Synthesis, Structural Characterization, Spectral Analysis and Antimicrobial Activities of Schiff Base Ligands and Their Metal Complexes Derived from 3-Aldehydosalicylic Acid

By B. M. Kalshetty, R. S. Gani, S.S.Karabasannavar & M.B.Kalashetti

Abstract - The synthesis of new Schiff base ligands formed by the condensation of 3-Aldehydosalicylic acid with equimolar quantities of 2-Amino-5-methyl pyridine, 4-Amino-5-phenyl-3-mercapto-[1,2,4] Triazole, 4-Amino-3-methyl-5-mercapto-Triazole, 7-Amino-4-ethyl Coumarin forming 2-(5- methyl pyridine-2-yl amino)-methyl-2-hydroxy-Benzoic acid (Scheme 1), 3-[(3-phenyl-5-mercapto-[1,2,4] triazole-4-yl amino)-methyl]-2-hydroxy-Benzoic acid (Scheme 4), 3-[(3- methyl-5-mercapto-[1,2,4] triazole-4-yl amino)-methyl]-2- hydroxy-Benzoic acid (Scheme 7), 3-[(4-Ethyl-Coumarin-7-yl amino)-methyl]-2-hydroxy-Benzoic acid (Scheme 11).

Keywords : 3-aldehydosalicylic acid, schiff bases, metal complexes, characterization and antimicrobial activity.

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The synthesis of new Schiff base ligands formed by the condensation of 3-Aldehydosaliclyc acid with equimolar quantities of 2-Amino-5-methyl pyridine, 4-Amino-5-phenyl-3-mercaptop-[1,2,4]-Triazole, 4-Amino-3-methyl-5-mercaptop-Triazole, 7-Amino-4-ethyl Coumarin forming 2-[5-methyl pyridine-2-yl amino]-2-hydroxy-Benzolic acid (Scheme 1), 3-[3-phenyl-5-mercaptop-[1,2,4] triazole-4-yl amino]-2-hydroxy-Benzolic acid (Scheme 4), 3-[3-methyl-5-mercaptop-[1,2,4] triazole-4-yl amino]-methyl[2-hydroxy-Benzolic acid (Scheme 7), 3-[4-Ethyl-Coumarin-7-yl amino]-methyl[2-hydroxy-Benzolic acid (Scheme 11). The coordination complexes of metal ions like Cu(II), Zn(II), Ni(II), Co(II), Cd(II) with the Schiff base ligands (lH2) having N- and S- donor system have been synthesized at various pH ranges. All the synthesized ligands and metal complexes were characterized by elemental analysis, IR, 1HNMR and Mass spectral data. Hence, the study reveals that the Schiff base ligands involved the donor groups – COOH, -OH, -SH and Nitrogen atom. The ligand LH2 in metal complexes Scheme 2, 5, 8 behaves as a monobasic bidentate ON-donor at pH 4 suggesting the non-involvement of Nitrogen and Sulphur atoms present in the Schiff base ligands. At the same pH 4 the (Scheme 12) also monobasic bidentate ON-donor.

At pH 7 the ligands behave as dibasic tridentate OON-donor (Scheme 4), OOS-donor (Scheme 9), OON-donor (Scheme 13) suggesting the non-involvement of sulphur atom with metal ion in (Scheme 4) and in (Scheme 13) and non-involvement of Nitrogen atom with metal ion in (Scheme 9). In case of (Scheme 9) sulphur makes coordination with metal ion first rather than Nitrogen atom because of its electronic configuration.

At pH 9 the ligand behaves as dibasic tetrate OOSN-donor with metal ions, suggesting the coordination of Nitrogen and Sulphur atom to the metal ions in the complex compounds. Metal complexes of Schiff bases show biological activities including antibacterial, antifungal, anticancer and herbicidal. The Schiff base complexes and derivatives of Coumarin, thiazolidinone and triazoles so far reported as anti-inflammatory, antioxidant, vasorelant, Cytotoxic, anti-HIV, anti-tubercular and anti-microbial and develop effective therapies. The medical application in the treatment of wounds and tumors, where the metal complex compounds of triazole is to impart strength, elasticity and impermeability of water. The metal complexes are still a major line of approach to develop new drugs. Thiazolidine ring present in a large number of biologically active compounds, the development of penicillin which shows the thiazolidine ring. However, analogous heteroaromatic Schiff bases derived from 3-aldehydosaliclyc acid (3ASA) have not been investigated so thoroughly the synthesized Schiff bases and metal complexes have been found to possess biological activity. The present developments of above Schiff bases with metal ions are most fascinating. The continuation of work on the synthesis of Schiff base derived from 3-aldehydosalicyc acid and its complex compounds with metal ions at different pH. All the Schiff bases were synthesized followed by standard procedures.

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on pH using different reaction conditions and are markedly different from these reported by Nag et al.  

\[
\text{COOH} \quad \text{OH} \quad \text{C} \quad \text{H} \quad \text{N} \quad \text{N} \quad \text{CH}_3
\]

2-(5-methyl pyridine-2-yl amino)-methyl-2-hydroxy-Benzoic acid (Scheme 1)

\[
\text{CH}_3 \quad \text{N} \quad \text{H} \quad \text{C} \quad \text{O} \quad \text{O} \quad \text{M} \quad \text{O} \quad \text{O} \quad \text{C} \quad \text{O}
\]

\[
\text{COOH} \quad \text{OH} \quad \text{C} \quad \text{H} \quad \text{N} \quad \text{N} \quad \text{NH} \quad \text{C}_6\text{H}_5
\]

3-[(3-phenyl-5-mercapto-[1,2,4] triazole-4-yl amino)-methyl] -2-hydroxy-Benzoic acid (Scheme 4)

\[
\text{CH}_3 \quad \text{N} \quad \text{H} \quad \text{C} \quad \text{O} \quad \text{O} \quad \text{M} \quad \text{O} \quad \text{O} \quad \text{C} \quad \text{O}
\]

\[
\text{COOH} \quad \text{OH} \quad \text{S} \quad \text{S} \quad \text{AR} \quad \text{M} \quad \text{O} \quad \text{Et} \quad \text{H}
\]

\[
\text{C}_2\text{H}_5 \quad \text{AR} \quad \text{M} \quad \text{O} \quad \text{Et} \quad \text{H}
\]

[ML(EtOH)]_2: M+ Zn, Cd, Cu, Ni, Co. OONS-donor At pH=9 (Scheme 6)
[ML(EtOH)₂]: M+ Zn, Cd, Cu, Ni, Co. OONS-donor at pH=9 (Scheme 6)

[M (LH)₂] M: Zn, Cd. OOS-donor at pH= 9 (Scheme 9).

3-[(3-methyl-5-mercapto-[1,2.4] triazoile-4-yl amino)-methyl] -2-hydroxy Benzoic acid (Scheme 7)

[ML(EtOH)₂] M: Cu, Zn, Ni, Co, Cd. OOONS- donor at pH=9

3-[(4-Ethyl-Coumarin-7-yl amino)-methyl] -2-hydroxy- Benzoic acid (Scheme 11)
II. Results and Discussion

All the Schiff base ligands were dark yellow in colour; they were air and moisture free crystalline solids. They were sparingly soluble in water, in aqueous sodium hydroxide solution. But, easily soluble in alcohol, owing to the possession of the –OH (Phenolic) and –COOH (Carboxylic) groups. The synthesis of coordination complexes with selected metal ions at various reaction conditions reported as follows:

An aqueous or 50% water-ethanol solution of the Schiff base ligands LH₂ Scheme 1,4,7,11 react with metal ions in (2:1) molar ratio forming metal complexes of the type [M(LH)₂] where M= Cu(II),Zn(II) and Cd(II). Hence, the formed metal complexes Scheme 2,5,8,12 indicating monomeric nature of metal complex with OOn- donor at pH-4.

\[ \text{MCl}_2 + 2\text{LH}_2 \rightarrow [\text{M(LH)}_2] + 2\text{HCl}. \]

Whereas M = Cu (II), Zn (II), and Cd (II).

50% water- alcohol solution of Schiff base ligands (Scheme 1,4,7,11) refluxed separately with alcoholic solution of metal ions in an equimolar quantities, forming the metal complexes of the type [M(LH)₂]: M= Zn, Cd, Scheme 3 suggesting the monomeric nature of metal complexes with OOn- donor at pH-7. Scheme 9 also indicating the monomeric nature of metal complex with OOS- donor at pH-7.

\[ \text{EtOH} \]
\[ \text{MCl}_2 (\text{alc}) + 2\text{LH}_2 (\text{alc}) \rightarrow [\text{M(LH)}_2] + 2\text{HCl}. \]

Reflux

In the same pH condition Scheme 13 were formed suggesting the monomeric nature of the metal complex with OOn- donor.

The alcohol solution of metal ions react with an alcoholic solution of LH₂ (Scheme 4) in 1:1 ratio in the presence of sodium- ethoxide and forming the bimetallic complexes of the type [ML (EtOH)]₂ (Scheme 6), (Scheme 10) and (Scheme 14) where M= Cu , Zn, Ni, Co and Cd. Suggesting dimeric nature of metal complexes with OOnS- donor, OOnS- donor and OOnO- donor respectively at pH-9.

\[ \text{EtOH} \]
\[ 2\text{MCl}_2 + 2\text{LH}_2 \rightarrow [\text{ML(EtOH)}]_2 + 4\text{HCl} \]

Reflux

M= Cu (II), Zn (II), Ni (II), Co (II), Cd (II).

All the synthesized metal complexes were sparingly soluble in water but were fairly soluble in absolute alcohol.

The physical characteristics and analytical data of the Schiff base ligands and metal complexes formed at various pH conditions reported in Table 1. The
molecular measurements of the complex compounds indicated the monomeric nature of complex compounds Scheme 2,5,8,12 at pH -4. The metal complex compounds Scheme 3, 9, 13, formed at pH-7 showed monomeric nature and the complexes Scheme 4, 6, 10, 14 at pH-9 showed diimeric nature. The synthesized complex compounds remained stable up to certain temperature and pH as recorded in the Table 2.

Table 1: The Physical and Analytical data of ligands and their Metal Complexes

<table>
<thead>
<tr>
<th>Scheme</th>
<th>Mol.Formula</th>
<th>Mol.Wt. (Obs)</th>
<th>Metal Ions</th>
<th>C (Obs)</th>
<th>H (Obs)</th>
<th>N (Obs)</th>
<th>S (Obs)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1.</td>
<td>C$_5$H$_7$O$_2$N$_2$</td>
<td>256</td>
<td>----</td>
<td>65.63</td>
<td>4.69</td>
<td>10.94</td>
<td>----</td>
</tr>
<tr>
<td>2.</td>
<td>CuC$_5$H$_7$O$_2$N$_2$</td>
<td>573.5</td>
<td>11.07</td>
<td>58.59</td>
<td>3.49</td>
<td>9.77</td>
<td>----</td>
</tr>
<tr>
<td>3.</td>
<td>ZnC$_5$H$_7$O$_2$N$_2$</td>
<td>575.7</td>
<td>11.41</td>
<td>58.36</td>
<td>3.47</td>
<td>9.73</td>
<td>----</td>
</tr>
<tr>
<td>4.</td>
<td>CdC$_5$H$_7$O$_2$N$_2$</td>
<td>622.4</td>
<td>18.06</td>
<td>53.99</td>
<td>3.21</td>
<td>8.99</td>
<td>----</td>
</tr>
<tr>
<td>5.</td>
<td>ZnC$_5$H$_7$O$_2$N$_2$</td>
<td>573.7</td>
<td>11.45</td>
<td>58.57</td>
<td>3.49</td>
<td>9.76</td>
<td>----</td>
</tr>
<tr>
<td>6.</td>
<td>CdC$_5$H$_7$O$_2$N$_2$</td>
<td>620.4</td>
<td>18.12</td>
<td>54.16</td>
<td>3.22</td>
<td>9.03</td>
<td>----</td>
</tr>
<tr>
<td>7.</td>
<td>C$_6$H$_8$O$_2$N$_4$</td>
<td>340.0</td>
<td>----</td>
<td>56.47</td>
<td>3.53</td>
<td>16.47</td>
<td>9.41</td>
</tr>
<tr>
<td>8.</td>
<td>CuC$_6$H$_8$O$_2$N$_4$</td>
<td>739.5</td>
<td>8.59</td>
<td>51.93</td>
<td>2.70</td>
<td>15.15</td>
<td>8.65</td>
</tr>
<tr>
<td>9.</td>
<td>ZnC$_6$H$_8$O$_2$N$_4$</td>
<td>741.7</td>
<td>8.86</td>
<td>51.77</td>
<td>2.69</td>
<td>15.10</td>
<td>8.63</td>
</tr>
</tbody>
</table>

Table 2: The synthesized organometallic complex compounds and the nature of coordination

<table>
<thead>
<tr>
<th>Scheme</th>
<th>pH</th>
<th>Metal-complex</th>
<th>Nature of Complex</th>
<th>Stable up to Temp.in °C</th>
<th>Donor-Atoms</th>
</tr>
</thead>
<tbody>
<tr>
<td>2</td>
<td>4</td>
<td>[ML(H$_2$)]</td>
<td>Monomer</td>
<td>236°C</td>
<td>OO-donor</td>
</tr>
<tr>
<td>3</td>
<td>7</td>
<td>[ML$_3$]</td>
<td>Monomer</td>
<td>228°C</td>
<td>OON-donor</td>
</tr>
<tr>
<td>5</td>
<td>4</td>
<td></td>
<td>Monomer</td>
<td>232°C</td>
<td>OO-donor</td>
</tr>
</tbody>
</table>
a) **Characterization of the Schiff Base Ligands**

The IR Spectra of Schiff base ligands (Scheme 1, 4, 7 and 11) and metal complexes compounds (Scheme 2, 5, 8, 12) at pH-4, pH-7 (Scheme 3, 9 and 13) and the metal complexes at pH-9 (Scheme 6, 10 14) were recorded in KBr pellets. The IR spectra of the 3ASA ligands exhibit intra molecular H-bonded (carboxylic – COOH and Phenolic –OH groups) stretches at 2910 Cm$^{-1}$ and 2710 Cm$^{-1}$, $\nu$(C=O) carboxylic stretches at 1695 Cm$^{-1}$, $\nu$(C-O) phenolic stretches at 1575 Cm$^{-1}$. The IR spectra of Aniline compounds show a characteristic band due to $\nu$(N=O) at 1628 Cm$^{-1}$ for monomer and 1628 Cm$^{-1}$ for dimer, the deprotonation of N-H moiety of Triazole indicated by the characteristic band due to $\nu$(C=N) at 1675 Cm$^{-1}$, supported to know, these signals shift ed to high field in the spectra of the metal complexes, indicating coordination through the ligands nitrogen atom to the metal ion. The formation of a metal Nitrogen bond is further supported by the presence of a band at 578 Cm$^{-1}$ for $\nu$(M -N) indicating the coordination of ligand to the central metal atom through the Nitrogen atom of the ligands. The IR-spectral data of ligands and the metal complexes listed in Table 3.

b) **$^1$HNMR – SPECTRA**

$^1$HNMR spectra of ligands and metal complexes were recorded in recorded in DMSO- d6 using tetra methyl silence (TMS) as internal standard. The $^1$HNMR-spectra of Schiff bases show the –SH proton at 613.2 – 613.7 ppm. Disappearance of this signal to the –SH proton in the metal complexes. In the metal complexes the deprotonation of the thio-group and this supported the coordination of ligand through sulphur atom to the central metal ion.

$^1$HNMR-Spectra of triazole ligands, the signals of NH2 protons appear at 610.3 ppm and 6 9.2 ppm. These signals shifted to high field in the spectra of the metal complexes. In Scheme 6, Scheme 10 in both cases the signals shifted and appear at 69.5 ppm to 69.7 ppm for organ metallic complexes with the ligand molecules. This indicates the bonding through the Nitrogen atom of the Schiff base ligands to the central metal ion. The aromatic protons gave signals at 61.2 ppm – 6 1.3 ppm. The $^1$HNMR spectroscopic data of the ligands and their metal complexes are given in the Table 4.

---

**Table 3 : IR Spectroscopic data (Cm-1) of the ligands and their metal complexes**

<table>
<thead>
<tr>
<th>Scheme</th>
<th>Functional group</th>
<th>IR-Values in Cm-1</th>
<th>Scheme</th>
<th>Functional group</th>
<th>IR-Values in Cm-1</th>
</tr>
</thead>
<tbody>
<tr>
<td>Scheme1</td>
<td>$\nu$(COOH)</td>
<td>3300cm$^{-1}$</td>
<td>Scheme8</td>
<td>$\nu$(COO-)</td>
<td>1748 cm$^{-1}$</td>
</tr>
<tr>
<td></td>
<td>$\nu$(OH)</td>
<td>3150 cm$^{-1}$</td>
<td></td>
<td>$\nu$(C=N)</td>
<td>1628 cm$^{-1}$</td>
</tr>
<tr>
<td></td>
<td>$\nu$(C=N)</td>
<td>1675 cm$^{-1}$</td>
<td></td>
<td>$\nu$(C-H)</td>
<td>3000 cm$^{-1}$</td>
</tr>
<tr>
<td></td>
<td>$\nu$(C-H)</td>
<td>3000 cm$^{-1}$</td>
<td></td>
<td>$\nu$(C=O)</td>
<td>1690 cm$^{-1}$</td>
</tr>
<tr>
<td></td>
<td>$\nu$(C=O)</td>
<td>1690 cm$^{-1}$</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Scheme2</td>
<td>$\nu$(COO-)</td>
<td>1748 cm$^{-1}$</td>
<td>Scheme9</td>
<td>$\nu$(COO-)</td>
<td>1748 cm$^{-1}$</td>
</tr>
<tr>
<td></td>
<td>$\nu$(C=N)</td>
<td>1675 cm$^{-1}$</td>
<td></td>
<td>$\nu$(C=N)</td>
<td>1628 cm$^{-1}$</td>
</tr>
<tr>
<td></td>
<td>$\nu$(C-H)</td>
<td>3000 cm$^{-1}$</td>
<td></td>
<td>$\nu$(C-H)</td>
<td>3000 cm$^{-1}$</td>
</tr>
<tr>
<td></td>
<td>$\nu$(C=O)</td>
<td>1690 cm$^{-1}$</td>
<td></td>
<td>$\nu$(C=O)</td>
<td>1690 cm$^{-1}$</td>
</tr>
<tr>
<td>Scheme3</td>
<td>$\nu$(COO-)</td>
<td>1749 cm$^{-1}$</td>
<td>Scheme10</td>
<td>$\nu$(COO-)</td>
<td>1748 cm$^{-1}$</td>
</tr>
<tr>
<td></td>
<td>$\nu$(C=N)</td>
<td>1620 cm$^{-1}$</td>
<td></td>
<td>$\nu$(C=N)</td>
<td>1628 cm$^{-1}$</td>
</tr>
<tr>
<td></td>
<td>$\nu$(C=O)</td>
<td>1690 cm$^{-1}$</td>
<td></td>
<td>$\nu$(C=O)</td>
<td>1690 cm$^{-1}$</td>
</tr>
<tr>
<td></td>
<td>-M-</td>
<td>578 cm$^{-1}$</td>
<td></td>
<td>-M-</td>
<td>577 cm$^{-1}$</td>
</tr>
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</table>
### Table 4: $^1$H NMR Chemical shifts of the ligand and their metal complexes

<table>
<thead>
<tr>
<th>Compound</th>
<th>Aromatic H</th>
<th>-CH$_3$</th>
<th>-CH$_2$</th>
<th>-CH=N</th>
<th>-SH</th>
</tr>
</thead>
<tbody>
<tr>
<td>Scheme 1</td>
<td>7.1 – 8.0 (m)</td>
<td>$\delta$ 1.2(t)</td>
<td>----</td>
<td>$\delta$ 10.3(s)</td>
<td>----</td>
</tr>
<tr>
<td>Scheme 2</td>
<td>7.1 – 8.0 (m)</td>
<td>$\delta$ 1.2(t)</td>
<td>----</td>
<td>$\delta$ 10.3(s)</td>
<td>----</td>
</tr>
<tr>
<td>Scheme 3</td>
<td>7.1 – 8.0 (m)</td>
<td>----</td>
<td>----</td>
<td>$\delta$ 10.3(s)</td>
<td>----</td>
</tr>
<tr>
<td>Scheme 4</td>
<td>7.1 – 8.0 (m)</td>
<td>----</td>
<td>----</td>
<td>$\delta$ 10.3(s)</td>
<td>$\delta$ 13.7(s)</td>
</tr>
<tr>
<td>Scheme 5</td>
<td>7.1 – 7.9 (m)</td>
<td>----</td>
<td>----</td>
<td>$\delta$ 9.7(s)</td>
<td>$\delta$ 13.7(s)</td>
</tr>
<tr>
<td>Scheme 6</td>
<td>6.9 – 8.0 (m)</td>
<td>$\delta$ 1.3(t)</td>
<td>$\delta$ 2.7(q)</td>
<td>$\delta$ 9.5(s)</td>
<td>$\delta$ 13.6(s)</td>
</tr>
<tr>
<td>Scheme 7</td>
<td>6.4 – 7.2 (m)</td>
<td>$\delta$ 1.3(t)</td>
<td>----</td>
<td>$\delta$ 9.2(s)</td>
<td>$\delta$ 13.7(s)</td>
</tr>
<tr>
<td>Scheme 8</td>
<td>6.7 – 7.8 (m)</td>
<td>$\delta$ 1.2(t)</td>
<td>----</td>
<td>$\delta$ 9.4(s)</td>
<td>$\delta$ 13.6(s)</td>
</tr>
<tr>
<td>Scheme 9</td>
<td>6.9 – 8.0 (m)</td>
<td>$\delta$ 1.3(t)</td>
<td>----</td>
<td>$\delta$ 9.3(s)</td>
<td>$\delta$ 13.6(s)</td>
</tr>
<tr>
<td>Scheme 10</td>
<td>6.9 – 7.2 (m)</td>
<td>$\delta$ 1.2(t)</td>
<td>$\delta$ 2.8(q)</td>
<td>$\delta$ 9.4(s)</td>
<td>$\delta$ 13.2(s)</td>
</tr>
<tr>
<td>Scheme 11</td>
<td>7.1 – 8.0 (m)</td>
<td>$\delta$ 1.2(t)</td>
<td>$\delta$ 2.9(q)</td>
<td>$\delta$ 9.2(s)</td>
<td>----</td>
</tr>
<tr>
<td>Scheme 12</td>
<td>7.1 – 8.0 (m)</td>
<td>$\delta$ 1.2(t)</td>
<td>$\delta$ 2.8(q)</td>
<td>$\delta$ 9.2(s)</td>
<td>----</td>
</tr>
<tr>
<td>Scheme 13</td>
<td>7.1 – 8.0 (m)</td>
<td>$\delta$ 1.3(t)</td>
<td>$\delta$ 2.9(q)</td>
<td>$\delta$ 9.3(s)</td>
<td>----</td>
</tr>
<tr>
<td>Scheme 14</td>
<td>7.1 – 8.0 (m)</td>
<td>$\delta$ 1.3(t)</td>
<td>$\delta$ 2.8(q)</td>
<td>$\delta$ 9.7(s)</td>
<td>----</td>
</tr>
</tbody>
</table>

### III. Experimental

3-Aldehydosalicylic acid24, 7-Amino-4-ethyl Coumarin25, 2-amino-5-methyl Pyridine26, 4-Amino-5-Phenyl-3-Mercapto-1, 2, 4-Trizole27, 4-Amino-5-methyl-5-mercapto-Triazole28, and Schiff base compounds were prepared by adopting the published standard procedures29, 30. The chemicals such as metal chlorides, sodium acetate and other solvents were obtained from Merck.
a) Synthesis of Schiff Base ligands

i. Synthesis of 2-(5-methyl pyridine-2-yl imino)-methyl-2-hydroxy-Benzoic acid

Equimolar ethanol solution (50 ml) of 3-Aldehydosalicylic acid and 2-amino-5-methyl pyridine was refluxed for 2 hours in round bottom flask. During the reflux a crystalline orange compound was separated (Scheme 1) filtered and dried by crystallizing in appropriate solvent. Followed by the same experimental methods the other Schiff base ligands Scheme 4, 7 and 11 were synthesized, the compounds were confirmed by physical and analytical data, IR and 1HNMR spectra were evidences for the Schiff base formation by the selected compounds.

ii. Preparation of Metal Complexes

All the metal complexes of Schiff bases were prepared by the standard reported method The metal salts were dissolved in minimum quantity of water and the solutions were added to hot ethanolic solution of the corresponding Schiff bases. After complete addition, little amount of sodium acetate was added and the mixture was refluxed for 4 hours. Crystalline solid was obtained, which was isolated by filtration, washed with hot water and dried in air.

b) Antibacterial Activity

Antibacterial activity of the all the Schiff base ligands and metal-complexes were evaluated against Staphylococcus aureus, Bacillus subtilis', Escherichia Coli and pseudomonas aeruginosa. Bacterial culture was prepared; each culture was added to the sterilized medium before solidification. The media with bacteria was poured into sterilized Petri dishes under aseptic condition. Different weights of Schiff base ligands and their metal complexes (1 mg, 5 mg and 10 mg) were placed on the surface of the culture and incubated at 300c for one day. After incubation zone of inhibitions (mm) were recorded.

IV. Conclusions

The different types of complex compounds of same ligand with metal ions have been obtained by varying pH and solvent during their synthesis. The basicity and denticity of the Schiff base scheme 1, 4,7,11 derived from 3-Aldehydosalicylic acid and different amines changes considerably with the role of pH from 3 to 9. At pH-3 ligands act as monobasic bidentate OON-donor ligands forms monometallic complex compounds of the type [ML(H)2] where M = Cu,Zn, Cd (Scheme 2,5,8,12). At pH-7 the ligand acts as a monobasic tridentate OON-donor ligand forming the monometallic complex compound of the type [ML2]: M = Zn, Cd (Scheme 3). At the same pH-7 the ligand formed OOS-donor monometallic complex compound of the type [ML(H)2]: M = Zn,Cd (Scheme 9). The Scheme 13 indicates the ligand acts as a dibasic tridentate OON-donor ligand forming the monometallic complex compound of the type [ML(EtOH)]: M = Zn, Cd.

Metal complexes at Ph-9 the ligands act as a dimeric tetra dente ONNS-donor (Scheme 10) and OONO-donor (Scheme 14). The basicity and denticity confirmed by various methods. The IR Spectra of the Schiff bases (Scheme 1,4,7,11) reveals that the non-involvement of carboxylic atom in coordination at pH-4, and metal complexes Scheme 2,5,8,12 reveals that the non-involvement of Nitrogen atom in the coordination. Here in these metal complexes the carboxylic O-atom and O- atom (Phenolic) involved in the coordination by exhibiting the δs(COO) and δ(COO) stretches of carboxylate group in the ranges 1560-1650 Cm⁻¹ (i.e., 1600 Cm⁻¹) and 1340 – 1430 Cm⁻¹ (i.e., 1400 Cm⁻¹) respectively. The energy difference between δs(COO) and δ(COO) is more than 200 Cm⁻¹ indicates the monodentate nature of the carboxylate moiety(31).

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References Références Referencias

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