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# Spectral, Thermal and Biological Studies of Mn(II) and Fe(II) Complexes of Schiff Base derived from *p*-Dimethylamino-benzaldehyde and Anthranilic Acid

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The solid complexes of Mn(II) and Fe(II) with tridentate Schiff base, *p*-dimethylaminobenzylidene anthranilic acid (PDBAA) derived from *p*-dimethyl aminobenzaldehyde and anthranilic acid have been synthesized and characterized by elemental analysis, molar conductivity, IR, electronic spectra, magnetic susceptibility, X-ray diffraction studies and by thermal studies. From the analytical and spectral data, the stoichiometry of the complexes has been found to be 1 : 1 (metal : ligand). The metal chelates have a general formula $[M(PDBAA)\cdot(H_2O)_3]$ (M = Mn(II) or Fe(II)). Electronic absorption data suggest octahedral geometry for these metals chelates. IR spectral data suggest that the ligand (PDBAA) behaves as a monobasic tridentate ligand with N : N : O donor sequence towards metal ions. Magnetic susceptibility measurement indicates paramagnetic behaviour for both the complexes. From electronic spectral data, various ligand field parameters such as Dq, B′, β and LFSE have been calculated for both the complexes, which are in good agreement with that of proposed geometries of the complexes. X-ray diffraction powder pattern indicates that these chelates are crystalline and crystallize with monoclinic system. The antifungal and antibacterial activities of the ligand and the complexes have been screened against several fungi and bacteria.

**Key Words: Transition metal, Schiff base, Characterization.**

## INTRODUCTION

The coordination behaviour and preparative techniques of Schiff bases have received much attention in recent years[1–3], because of their preparative accessibility, structural variety, varied denticity and subtle steric and electronic control on their framework leading to complexes of different nuclearities in variable oxidation states of the metal ions. Mononuclear complexes derived from Schiff bases proved to be valuable catalysts in various organic reactions, especially in enantioselective transformation[4]. It has been acknowledged that the primary event in mammalian vision is a light-initiated *cis*-to-*trans* isomerization of the retinal chromophore bond, *via* a protonated Schiff base, to a lysine residue in the opsin apoprotein[5, 6]. Different Schiff bases and their metal complexes find applications

as antituberculosis[7], anticonvulsant[8] and as potential anthelmintic[9] agents. A Schiff base derived from *p*-dimethylaminobenzaldehyde and anthranilic acid was reported[10] to possess such biological activity. In continuation of our earlier work[11] here we are reporting the synthesis, characterization and biological activity of manganese(II) and iron(II) complexes of Schiff base *p*-dimethylamino-benzaldehyde anthranilic acid (PDBAA) in the present communication.

## EXPERIMENTAL

Metal salts used for synthetic and analytical work were AR/LR grade. All reagents and solvents used were purified by standard method and dried before use. The ligand PDBAA is synthesized by the same process as reported earlier[11].

### Synthesis of metal complexes [M(PDBAA)·$(H_2O)_3$] (M = Mn(II) or Fe(II))

1% (w/v) Schif base solution in hot distilled water was prepared. To this solution, metal salt solution (1 mg/mL) prepared in distilled water was added slowly with continuous stirring. The mixture was heated for 5 min on a burner; no precipitate was obtained. Then the mixture was cooled to room temperature and pH was raised with alcoholic ammonia (for Mn(II) complex pH was raised to 8.0 and for Fe(II) complex pH was raised to 5.0). The metal complexes were separated out under these conditions. The precipitate was suction filtered, washed with distilled water and final wash was given with absolute ethanol. Each metal complex was dried under vacuum at room temperature and recrystallised from acetic acid.

Elemental analyses of metal complexes were determined by the method as reported in literature[12]. Solubility of the metal complexes was investigated in various polar and non-polar solvents. Molar conductivity was measured in DMSO ($10^{-3}$ M) solution using Toshniwal TSM-15 conductometer. Magnetic susceptibility measurements were made at room temperature on Gouy balance using $Hg[Co(SCN)_4]$ as calibrant. Electronic absorption spectra were recorded on a Shimadzu UV-2100 spectrophotometer, while IR spectra (KBr pellets) were recorded on a Shimadzu FTIR-4200 spectrometer in the range 4000–400 $cm^{-1}$. X-ray diffractograms were recorded on X-ray diffractometer supplied by M/s Philips, Holland. TG and DT analyses of metal complexes were carried out in nitrogen atmosphere in the range of 25–900°C on Shimadzu DTG-50 with a heating rate 10°C $min^{-1}$ using alumina as a standard.

### Microbial activities

All metal complexes were screened against gram positive bacteria (*Staphylococcus aureus*), gram negative bacteria (*Escherichia coli*) at various concentrations (0.125–1.0 mg/mL) in DMSO as a system by agar plate method using echinocandin as a control.

The liquid culture was used as an inoculum. This inoculum was added to Sabouraud dextrose agar. This was then poured into petri-dishes and allowed to

solidify. Then holes of 6 mm diameter were punched carefully using a sterile cork-borer and these were completely filled with the test solutions. The plates were incubated for 24 h at 37°C. Then zones of inhibition were recorded against the desired micro-organisms.

## RESULTS AND DISCUSSION

The analytical data of the complexes and their magnetic moment values are given in Table-1 whereas ligand field parameters are given in Table-2. The complexes are coloured solids, found to be stable at room temperature. However, these decompose at temperatures higher than 280°C. The metal complexes are soluble in DMSO, while they are insoluble in methanol, ethanol, chloroform, carbon tetrachloide, acetone, ethyl acetate, petroleum ether, diethyl ether, 1,4-di-oxane, DMF, etc. The molar conductivity values of $10^{-3}$ M solutions of the complexes (0.84–0.91 s $cm^2$ $mol^{-1}$) in DMSO at room termperature show non-electrolytic nature of the complexes[13].

The IR spectra of both the metal complexes show broad band of medium intensity at 3200 $cm^{-1}$ which have been assigned to the ν(OH) mode of coordinated water molecule. The new peak at *ca.* 860 $cm^{-1}$ suggests the presence of coordinated water molecule[14] which is further corroborated by TG and DT analyses. The infrared spectrum of the ligand shows a broad band at 2800 $cm^{-1}$ which is a characteristic of ν(OH) stretching of carboxylic[15] group. In the spectra of metal complexes the broad band disappears indicating deprotonation of the ligand and involvement of carboxylic oxygen atom in bonding with metal ion. The ligand shows characteristic band at 1680 $cm^{-1}$ which can be assigned to ν(C═O) mode of carboxylic group. This band is shifted to 1600 $cm^{-1}$ in the corresponding spectra of metal complexes. Another band appearing at 1615 $cm^{-1}$ in the spectrum of ligand is considerably lowered to 1540 $cm^{-1}$ in the corresponding spectrum of metal complex indicating involvement of the azo-methine nitrogen atom during chelation[16]. The weak band appearing at 518 and 420 $cm^{-1}$ can be assigned to ν(M—N) and ν(M—O) stretching vibrations, respectively[17, 18].

The electronic absorption spectrum of ligand shows three high intensity bands lying at 29499, 40650 and 46083 $cm^{-1}$ assigned to $n \rightarrow \pi^*$, $\pi \rightarrow \pi^*$ and $\sigma \rightarrow \sigma^*$ transitions respectively[19].

The electronic absorption spectrum of Mn(II) complex indicates the absorption bands at 18382, 29940 and 38610 $cm^{-1}$ which can be assigned to the transitions $^6A_{1g} \rightarrow {}^4T_{1g}$, $^6A_{1g} \rightarrow {}^4T_{2g}$ and charge transfer band respectively[20]. The Mn(II) complex exhibits room temperature magnetic moment value at 5.92 B.M. which is the same as spin-only value of 5.92 B.M. expected for an s = 5/2 system. The above magnetic moment value is indicative of the presence of $^6A_{1g}$ ground state associated with an octahedral[21] structure.

The Fe(II) complex exhibits three main absorption bands around 20000, 29499 and 38610 $cm^{-1}$ which can be assigned to the transitions $^6A_{1g} \rightarrow {}^4T_{1g}$,

TABLE-1
ELEMENTAL ANALYSIS, PHYSICAL AND SPECTROSCOPIC DATA OF THE SCHIFF BASE AND ITS Mn(II) AND Fe(II) COMPLEXES

| Compound (Colour) | m.f. (m.w.) | Yiel % (m.p., °C) | Molar conductivity ($s\ cm^2\ mol^{-1}$) | $\mu_{eff}$ (B.M.) | Found (Calcd.), % | | | | IR ($cm^{-1}$) | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | | | | | C | H | N | M | ν(O—H) —COOH | ν(O—H) Coordinated $H_2O$ molecule | ν(C=O) | ν(C=N) | ν(M=N) | ν(M—O) |
| Ligand PDBAA (Orange) | $C_{16}H_{16}N_2O_2$ (268) | 71 (186) | — | — | 70.19 (71.64) | 5.18 (5.97) | 9.72 (10.45) | — | 2800 | — | 1680 | 1615 | — | — |
| [Mn(PDBAA)$(H_2O)_3$] (Light brown) | $C_{16}H_{21}N_2O_5Mn$ (375.94) | 63 (>300) | 0.84 | 5.92 | 50.97 (51.07) | 5.51 (5.58) | 7.38 (7.45) | 14.56 (14.61) | — | 3300 | 1615 | 1590 | 518 | 420 |
| [Fe(PDBAA)$(H_2O)_3$] (Dark brown) | $C_{16}H_{21}N_2O_5Fe$ (376.85) | 61 (>300) | 0.91 | 4.82 | 50.92 (50.95) | 5.53 (5.57) | 7.38 (7.43) | 14.80 (14.82) | — | 3300 | 1615 | 1590 | 518 | 420 |

$^6A_{1g} \rightarrow {}^4E_g$ and charge transfer band respectively[22]. The room temperature magnetic measurements of Fe(II) complex exhibit a magnetic moment of 4.82 B.M. which suggests the presence of four unpaired electrons with high-spin Fe(II) complex[23, 24].

The ligand field parameters like ligand splitting energy (10 Dq), Racah inter-electronic repulsion parameter (B), covalent factor (β) and ligand field stabilization energy (LFSE) has been calculated. These values are compiled in Table-2. The B and β values have been calculated following standard equations[25, 26]. The B-values are lower than the free ion values, thereby indicating the orbital overlap and delocalization of *d*-orbitals. The β-values obtained are less than unity suggesting considerable amount of covalent character of the metal-ligand bonds[25].

TABLE-2
ELECTRONIC SPECTRAL DATA AND LIGAND FIELD PARAMETERS OF THE COMPLEXES

| Compound | Electronic absorbance ($cm^{-1}$) ($\varepsilon \times 10^4$) $dm^3$ $mol^{-1}$ $cm^{-1}$ | Band Assignments | Ligand field parameters | | | | |
|---|---|---|---|---|---|---|---|
| | | | Dq ($cm^{-1}$) | $\nu_2/\nu_1$ | B ($cm^{-1}$) | β | LFSE (kcal $mol^{-1}$) |
| PDBAA | 29499 (2.1) | $n \rightarrow \pi^*$ | — | — | — | — | — |
| | 40650 (1.4) | $\pi \rightarrow \pi^*$ | | | | | |
| | 46083 (3.1) | $\sigma \rightarrow \sigma^*$ | | | | | |
| $[Mn(PDBAA)(H_2O)_3]$ | 18382 (0.01) | $^6A_{1g} \rightarrow {}^4T_{1g}$ | 18382 | 1.62 | 894 | 0.93 | 52.66 |
| | 29940 (0.92) | $^6A_{1g} \rightarrow {}^4T_{2g}$ | | | | | |
| | 38610 (0.88) | Charge transfer | | | | | |
| $[Fe(PDBAA)(H_2O)_3]$ | 20000 (0.01) | $^6A_{1g} \rightarrow {}^4T_{1g}$ | 20000 | 1.48 | 541 | 0.51 | 57.30 |
| | 29499 (2.05) | $^6A_{1g} \rightarrow {}^4E_g$ | | | | | |
| | 38610 (1.66) | Charge transfer | | | | | |

Thermal studies (TG and DTA) of the complexes revealed loss of three moles of coordinated water per mole of complex in the case of Mn(II) and Fe(II) complexes. The loss of coordinated water in both the complexes is a single step process, indicated by sharp exothermic peak. The removal of coordinated water molecule from the metal complexes ranges around 280°C. The TGA curve shows sharp decrease at around 280°C suggesting slow but steady decomposition of the compound. The decomposition continues up to the temperature 700–800°C and the horizontal nature of the curve indicates the presence of thermally stable residual metal oxide. The thermal analysis data of both the complexes are summarized in Table-3.

The X-ray diffraction pattern for both the metal complexes has been determined between 2θ range from 5–80° and data has been summarized in Tables 4 and 5. The major refluxes have been indexed by using computer software. The data indicate the monoclinic crystal systems for both the complexes.

**Biological Activity**

The ligand and its metal complexes were screened for antibacterial and

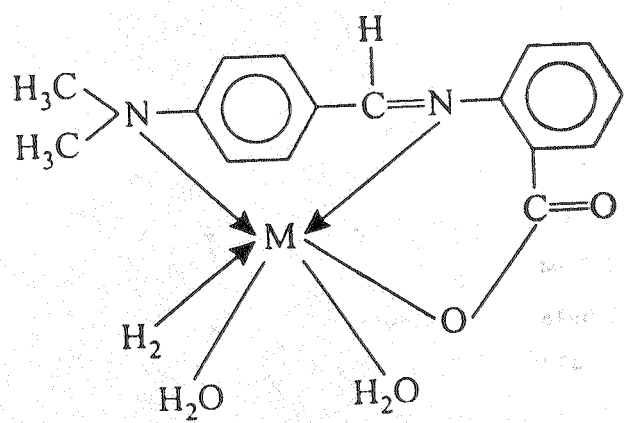
antifungal activities by agar plate method using echinocandin as control. The antibacterial activity of the compounds was tested against *E. coli* (gram-negative) and *Staphylococcus aureus* (gram-positive). The antifungal activity of the compounds was tested against *Candida albicans, Candida krusei, Candida glabrata* and *Aspergillus fumigatus*. The concentration used for testing was 0.125–1.0 mg/mL in DMSO.

TABLE-3
THERMAL DATA OF METAL COMPLEXES

| [Compound] m.f. (m. w.) | Temperature at which the coordinated water was removed (°C) | Thermal analysis, Obs (Theor.), % | | | Nature of DTA curve (inference) |
|---|---|---|---|---|---|
| | | Coordinated water content | Organic content | Metal oxide content | |
| [Mn(PDBAA)$(H_2O)_3$] $C_{16}H_{21}N_2O_5Mn$ (375.94) | 282 | 13.83 (14.36) | 67.23 (66.77) | MnO 18.94 (18.87) | Exotherm (decomposition) |
| [Fe(PDBAA)$(H_2O)_3$] $C_{16}H_{21}N_2O_5Fe$ (376.85) | 280 | 14.10 (14.33) | 66.73 (66.60) | FeO 19.17 (19.07) | Exotherm (decomposition) |

The results (Table-6) reveal that the ligand is inactive towards all strains. Both the metal complexes are fungitoxic in nature. Fe(II) complex was found to be more active than Mn(II) metal complex for all fungal strains. In general, metal complexes are more active than their parent ligand and hence may serve as vehicles for activation of the ligand as principal cytotoxic[27, 28] species.

Based on the foregoing observations from the molecular weight determinations, molar conductance, analytical, magnetic moment, spectral data and thermal analyses it is suggested that the Mn(II) and Fe(II) complexes exhibit monomeric octahedral configuration. X-ray diffraction studies suggest monoclinic crystal system and space group $P_{2/m}$ for all the metal complexes. Metal complexes are more active than their parent ligand. Tentative structures of the complexes are given in Fig. 1.




M = Mn(II) or Fe(II)[Octahedral].

TABLE-4
CELL DATA AND CRYSTAL LATTICE PARAMETERS FOR $[Mn(PDBAA)\cdot(H_2O)_3]$

a (Å) = 17.2468 ± 0.0268, b (Å) = 22.6517 ± 0.0601, c (Å) = 22.7037 ± 0.0375, Standard deviation: 0.69%, $\alpha = 90°$, $\beta = 84.23°$, $\gamma = 90°$, Volume $(Å)^3 = 2326.28$, $d_{cal} = 1.0731$ g/cm$^3$, $d_{obs} = 1.0737$ g/cm$^3$, Z = 4, Crystal system = Monoclinic, Space group = P2/m, % Porosity = 0.06

| $I/I_o$ | $d_{obs}$ | $d_{cal}$ | h | k | l |
|---|---|---|---|---|---|
| 100 | 13.5963 | 13.5798 | 1 | 0 | 1 |
| 2 | 6.7458 | 6.8442 | 2 | 1 | 3 |
| 90 | 4.5349 | 4.5265 | 3 | 3 | 4 |
| 31 | 4.4503 | 4.4463 | 2 | 4 | 3 |
| 28 | 3.5335 | 3.5358 | 3 | 5 | 4 |
| 45 | 3.4409 | 3.4221 | 4 | 2 | 6 |
| 15 | 3.3919 | 3.3773 | 3 | 3 | 5 |
| 2 | 3.1964 | 3.2021 | 3 | 5 | 3 |
| 2 | 3.0857 | 3.0820 | 1 | 3 | 3 |
| 2 | 2.8268 | 2.8319 | 3 | 1 | 2 |
| 5 | 2.7334 | 2.7330 | 6 | 1 | 7 |
| 7 | 2.6272 | 2.6325 | 3 | 7 | 3 |
| 7 | 2.5159 | 2.5188 | 4 | 0 | 7 |
| 4 | 2.4604 | 2.4539 | 5 | 6 | 7 |
| 6 | 2.3900 | 2.3894 | 4 | 3 | 7 |
| 2 | 2.2653 | 2.2647 | 7 | 2 | 8 |
| 1 | 2.2279 | 2.2244 | 6 | 6 | 7 |
| 10 | 2.1651 | 2.1671 | 5 | 8 | 6 |
| 6 | 2.1292 | 2.1293 | 6 | 7 | 8 |
| 4 | 2.0671 | 2.0697 | 8 | 3 | 10 |
| 7 | 2.0228 | 2.0247 | 6 | 5 | 6 |
| 3 | 1.9838 | 1.9794 | 8 | 1 | 9 |
| 2 | 1.9644 | 1.9591 | 7 | 7 | 9 |
| 3 | 1.9047 | 1.9060 | 2 | 10 | 1 |
| 3 | 1.8737 | 1.8718 | 5 | 8 | 8 |
| 2 | 1.7813 | 1.7804 | 4 | 9 | 7 |
| 3 | 1.7194 | 1.7194 | 1 | 8 | 4 |
| 3 | 1.6918 | 1.6933 | 8 | 7 | 9 |
| 4 | 1.6472 | 1.6463 | 2 | 1 | −1 |
| 2 | 1.5575 | 1.5568 | 3 | 7 | 7 |

TABLE-5
CELL DATA AND CRYSTAL LATTICE PARAMETERS FOR [Fe(PDBAA)$(H_2O)_3$]

a (Å) = 17.2233 ± 0.0358, b (Å) = 22.8264 ± 0.0933, c (Å) = 22.6272 ± 0.0532, Standard deviation: 0.73%, $\alpha = 90°$, $\beta = 82.33°$, $\gamma = 90°$, Volume $(Å)^3 = 2318.01$, $d_{cal} = 1.0795$ g/cm$^3$, $d_{obs} = 1.0816$ g/cm$^3$, Z = 4, Crystal system = Monoclinic, Space group = P2/m, % Porosity = 0.19y

| $I/I_o$ | $d_{obs}$ | $d_{cal}$ | h | k | l |
|---|---|---|---|---|---|
| 96 | 13.5951 | 13.5546 | 1 | 0 | 1 |
| 100 | 13.4310 | 13.5546 | 1 | 0 | 1 |
| 7 | 7.2980 | 7.2993 | 1 | 0 | 2 |
| 42 | 4.5637 | 4.6553 | 0 | 5 | 0 |
| 27 | 4.3816 | 4.3652 | 2 | 4 | 2 |
| 28 | 4.3140 | 4.2996 | 4 | 0 | 5 |
| 3 | 3.8746 | 3.8849 | 3 | 3 | 3 |
| 14 | 3.4814 | 3.4641 | 2 | 1 | 1 |
| 23 | 3.3847 | 3.3780 | 5 | 0 | 6 |
| 3 | 2.7104 | 2.7154 | 5 | 5 | 6 |
| 4 | 2.5088 | 2.5101 | 1 | 6 | 3 |
| 4 | 2.4475 | 2.4487 | 5 | 4 | 5 |
| 4 | 2.1368 | 2.1387 | 7 | 3 | 10 |
| 5 | 1.6433 | 1.6453 | 4 | 6 | 8 |
| 1 | 1.5282 | 1.5293 | 2 | 6 | 6 |
| 8 | 1.4421 | 1.4417 | 4 | 9 | 2 |

TABLE-6
ANTIFUNGAL AND ANTIBACTERIAL STUDIES

| Compound | Conc. (mg/mL) | Zone size in mm | | | | | |
|---|---|---|---|---|---|---|---|
| | | *C. albicans* | *C. krusei* | *C. glabrata* | *A. fumigtus* | *S. aureus* | *E. coli* |
| PDBAA | 0.125 | — | — | — | — | — | — |
| | 0.250 | — | — | — | — | — | — |
| | 0.500 | — | — | — | — | — | — |
| | 1.000 | — | — | — | — | — | — |
| [Mn(PDBAA)$(H_2O)_3$] | 0.125 | 13 | 14 | 10 | — | — | — |
| | 0.250 | 16 | 14 | 10 | 11 | — | — |
| | 0.500 | 16 | 14 | 10 | 11 | — | — |
| | 1.000 | 16 | 15 | 11 | 12 | — | 10 |
| [Fe(PDBAA)$(H_2O)_3$] | 0.125 | 15 | 14 | 12 | 11 | — | — |
| | 0.250 | 15 | 16 | 12 | 11 | — | — |
| | 0.500 | 16 | 16 | 12 | 12 | — | — |
| | 1.000 | 16 | 16 | 14 | 12 | — | — |

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