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X- RAY DIFFRACTION STUDIES AND ANTIBACTERIAL ACTIVITY OF SCHIFF BASE METAL COMPLEXES DERIVED FROM DEHYDROACETIC ACID

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ABSTRACT

The metal complexes with bidentate Schiff base ligands derived from dehydroacetic acid and 2,4 –dibromoaniline were characterized by X Ray diffraction spectral studies. The Antibacterial activities of the ligands and their metal complexes have been screened in vitro against against *E. coli* and *Staphylococcus species* using Cefixime as standard. The toxicity of metal chelates follow the order Cu (II) > Co (II) > Mn (II) > Fe (III) > Ni(II).

KEYWORDS: Schiff base, transition metal complexes, X-ray diffraction, Antibacterial activity.

INTRODUCTION:-

The X–ray diffraction is a powerful method to understand the structure of the compound. X–ray method, can determine the structure and symmetry properties of complexes. It gives information of inter atomic distance, bond angles and electronic arrangement in a complex. It is possible to observe the molecules indirectly by using X-ray radiation of very small wavelength of the order of 10^{-8} cm and involves the studies of crystalline solids by the phenomenon of X-ray diffraction. When the beam of monochromatic X-ray strikes the plane of atoms in the crystal, an interference phenomenon is observed.

The disease causing microorganisms are called pathogens. The growth of pathogens and other harmful bacteria is controlled by antibacterial agents which is essential for survival of the mankind. *Escherichia coli* is a gram-negative rod shaped bacteria is one of the main species of bacteria living in the lower intestines of mammals known as gut flora. When located in the large intestine it actually assists with waste processing vitamin K production and food absorption.

Staphylococcus is genus of gram –positive bacteria. They appear round form in grape like cluster.[37] *Staphylococcus aureus* is the most common cause of infections. *Staphylococcus aureus* is a spherical bacterium frequently living on the skin or in the nose of a healthy person that can cause a range of illnesses from minor skin infections (such as pimples, boils and cellulitis) and abscesses to life-threatening diseases such as *pneumonia* meningitis endocarditis Toxic shock syndrome (TSS) and *septicemia.*

MATERIAL AND METHODS:-

The X-ray powder diffraction of representative metal complexes was scanned at Department of Physics Shri chhatrapati Shivaji College,Omerga on Rigaku, Miniflex Desktop X-ray Diffractometer attached

to a digital computer along with graphical assembly in which Cu-K radiation source connected with the tube Cu/30Kv/15 mA producing 1.543 A^0 wavelength radiation was used with scanning rate 2^0/min. Each test sample of 200-300 mesh size weighing minimum amount 10 mg was spread in the form of film and spectra were scanned in the range of $2\theta = 20$ to 80^0.

The culture of human pathogenic bacteria *Escherichia coli* and *Staphylococcus species* were collected from department of Microbiology , Adarsh college, Omerga Aseptic techniques were employed to prepare the culture medium of all the test microorganism and the strains were maintained on Nutrient Agar slant at 4°C. The Nutrient Agar medium was used for screening the antibacterial activity of *E. coli* and *Staphylococcus species*

EXPERIMENTAL:-

The X-ray diffractograms of representative metal complexes were scanned in the range $5\text{-}100^0$ at λ=1.543 Å. The diffractograms and associated data depict the 2θ value for each peak relative intensity and observed inter planar spacing (d-values). The information gathered from the diffractogram and associated data was used for indexing the pattern and to find out the unit dimensions and space group. The positions of each reflection with the intensity are recorded. Inter planar spacing d_{hkl} were calculated from 2θ values using the relation $d=n\lambda/\sin\theta$. The computer programme used for indexing data was Powder-X. The preliminary data in the form of 2θ and intensities fed to the computer programme and all differences (d-obs.) are calculated as required. All the possible combinations of h k l plane and d values observed are arranged in the decreasing order.

In this programme all the essential features of X-ray programme are present and in addition it calculates the deviation in lattice parameters a b c in terms of Å unit and α, β and γ in terms of degree minutes. When the system of compound is unknown the observed data is first tested with the isometric chart and Hul-Devy`s curve for tetragonal and hexagonal system. If these tests are proved to be negative then the data is subjected to programme bar and an attempt is made for the existence of lower symmetry and then data can be index in an orthorhombic system. The failure of all these tests is an indication for the existence of lower symmetry and then data can be indexed on either monoclinic or triclinic system. The precise lattice parameters and the deviation are obtained from Powder-X programme.

Antibacterial activities were studied by the well diffusion method. The nutrient agar medium and petri plates were used. The compounds were dissolved in DMSO making known stock solution. The petri plates fill with nutrient agar media seeded with of *E. coli* and *Staphylococcus species* separately and left to solidification well was dug in agar media using sterile metallic borer in each plate, the test solution filled using micropipette. The plate were incubated 24 hours at 35^OC.The inhibition zones were recorded in terms of diameter of zone of inhibition of growth of bacteria. The antibacterial activity of a common standard drug *Cefixime* was also recorded maintaining the same protocol at the same concentrations and solvent.

RESULTS AND DISCUSSION:-

The X-ray powder data of all the main peaks have been indexed independently by trial and error method. The indexed powder diffraction data the unit cell data crystal lattice parameters of complexes are presented in Table 1. The data was indexed such that the standard deviation in the lattice parameter reaches minimum value. The standard deviation observed for Cu(II) complexes is within permissible limit of 2%. The crystal volume is obtained from indexing of the diffraction pattern. The Z value was calculated and rounded up to the nearest whole number. The porosity percentage was calculated from the observed and calculated densities. The density calculated from diffraction data and the observed density was found to be very close to each other indicating perfection in indexing. The observed density of Cu(II) complexes is $2.8823 gcm^{-3}$ and calculated density is $2.7672\ gcm^{-3}$ and the porosity percentage is 3.993.The complexes crystallises in the monoclinic crystal system with 2 molecules per unit cell. The probable space group is P2/m.

Table1:- Indexed X-ray Diffraction Data of Cu(II) Complex of Ligand L

Miller Indices h k l	sin²θ obs.	sin²θ calc.	Delta (Å)	2θ obs.	2θ calc.	d obs (Å)	Relative intensity
0 0 1	0.004854	0.004807	0.000047	7.990	7.951	11.0567	76.00
1 0 1	0.010498	0.010515	-0.000017	11.762	11.771	7.5179	100
0 1 0	0.010908	0.010884	0.000025	11.990	11.976	7.3754	86.00
1 1 0	0.014811	0.014763	0.000048	13.980	13.958	6.3295	12.00
0 1 1	0.015877	0.015691	0.000186	14.477	14.392	6.1133	10.00
-2 0 3	0.047796	0.047805	-0.000009	25.256	25.259	3.5234	18.00
-1 2 1	0.050334	0.050391	-0.000057	25.930	25.945	3.4334	08.00
3 1 1	0.056066	0.056091	-0.000025	27.393	27.400	3.2532	17.00
2 2 0	0.059243	0.059051	0.000192	28.175	28.128	3.1648	07.00
4 0 0	0.063507	0.062068	0.000019	28.857	28.853	3.0915	18.00
1 1 3	0.063507	0.063513	-0.000006	29.192	29.194	3.0567	09.00

Unit cell data and crystal lattice parameter

Crystal System: **Monoclinic P**

a = 12.654726 ± 0.0099 Å
b = 7.383708 ± 0.0072 Å
c = 11.368162 ± 0.0094 Å
Density (d_{obs}) = 2.8823 g cm^{-3}
Density (d_{cal}) = 2.7672 g cm^{-3}
Porosity % = 3.993
Particle size = 308.3721 Å

α = 90.00 ± 0.000 DEG
β = 102.227463 ± 0.087DEG
γ = 90.00 ± 0.000 DEG
Z = 2
Space group = P 2/m
Volume = 1038.13 $Å^3$

Table2.shows the Antibacterial activity of ligands and their metal complexes(% inhibition) The synthesized ligands and their metal complexes were screened for their antibacterial activity against bacteria *Staphylococcus aureus* (gram positive) and *Escherichia Coli* (gram negative) organisms. The ligands and their metal complexes possess antibacterial activity at 500 and 1000 ppm concentrations. The biological activity of free ligands is lower than their respective complexes. Also the activity increases with the increasing concentration of complexes. However the activities are lower than standered antibiotic *Cefixime*. The toxicity of metal chelates follow the order Cu(II)>Co(II) >Mn(II)>Fe(III)>Ni(II).

It is observed that the halogen substituted ligands and there metal complexes show more activity than nonsubstituted ligands and its metal complexes.

Table 2:-Antibacterial activity of ligands and their metal complexes (% inhibition)

Test Compound	Diameter of inhibition zone (mm)			
	Escherichia coli		*Staphylococcus aureus*	
	500 ppm	1000 ppm	500 ppm	1000 ppm
Ligand	10 (25.00)	14 (31.11)	08 (19.51)	12 (28.88)
Cu Complex	21 (52.50)	23 (51.11)	19 (46.34)	24 (53.33)
Ni Complex	11 (27.50)	13 (28.88)	12 (29.26)	15 (33.33)
Co Complex	19 (47.50)	20 (44.44)	17 (41.46)	22 (48.88)
Mn Complex	16 (40.00)	18 (40.00)	16 (39.02)	17 (37.77)
F Complex	13 (32.50)	16 (35.55)	14 (34.14)	18 (40.00)
Ceffixime	40	45	41	45

CONCLUSION:-

X-ray powder pattern investigation suggests that all the complexes are crystalline in nature. The qualities of XRD were excellent which qualitatively suggests very high degree of crystallanity for metal complexes. Various strong reflections in the XRD pattern were used to calculate crystal lattice parameters by using WinPLOTR computer program. Most of the metal complexes have monoclinic crystal systems with space group P 2/m. The metal complexes show better antibacterial activity than their respective ligands.

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