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Journal of Chemical and Pharmaceutical Research

ISSN No: 0975-7384
CODEN(USA): JCPRC5

J. Chem. Pharm. Res., 2011, 3(1):72-78

Synthesis, Characterization and Antimicrobial Screening of Fe (III)-Schiff Base Complex

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ABSTRACT

The study report the synthesis of Fe(III)-with 4-benzylidine amino benzoic acid and their characterization using FTIR and magnetic susceptibility studies. Our study reveals the presence of different mode of linkages of the ligand with Fe(III).The comparison of the IR Spectra of the ligand imply that the ligand is bidented with the carboxyl group and azomethine nitrogen as the two coordination sites. The complexes exhibit an identical pattern suggesting them to be isostructural with six coordinated spin free octahedral complexes. The complexes have been screened for their antibacterial and antifungal activity. The result of this study shows that the Fe(III) complex is effective against fungal pathogen than the bacterial pathogens.

Keywords: Fe (III)-schiff base complex, synthesis, characterization, antimicrobial activity

INTRODUCTION

Coordination complexes are gaining increasing importance in recent years, particularly in the design of repository slow release or long acting drugs in nutrition and in the study of metabolism. Chemists have reported on the chemical, structural and biological properties of Schiff bases [1,2]. Schiff bases are characterized by the –N=CH (imine) group. Heterocyclic Schiff base complexes play important role in various biological activities [3-6]. Schiff bases are active against a wide range of organism. viz. *Mycobacteria, Erysiphe gramini,Bacillus magatherium, Bacillus subtilis, Escherichia coli, Protius valgaris and Plasmopora viticola.*

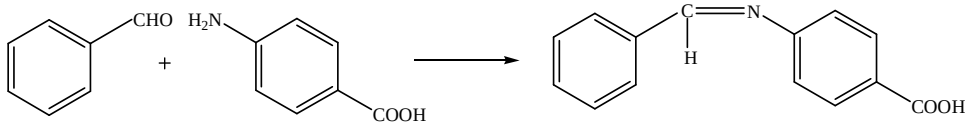
In comparison with antibacterial activity, antifungal activity is studied much more because bacteria can achieve resistance to antibiotics through biochemical and morphological modifications [7]. Bacteria are several types of microscopic or ultra-microscopic single-celled organisms occurring in enormous numbers everywhere in nature, not only in land, sea and air, but also on or in many parts of the tissue of plants and animals, and forming one of the main biologically interdependent groups of organisms in virtue of the chemical changes which many of them bring about, viz. all forms of decay and the building up of nitrogen compound in the soil

A wide number of researches report the synthesis and characterization of many metal complexes of Schiff bases derived from aromatic aldehyde and substituted anilines. It is well known from the literature that there is no report of Schiff base complexes of Fe(III) formed from 4-benzylidene amino benzoic acid. Hence an attempt has made to the synthesis of Fe(III)- Schiff base complexes with 4-benzylidene amino benzoic acid.

EXPERIMENTAL SECTION

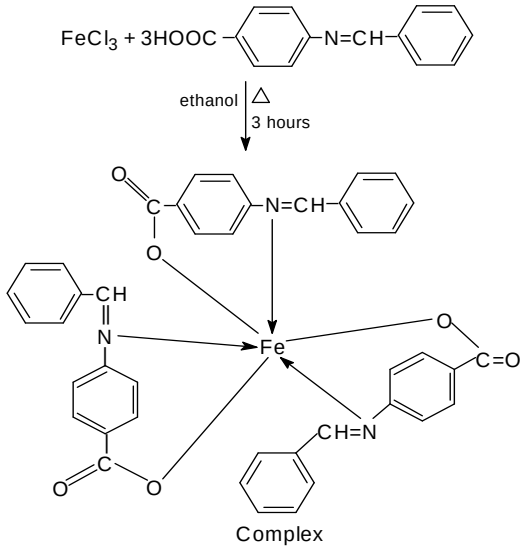
Preparation of ligand

4-amino benzoic acid (1mm) and benzaldehyde (1mm) were refluxed together for 3 hour in the presence of a drop of concentrated HCl. The volume of the mixture was reduced to half. It was then cooled in a refrigerator when light yellow crystals separated. It was filtered, washed several times with cold ethanol and dried.



Synthesis of Fe (III) complex

The complex was prepared by carrying out the *insitu* reaction of 4-benzylidene amino benzoic acid and the metal salt ferric chloride. 4-amino benzoic acid (1mm) and bezaldehyde (1mm) were taken together in ethanol (30ml) and refluxed for an hour after which ferric chloride (1mm) in ethanol was added and refluxing is continued for another 3 hours. Upon cooling microcrystalline ferric complex was precipitated. It was filtered, washed thoroughly with ethanol and dried. The yield was quantitative. The structure of the complex is-



Characterization of ligand

IR spectra were recorded in KBr medium on a Perkin-Elmer 783 spectrophotometer. UV-Visible spectrum of complex was recorded in DMSO on a shimadzu UV-1601 spectrophotometer. The ligand is expected to be bidentate, the possible coordination sites being the azomethine nitrogen and the carboxyl group. The N-H stretching absorption in the free ligand occurs at 3330 cm^{-1}. The ligand band due to C=N mode appear at 1612 cm^{-1}.

Region in cm^{-1}	Observed Frequency in cm^{-1}	Correlation
3000-2600	2560	OH stretching (–COOH)
1700-1550	1653	C=N stretching
1300-1100	1141	C–N stretching
1750-1550	1710	C=O stretching

Characterization of Fe(III) complex

IR spectra were recorded in KBr medium on a Perkin-Elmer 783 spectrophotometer. UV-Visible spectra of complex were recorded in DMSO on a shimadzu UV-1601 spectrophotometer. The magnetic property of the metal complex was studied by magnetometer. C=N mode undergo a shift to lower frequency and the band due to is observed as a strong peak in the region 1600 cm^{-1}, suggesting participation of exocyclic azomethine nitrogen in the complex formation.

Region in cm^{-1}	Frequency in cm^{-1}	Correlation
400-650	650	M-C band
1700-1550	1653	C=N stretching
1300-1100	1141	C–N stretching
1750-1550	1710	C=O stretching

Antimicrobial activity

Mueller Hinton agar (Beef extract 0.2g, Peptone 1.75g, starch 0.15g, agar2.0g, distilled water 100ml, pH 7.5) prepared with lawn culture using desired test organisms. The inoculated plates were kept aside for few minutes. Using well cutter two well are made in those plates at required distance. In each step of well cutting. The well cutting was thoroughly wiped with alcohol. Using one of different solvents with selected chemically sterilized micropipette, 20 extract was added into one well and in another well the same volume of corresponding controls (solvent without chemical extract) were added. After 24 hours for the diffusion, the Mueller Hinton agar plates were incubated at 37oC for antimicrobial analysis. After incubation, the zone of inhibition was analyzed and recorded.

Antifungal activity

The sterilized Sabouraud's dextrose agar medium (Dextrose 4.0g ,Mycological peptone 1.0g, agar 2.0g pH 5.0 ,distilled water 100ml)was poured to a Petri dish in a uniform thickness and kept aside for solidification. Using sterilized swabs, even distribution of lawn culture was prepared using desired fungi such as *A.nigar* ,P.notatum, C.albicans,C.tropicalis. in SDA plates. The inoculated plates were kept aside for few minutes. Using well cutter, two wells are made in at required distance. In each step of well cutting, the well cutter was thoroughly wiped with alcohol using sterilized micropipette, one of different solvents with compound was added in to one well and in another 20µ well the same volume of corresponding controls (solvent without compound) were added. After diffusion, the plates were incubated at room temperature for 24-48 hours. After incubation, the inhibition of growth was analyzed and results were recorded.

Determination of minimum inhibitory concentration

One ml of extract (1mg/ml) was incorporated into one ml of nutrient broth and Sabouraud's dextrose broth and serially diluted to obtain concentration of 1000µg/ml, 500µg/ml, 250µg/ml, 125µg/ml and 62.5µg/ml respectively. 20 bacterial, fungal inoculums were added to each of the test tubes. The tube without the extract served control. The tubes were inoculated at room temperature and readings were recorded after a period of 24 hrs for bacteria and 3 days for fungi. MIC was recorded as the lowest concentration of the extract at which no visible growth of the bacterial and fungal occurred after a period of seven days incubation.

RESULTS AND DISCUSSION

Magnetic susceptibility

The room temperature magnetic moment of Fe(III) chelate is found to be 1.43BM indicating paramagnetic, low spin d^2sp^3 complex with ground state term 2D. The magnetic moment value can be calculated as follows (Table1).

a) Calibration of Instrument

Sr. No.	Current passes through 50% $FeCl_3$ Solution (Ampere)	Magnetic field (Gauss)	Reading
1	0	0	1.19
2	1	85	1.13
3	2	169	1.20
4	3	252	2.04
5	4	324	1.47
6	5	381	1.52

b) Observation table for calculation of magnetic susceptibility of Fe(III)-Schiff complex in Dimethyl Formamide

Sr. No.	Current passes through Experimental Solution (Ampere)	Magnetic field (Gauss)	Reading
1	0	0	1.41
2	1	85	1.45
3	2	169	1.49
4	3	252	1.52
5	4	324	1.54
6	5	381	1.56

Observation:

1. Weight of Density Bottle = 6.86
2. Weight of Density Bottle + Complex + Solvent = 19.49
3. Weight of Density Bottle + Water = 20.61

Calculation:

a) Calculation of Density of Solution by formula

$$\rho = \rho_{\text{water}} \frac{(c-a)}{(b-a)}$$

b) Calculation of magnetic susceptibility by using formula

$$\chi = \frac{2[1 + a / A]}{H} \times \rho\, gh$$

A = area of wide limb.
a = area of narrow limb.
H = magnetic field (Gauss).
e = density of solution.
g = acceleration due to gravity
h = difference in the height of solution raises

c) The magnetic moment is calculated by using formula.

$$\mu_{eff} = 2.84\sqrt{(xm) \times T}\, B.M.$$

$$\mu_{eff} = Us = \sqrt{n(n+2)}\, B.M.$$

The IR and magnetic susceptibility studies of the complex indicate that it is an octahedral complex of the type Fe-L3 (Fig .1).

Antimicrobial activity:

Many physical techniques play their role in the elucidation of the geometric and electronic structure of a metal complex. The Fe(III)- metal complexes were effective against all the test organisms, both in ethanol and methanol extract form. The methanol extract was more active against the test organisms than the ethanol extract. The antimicrobial activity of ethanol extract of selected complexes in well method was performed and was found to be effective.

Table : Antimicrobial activity of methanol extract of Fe(III) complex (Zone of inhibition in mm)

S. No.	Name of the Pathogens	Fe(III) (mm)	Ligand (mm)
1	*S. aureus*	16	11
2	*S. typha*	10	No Zone
3	*E. coli*	10	11
4	*P. aeruginosa*	12	13
5	*S. dysenteriae*	14	11
6	*A. niger*	No Zone	14
7	*P. species*	15	16
8	*C. albicans*	15	10
9	*C. tropicalis*	11	14

Antifungal activity

The minimum inhibitory concentration of ethanol extract of Fe(III) against *S. aureus* was 500µg/ml, *P. aeruginosa* was 1000µg/ml, *S. dysenteriae* was 500µg/ml, for *A. niger* 1000µg/ml, *P. notatum* was 1000µg/ml, *C. albicans* was 500µg/ml, and for *C. tropicalis* was 500µg/ml. It is calculated by using formula –

$$\text{MIC of bacteria} = \frac{\begin{array}{c}\text{amount of antibiotic per disc required to inhibit}\\ \text{the test bacterium at given size of zone}\end{array}}{\begin{array}{c}\text{amount of antibiotic per disc required to inhibit}\\ \text{the control strain at the same size of zone}\end{array}} \times \text{MIC of control strain}$$

Table: Minimum inhibitory concentration of ethanol extract of Fe(III) on selected bacteria and fungi

S.No.	Name of the Pathogens	Minimum inhibitory concentrations					
		2000	300	220	120	42.3	11.25
1	*S. aureus*	–	+	+	+	+	+
2	*S. typha*	–	+	–	–	–	–
3	*E. coli*	+	+	–	–	–	–
4	*P. aeruginosa*	+	–	+	–	–	–
5	*S. dysenteriae*	–	+	+	–	–	–
6	*A. niger*	–	–	+	–	–	–
7	*P. species*	–	+	+	–	–	+
8	*C. albicans*	+	–	+	–	+	+
9	*C. tropicalis*	–	+	–	–	–	+

Various studies [8] have shown a relationship between the metal ions and their metal complexes as antitumour and antibacterial agents [9], which is a subject of great interest. The inorganic pharmacology started to be an important field with more than 25 inorganic compounds being used in thereby as antibacterial, antiviral and anticancer drugs [10]. It was seen that the biologically active compounds become more bacteriostatic and carcinostatic upon chelation with metal ions. Schiff bases have also attracted considerable attention in terms with their chelating abilities and analytical applications [11, 12].

CONCLUSION

Thus the present studies show the significant Antimicrobial activity to *S. aureus* and *C. albicans*. The further study is needed for the identification of active site. It is essential to predict the leading molecule and drug like property at the onset of drug design which will helps in drug development. Study of the magnetic moment and infrared spectra can often provide the most detailed information about the formation of complex.

Acknowledgement
Authors are very thankful to UGC, New Delhi for providing financial assistance to this project work.

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