fascinating framework structures. These polymeric materials show many potential uses with desirable chemical and physical properties such as several applications as heterogeneous catalysts, separation of toxic and carcinogenic metal ions, water and soil remediation, semiconductors [1-8].Crystalline orthorhombic nature of chelate polymers of hydroxamic acid with first row transition metal ions was determined by X- ray diffraction technique[9] Literature survey reveals that coordination complexes of transition metal ions arecrystalline with octahedral, tetrahedral or square planar geometry[10].

Experimental

Chemicals: All chemicals used were of A.R. grade.

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^0 was obtained. Filtration reduces noise due to white radiation and increases resolution also.

Synthesis of Chelate Polymers synthesis of polymers has been carried out in two steps. Bis- ligands are prepared and characterized and then mixed with the equimolar quantities of metal acetates in suitable solvents.DMF was used as medium; the temperature of the reaction mixture was maintained at approximately 120°C to 150°C. The colored product obtained with different transition metals was filtered, washed thoroughly and dried. Repeated washings ascertained the purity, as the recrystallization is not possible due to insolubility.

Results and discussion

Due to their high unreactibility and insolubility the characterization is difficult, however the elemental analysis has been carried out carefully and with the help of their magnetic and spectral properties the tentative structure may be proposed for these polymers [11]. The similarities of IR spectra of all the poly chelates indicates

that the mode of coordination of the ligands is same in all cases irrespective of metal atoms used except the number of water molecule attached. The stereochemistry of polymers and the oxidation state of central metal atom were confirmed by the magnetic and electronic studies. The results of all these studies are in good agreement with each other.

X-ray diffraction of chelate polymers

X-ray diffraction analysis investigates structure through the use of diffraction. When X-radiation interacts with the electrons of a substance, the X rays are diffracted. The diffraction pattern depends on the wavelength of the X rays employed and on the structure of the object. Radiation of wavelength ~ 1 angstrom (Å), that is, of the order of atomic dimensions, is used to investigate atomic structure. The methods of X-ray diffraction analysis are used to study, for example, metals, alloys, minerals, inorganic and organic compounds, polymers, amorphous materials, liquids, gases, and the molecules of proteins and nucleic acids. X-ray diffraction analysis has been used most successfully to establish the atomic structure of crystalline substances because crystals have a rigid periodicity of structure and constitute naturally produced diffraction gratings for X rays[12].In the present investigation the x-ray diffractograms of chelate polymers are recorded. The 'd' values of reflection were obtained using Bragg's equation (n λ = 2d sin θ). These values of sin 2θ 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 Tables 1&2. The lattice parameters and Miller's indices were computed. The indexing was performed with the help of powdered diffraction package PDP, which is standard software for the interpretation of the x-ray powder diffractograms. The calculated and the observed 2θ values, the relative intensity, the interplaner distance d along with Miller's indices for corresponding angles are tabulated for the chelate polymer.Both the chelate polymers are found to be orthorhombic crystal.

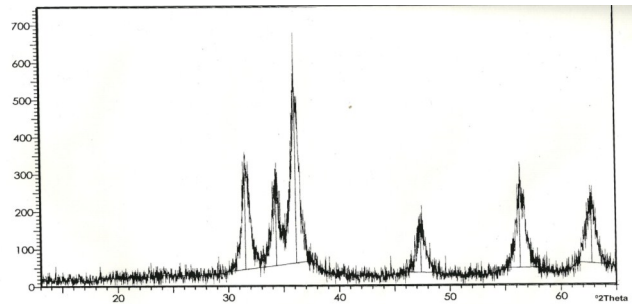

Fig. 1- X-Ray diffractograms of Zn (II) SBDNPH

Table 1- X-Ray diffraction data of Zn (II) SBDNPH Chelate Polymer

Peak no.	2θ Observed	2θ calculated	Delta 2θ	I/I_{max}	h	k	l	D Observed
2	31.57	31.57	0	59.03	1	1	1	2.831
4	36.13	36.13	0	100	11	1	0	2.484
5	47.39	47.34	0.05	24.65	13	0	2	1.917
6	56.33	56.33	0	44.27	1	2	1	1.632
7	62.77	62.77	0	34.49	13	4	1	1.479

Type of crystal system-Orthorhombic
Lattice Parameter: a = 39.263 A0, b = 3.459 A0, c = 4.968 A0
Volume of unit cell: 674.708 A0

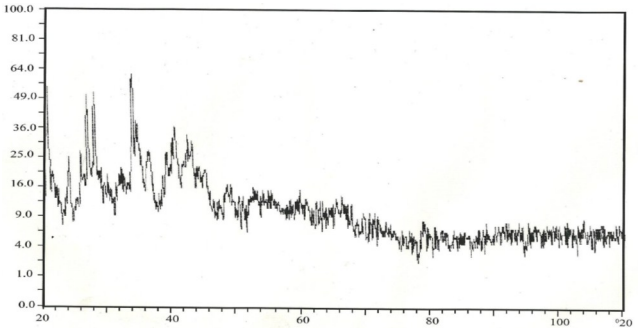

Fig. 2 -X-Ray diffractograms of Mn (II)ABPH

Table 2- X-Ray diffraction data of Mn(II) ABPH Chelate Polymer

Peak no.	2θ Observed	2θ calculated	Delta 2θ	I/I_{max}	h	k	l	D Observed
1	20.31	20.32	0	100	0	1	3	4.368
2	23.67	23.71	-0.04	21.6	0	4	3	3.756
3	25.53	25.53	0	19.4	1	7	1	3.486
4	26.62	26.62	0	56.33	2	2	2	3.346
6	33.48	33.48	0	73.23	3	1	0	2.674
8	38.49	38.54	-0.04	19.71	3	6	0	2.337
9	40	40.04	-0.03	35.21	2	6	4	2.252
10	42.08	42.12	-0.04	23.94	1	11	3	2.145
11	44.41	44.42	-0.01	11.26	3	3	4	2.038
12	48.13	48.18	-0.01	7.04	4	3	2	1.889
13	49.74	49.78	-0.05	5.63	0	4	7	1.832
15	77.86	77.89	-0.03	4.6	5	15	0	1.226

Type of crystal system-Orthorhombic
Lattice Parameter: a = 8.059 A0, b = 28.299 A0, c = 13.262 A0
Volume of unit cell: 3024.5535 A0

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