Microbial Screening of Cobalt (II) and Nickel (II) Complexes of Acetone Thiosemicarbazone, Synthesis and Characterisation

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Abstract: The present investigation involves the preparation and characterization of Nickel (II) and Cobalt (II) nitrate complexes using acetone thiosemicarbazone as ligand. The complexes were characterized by conductivity, spectral and biological studies.

Keywords: Acetone thiosemicarbazone, conductivity, spectral studies, biological studies

1. Introduction

Thiosemicarbazone complexes of transition metals have attracted special attention in the field of biology, due to their effective biological commotion against virus, protozoa and certain kinds of tumor and also antitubercular activity (1-3). The most active research area in the field of inorganic chemistry is Co-ordination chemistry. Co-ordination compounds play an essential role in industry and in Biology and its significance depends on elements, particularly metals in various oxidation states which exhibit the capacity of binding donor molecules. The Co-ordination complexes formed by Cobalt (II) are found to be brown or brownish black crystalline solids (4-5). The co-ordination complexes formed by Nickel (II) are found to be Octahedral or distorted Octahedral having light pink or blue or green crystalline solids (6-9).

2. Experimental Work

2.1 Preparation of Ligands

Preparation of acetone thiosemicarbazone

The following general method was adopted for the preparation of Acetone thiosemicarbazone. Ethanolic solution of thiosemicarbazide and acetone were mixed in the molar ratio of 1:1. The mixture was refluxed for about 6 hours in a water bath. After the completion of reaction, excess of the solvent was removed by filtration and the white coloured product obtained was dried. It was further purified by recrystallisation using the same solvent.

2.2 Preparation of complexes

i) Preparation of Nickel (II) nitrate complexes of the ligands acetone thiosemicarbazone:

Ethanolic solution of nickel (II) nitrate and acetone thiosemicarbazone were mixed in the molar ratio of 1:3. The mixture was heated in a water bath for 2 hours. The excess solvent was evaporated and dried. Washing with acetone and ether for several times purified this. The complex was pink colour solid. Decomposition temperature was found to be above 190°C.

ii) Preparation of Cobalt (II) nitrate complex of the ligands acetone thiosemicarbazone:

Ethanolic solutions of cobalt (II) nitrate and acetone thiosemicarbazone were mixed in the molar ratio 1:3 and the mixture was heated on a water bath for 2 hours. Then excess alcohol was evaporated and the residue was washed with acetone and ether several times. The complex was found to undergo decomposition above 190°C. The complex was soluble in water and sparingly soluble in alcohol.

3. Results and Discussion

The present investigation involves the preparation, characterization and biological investigation of Nickel (II) and Cobalt (II) nitrate complex using acetone thiosemicarbazone as a ligand. The complexes were characterized by conductivity, spectral and biological studies.

The Nickel (II) nitrate complex was prepared using the ligand acetone thiosemicarbazone. The Nickel(II) nitrate complex was found to be pink coloured amorphous solid. It was soluble in water and sparingly soluble in alcohol but not in ether, acetone and chloroform. The Cobalt (II) nitrate complex was prepared using the ligand acetone thiosemicarbazone. The complex thus obtained was found to be brown colored amorphous solid. It was soluble in water and sparingly soluble in alcohol but not in ether, acetone and chloroform.

Physical and Conductivity study

The melting point of the ligand and the yield of Nickel (II) and Cobalt (II) complexes and the conductance data of these two complexes are given in the table 1.

Table 1 Physical Data of the Ligand and Ni(II) and Co(II) Complexes

<table>
<thead>
<tr>
<th>Name</th>
<th>% Yield</th>
<th>Melting Point</th>
<th>Conductance</th>
</tr>
</thead>
<tbody>
<tr>
<td>Ligand</td>
<td>82%</td>
<td>196</td>
<td>56</td>
</tr>
<tr>
<td>Nickel(II) Complexes</td>
<td>86%</td>
<td>128</td>
<td>65</td>
</tr>
<tr>
<td>Cobalt(II) Complexes</td>
<td>85%</td>
<td>136</td>
<td>87</td>
</tr>
</tbody>
</table>
Infrared spectra

The IR spectra of acetone thiosemicarbazone, Nickel (II), Cobalt (II) nitrate complex were taken and analyzed mainly for certain specific frequency which are directly involved in complex formation and the values are given below the table 2. The spectra of the respective ligand and complexes are shown in the figure 1,2,3.

Considerable changes in I-R spectra of the Nickel (II) complex is the appearance of υ(C=N) band in the region of 1604.77as compared to 1652.32 cm⁻¹ in the ligand thus showing a shift towards lower wave number. Coordination of azomethane is confirmed by the presence of a new band at 421-492 cm⁻¹ which can be assigned to υ(M-N) for Nickel(II) and Cobalt(II) complexes.

The IR Spectra of the ligand exhibit a medium intensity band at1268.51 cm⁻¹ due to C=S stretching vibrations. The corresponding band in the spectra of the Nickel (II), Cobalt (II) nitrate complex appear in the region of 1235.9&1230.0 respectively. This lower shift may due to the participation of thiosemicarbonyl group in complexation. This is also supported by the appearance of new band in the region of 420 due to υ(M-S) stretching vibrations. The broad band in the region 3400-2900 cm⁻¹ indicates the presence of water molecules. These regions appear due to OH stretching vibrations merged with NH absorption bands. A sharp peak at 1635.44 is due to H-O-H bending vibrations.

Generally the IR spectrum of the nitrate ion υ2 (out-of-plane deformations), υ3 (double degenerate) υ4 (double degenerate in the plane bending). The IR spectra of coordinated nitrate has been reported in literature survey. υ1 (symmetric) is normally inactive but sometimes becomes weakly active through crystal; interactions. The magnitude of υ4-υ1 decides the nature of the coordinating nitrate group. A difference if nearly 100 cm⁻¹ shows the presence if monodentate nitrate groups and for bidentate nitrate groups this difference is greater than 150 cm⁻¹. Both the Nickel (II), Cobalt (II) nitrate complexes exhibit absorption at 1384 cm⁻¹ characteristic of the ionic nitrate groups.

Table 2: Important IR Absorption Bands (in cm⁻¹) Of Ligands, Ni(II) and Co(II) Nitrate Complexes

<table>
<thead>
<tr>
<th>Name of the Sample</th>
<th>υ N-H</th>
<th>υ C=H</th>
<th>υ C=N</th>
<th>υ C=S</th>
<th>υ CO-H</th>
<th>υ H-OH</th>
<th>υ N-N</th>
<th>υ NO₃ Ionic</th>
</tr>
</thead>
<tbody>
<tr>
<td>LIGAND- ACETONE THIOSEMICARBAZONE</td>
<td>-</td>
<td>2977.9</td>
<td>1268.5</td>
<td>3371.8</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Ni(II) NITRATE COMPLEX</td>
<td>-</td>
<td>2954.8</td>
<td>1604.8</td>
<td>1235.9</td>
<td>3350.4</td>
<td>1635.4</td>
<td>434.1</td>
<td>1384.1</td>
</tr>
<tr>
<td>Co(II) NITRATE COMPLEX</td>
<td>802.8</td>
<td>-</td>
<td>1230.0</td>
<td>-</td>
<td>1629.3</td>
<td>-</td>
<td>1384.2</td>
<td></td>
</tr>
</tbody>
</table>

Biological studies

Thiosemicarbazone possess a wide spectrum of medicinal properties. The ligand and the metal complexes were screened for antibacterial activities against some of the pathogenic bacteria like Pseudomonas putida and Pseudomonas aures by using cup method. The diameter (mm) of the inhibition zone around each cup was measured after 24hours and results are listed in table 3.

The results clearly illustrate that the compounds have antibacterial activity against all the organisms tested. The ligand acetone thiosemicarbazone have no effect on the growth of bacterial compared to metal complexes. The antibacterial activity of nickel (II) complexes against Pseudomonas putida and Pseudomonas aures were much higher compared to other bacterias used. Further it was confirmed all the complexes have higher activity at higher concentration (1000µg) than lower concentration (500 µg).

The work assumes importance as many metal thiosemicarbazone are found to have significant antibacterial activities. The results reveal that all the metal complexes are more toxic than the ligand.

Table 3 Antibacterial Activity of the Ligand and Metal Complexes Evaluated By minimum Inhibitory Concentration

<table>
<thead>
<tr>
<th>Ligand/Complexes</th>
<th>Pseudomonas auras</th>
<th>Pseudomonas putida</th>
</tr>
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<tbody>
<tr>
<td>S1- Co(II) thiosemicarbazone complex</td>
<td>++++</td>
<td>++++</td>
</tr>
<tr>
<td>S2- Ligand</td>
<td>+</td>
<td>+</td>
</tr>
<tr>
<td>S3- Ni(II) thiosemicarbazone complex</td>
<td>++++</td>
<td>++++</td>
</tr>
</tbody>
</table>

Inhibition diameter in MM (% inhibition); +, 6-10 (27-45%) ; ++ 10-14 (45-64%) ; +++ 14-18 (64-82%) ; ++++ >18 (82-100%). Per anti inhibition values are relative to inhibition (22mm) with 100% inhibition.

The ligand and the metal complexes were screened for antifungal activities against some of the pathogenic fungal like Aspergillus Fumigates and Trichodermna Veridi using cup method. The diameter (mm) of the inhibition zone around each cup was measured after 24hours and results are listed in table 4

Table 4: Antifungal Activity of the Ligand and Metal Complexes Evaluated By Minimum Inhibitory Concentration

<table>
<thead>
<tr>
<th>Ligand/Complexes</th>
<th>Aspergillus Fumigatus</th>
<th>Trichodermna Veridi</th>
</tr>
</thead>
<tbody>
<tr>
<td>S1- Co(II) thiosemicarbazone complex</td>
<td>++++</td>
<td>++++</td>
</tr>
<tr>
<td>S2- Ligand</td>
<td>+</td>
<td>+</td>
</tr>
<tr>
<td>S3- Ni(II) thiosemicarbazone complex</td>
<td>++++</td>
<td>++++</td>
</tr>
</tbody>
</table>

Inhibition diameter in MM (% inhibition): +, 6-10 (27-45%) ; ++ 10-14 (45-64%) ; +++ 14-18 (64-82%) ; ++++ >18 (82-100%). Per anti inhibition values are relative to inhibition (22mm) with 100% inhibition.

4. Conclusion

Acetone thiosemicarbazone and their complexes were found to have significant role in antimicrobial activities. The yield obtained was good and the biological studies also resulted well.
References