CHAPTER VII

RESULTS AND DISCUSSION

7. CURCUMIN(Cur)-L-PHENYLALANINE(Phe) SYSTEM

This chapter deals with synthesis, characterization, magnetic, electrochemical, thermal and BSA binding studies of Schiff base derived from curcumin and L-Phenylalanine (Cur-Phe) and its mononuclear Mn(III) and Fe(III) complexes.

7.1 Cur-Phe Schiff base ligand

7.1.1 Elemental Analysis

The Schiff base ligand Cur-Phe was synthesized by the condensation of Curcumin and L-Phenylalanine. It was then complexed with Mn(III) and Fe(III) metal ions. The ligand and its complexes are found to be stable towards air, and are soluble in solvents DMF, DMSO etc. The elemental analytical data of the Schiff base ligand and its complexes are given in the Table 7.1.1. It is seen that the theoretical values agree well with the experimental results.

Table 7.1.1 Elemental analytical data of Cur-Phe Schiff base ligand and its complexes

(Theoretical values in parentheses)

<table>
<thead>
<tr>
<th>Compound</th>
<th>Mol. Formula</th>
<th>% M</th>
<th>% C</th>
<th>% N</th>
<th>% H</th>
</tr>
</thead>
<tbody>
<tr>
<td>Cur-Phe</td>
<td>C_{39}H_{38}N_{2}O_{8}</td>
<td>-</td>
<td>70.14</td>
<td>4.33</td>
<td>5.64</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td>(70.6)</td>
<td>(4.2)</td>
<td>(5.7)</td>
</tr>
<tr>
<td>Mn(III)-Cur-Phe</td>
<td>[Mn(C_{39}H_{40}N_{2}O_{10})]OAc</td>
<td>7.54</td>
<td>61.95</td>
<td>3.24</td>
<td>5.46</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td>(7.3)</td>
<td>(62.3)</td>
<td>(3.7)</td>
</tr>
<tr>
<td>Fe(III)-Cur-Phe</td>
<td>[Fe(C_{39}H_{40}N_{2}O_{10})]Cl</td>
<td>7.28</td>
<td>62.19</td>
<td>3.67</td>
<td>5.42</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td>(7.4)</td>
<td>(62.2)</td>
<td>(3.7)</td>
</tr>
</tbody>
</table>
7.1.2 $^{13}$C-NMR spectral analysis

The $^{13}$C-NMR spectrum of the Schiff base ligand (Cur-Phe) is shown in Fig 7.1.2.

$^{13}$C-NMR Spectrum of Cur-Phe shows a signal at $\delta$ 56.12 which is assigned to two methyl carbons. The signals in the range $\delta$ 110.62-127.85 are due to aromatic carbons. The signal at $\delta$ 146.93 is assigned to two hydroxyl carbons. The signal at $\delta$ 148 is assigned to two C-O carbons. The Signal at $\delta$ 158.94 corresponds to the two azomethine carbon atoms. Signal at $\delta$ 183.42 is due to the two carboxylate carbons.

Fig 7.1.2 $^{13}$C-NMR Spectrum of Cur-Phe
7.1.3 EI mass spectral analysis

The EI mass spectrum of Cur-Phe given in Fig 7.1.3, shows the molecular ion (M⁺) peak at m/z = 662 corresponding to the molecular weight of the ligand. The peaks at m/z = 628, 602, 576, 559, 534, 402, 364, 322, 280, 182, 158, 128 and 111 corresponds to the fragments C₃₉H₃₇N₂O₆, C₃₇H₃₅N₂O₆, C₃₅H₃₃N₂O₆, C₃₄H₃₁N₂O₄, C₂₉H₂₄O₂, C₂₁H₂₀N₂O₄, C₂₀H₂₀NO₃, C₂₀H₁₈N₂, C₁₃H₁₂N, C₁₀H₉NO, C₉H₅N and C₆H₇O₂ respectively. This confirms the structure of Cur-Phe.

Fig 7.1.3 EI Mass Spectrum of Cur-Phe
7.1.4 FT-IR Spectral Studies

The FT-IR spectrum of Cur-Phe Schiff base ligand is depicted in Fig 7.1.4. It shows an intense peak at 1628 cm$^{-1}$ which corresponds to the imine ν(-C=N) vibration. The intense peak at 1561 cm$^{-1}$ is due to (COO$^{-}$) asymmetric stretching and the peak at 1411 cm$^{-1}$ is due to (COO$^{-}$) symmetric stretching vibration of carboxylic acid group. The band at 3424 cm$^{-1}$ corresponds to ν(-OH) stretching vibration. The band at 1027 cm$^{-1}$ represents the ν(C-O) stretching in C-O-CH$_3$ group. This confirms the successful condensation of Curcumin and L-Phenylalanine.
7.1.5 Structure of Schiff base ligand Cur-Phe

From the above analytical and spectral data, it has been confirmed that the Schiff base Cur-Phe, is a tetridentate ligand, and its elucidated structure, drawn using ChemBioOffice 12.0 software is given in Fig 7.1.5. The low energy of the molecule indicates the stability of the ligand.

**In brief**

Colour: Orange
Yield: 63%
Melting point: 178 °C
Mol Formula: C_{39}H_{38}N_{2}O_{8}
% of Elements: C, 70.14; H, 5.64; N, 4.33
$^{13}$C NMR: δ 158.94 (C=N), δ 183.42 (COO$^-$)
EI Mass: m/z = 662
FT-IR: 1628 cm$^{-1}$(C=N), 1561 cm$^{-1}$(COO$^-$)

Total energy: 38.147 kcal/mol

**Fig 7.1.5 Structure of Cur-Phe**