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Department of Chemistry, Jawaharlal Nehru Arts Commerce and Science College, Wadi, Nagpur, Maharashtra, India Synthesis, characterization and biological activity of schiff base derived from sulphanilamide and its metal complexes with cobalt, nickel and copper

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Abstract

Schiff base have been prepared by the condensation of sulphanilamide with salicylaldehyde. The Schiff base was characterized by elemental analysis and spectral method. The metal complexes of Co (II), Ni (II) & Cu (II) have been prepared by the reaction with Schiff base. The analytical, spectral and magneticstudy data support the octahedral structure for all the complexes. The biological activities of Schiff base and their metal complexes have been studied by screening the compounds, against microorganisms such as *E.coli* and *S. aureus*.

Keywords: Cucumber, boron, yield, quality, Konkan

Introduction

The Schiff bases have remarkable property of forming complexes. The Schiff bases and their metal complexes show a wide, spectrum of applications such as biochemical ^[1], analytical ^[2], industrial ^[3], and antimicrobial ^[4] agents. Metal ions are involved in a large number of chemical reactions by virtue for their ability to coordinate to simple or polymeric donor species ^[5]. The present paper describes the synthesis of Co (II), Ni(II) and Cu(II) complexes using Schiff base derived by the condensation of sulphanilamide with salicylaldehyde. The compounds have been characterized by the elemental analysis, Electronic spectral data, IR, NMR and magnetic susceptibility measurements. The antimicrobial activities of the compounds against some selected organisms have also been reported.

Material and Methods

All the chemicals used were of AR grade. All the solvents were purified by standard methods. The elemental analysis of the carbon, hydrogen and nitrogen were performed at RSIC Chandhigarh. Sulphur estimation was done by standard messenger's method. The metal contents of the complexes were analyzed by EDTA titration IR spectra of Schiff base and metal complexes were recorded on FTIR spectrophotometer at CDRI Lucknow, NMR at RSIC Chandhigarh, electronic spectra of the complexes were recorded in the 270-1200 nm range on GB cintra spectrophotometer. All the compounds are screened for their antibacterial activity against *E.coli* and *S. aureus* using disc diffusion method at Department of Biotechnology, Sant Gadge Baba University Amravati.

Preparation of ligand

Sulphanilamide (4g) was dissolved in 10 ml of DMF (N'N' Diethyl formamide) in round bottom flask and to this solution salicylaldehyde in 1:1 molar proportion was added and acidic condition was made by adding 2-3 drops of concentrated hydrochloric acid. The mixture was refluxed on oil bath by fitting water condenser near about 1-2 hour's at 150-160°C, with controlled temperature. Stop heating and allow the reaction mixture at room temperature then distilled water was added in small installment with constant and vigorous stirring yellow turbid solution part was removed and thick liquid sticked to beaker was left over and was treated with distilled water in small installment with stirring near about 30 min orange coloured Substance was formed the product was filtered off and dried in vaccum dessicecator. The yield was 90% m.p 210°C.

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Preparation of complexes

The SbsC-M^{II}, $[M=Co^{II},Ni^{II} \text{ and } Cu^{II}]$ complexes were synthesized by a following method.

Metal and ligand was taken in the molar ratio 1:2 for reaction. Metal and ligand was dissolved in minimum quantity of distilled water and DMF separately. Then the solution was warmed in water bath. Both the solution were mixed in warm condition with constant stirring then the mixture was digested for few minutes with constant stirring. Add aquious ammonia (50%) drop by drop precipitate was formed at pH 6-7 and the solid complex collected was washed with hot double distilled water to remove excess of metal present in the reaction.

Results and Discussion

The elemental analysis data and some physical properties of the synthesized complexes are reported in Table-1. The elemental analysis indicates that the complexes have the general formula $[M^{II} (SbsC)_2 - 2H_2O]$. All the complexes are insoluble in methanol, ethanol, diethylether but soluble in DMF and DMSO.

IR Spectra

The I. R. spectra of the complexes, in comparison with those of the free ligands, display certain changes which gives and idea about the type of bonds and their structure. The ligand SbsC, exhibit a short & broad band in the region 3200 -3500 cm^{-1} may be assigned to υ (OH) stretching vibration ^[6]. It is further observed that bands in the region. 3200 0 3500⁻¹ are in the same region in coordination complexes this indicates non participation of phenolic group in complexation ^[7, 8]. In the free ligand indicate band around the region 1260-1180 cm⁻¹ and 1390-1330 cm⁻¹ which may be assigned for υ (C-0) stretching and v (0-H) bending in phenol [6, 9]. In case of complexes all these bands observed in the same range indicating that no coordination through phenolic group. The υ (HC=N) frequency of SbsC occurs at 1617 cm -1 this band shift to lower frequency by 5-10 cm⁻¹ in the complexes, indicating coordination through the azomethine nitrogen ^{[10,} ^{11]}. This is again supported by the appearance of a new low intensity bands in the region 470-498cm⁻¹ in complexes attributated to v (M-N) vibration [8]. The persual of the I. R of SbsC ligand indicate sharp band around 1380 cm⁻¹ which may be SO₂-NH₂ streching vibration ^[12]. This bands shifts to lower or higher energy 10-15 cm⁻¹ in all the complexes, indicating that the participation of oxygen in coordination with metal ion which further supported by υ (M-O) ^[13, 14].

¹H NMR

The Schiff base SbsC ¹H NMR exhibits peak in the range 12.7 -12.9 δ ppm and 13.4 - 13.6 δ ppm which may be due to phenolic - OH involved in hydrogen bonding. In complexation these peaks assigned at same position as that of ligand, indicates that – OH group does not involved in complexation ^[9, 15]. SbsC shows aromatic amine 7.9 - 8.1 δ ppm ^[12]. Which is further on complexation shows up field 5.3 - 5.5 δ ppm indicating that the coordination through oxygen with metal ion. Signal appeared in the range 2.62.7 δ ppm may be designated as HC = N – group after complexation it shift to up field 2.3-2.5 δ ppm confirmed that the complexation occurs through Nitrogen of imine group.

Magnetic

The magnetic moments of the complexes are given in table -1 the values of the cobalt (II), Nickel (II) and Copper (II) complexes are 4.73, 3.38 and 2.01 B.M respectively. Suggesting high spins octahedral geometry for all the complexes and show good agreement with the proposed structure of the ligand and its complexes. [Fig-2]. Six coordinate geometry with tow water molecules in axial positions and the two Nitrogen and oxygen molecule from imine and sulphonamide group respectively ^[16, 17, 18].

Electronic Spectra

The electronic spectra of the complexes were recorded in the 400-1200 nm range. The electronic spectra of the cobalt (II) complex exhibit two bands of 1190.4 nm & 590 nm which may be assigned to transition ${}^{4}T_{1g}$ (F) $\rightarrow {}^{4}T_{1g}$ (P) suggested octahedral geometry ⁽¹⁹⁾. The spectra of Nickel (II) complex exhibits single band 421 nm which may be transition ${}^{3}A_{2g}$ (F) $\rightarrow {}^{3}T_{1g}$ (P) (20).. The electronic spectra of the copper (II) complex exhibit a band 480 nm. The transition can be assigned for charge transfer which are characteristic of distorted octahedral geometry ^[21].

Antimicrobial Activity

The antimicrobial activity of the sulphadrug, ligand and corresponding complexes were tested against Escherichia coli (Gram negative) and staphylococuus aureus (Gram positive) bacteria using the disc diffusion method. The results are presented in (table - 2). From the data it is observed that the most of the complexes. Shown higher or equal activity as compared to the ligand and sulphadrug. The results suggest that on complexation antibacterial activity is increase due to the presence of metal & azomethine group ^[22]. From the result it is also observed that the activity of the compound increases as the concentration increases and at higher concentration compound shows good activity.

| Compounds Empirical Formula | Formula | Found (Caled) % | | | | Metal found | Meff. B. | 0/ Viold | |
|---|---------|-----------------|-------------|---------------|---------------|-------------|----------|-----------|---------------------|
| | | С | Н | Ν | S | (Caled) | M 70 110 | 70 I leiu | \mathbf{I} M.F(C) |
| $SbsC C_{13}H_{12}N_2S_1Q_3$ | 276 | 56.20 (56.52) | 3.98 (4.34) | 10.20 (10.14) | 12.02(11.59) | - | - | 90-95% | 215 |
| [Co(II)(SbsC) ₂ 2H ₂ O] | 646.93 | 48.59 (48.22) | 3.28 (3.70) | 8.78 (8.65) | 9.70 (9.89) | 9.72 (9.10) | 4.73 | 70% | |
| [Ni (II) (SbsC) ₂ 2H ₂ O] | 651.54 | 50.67 (50.26) | 3.72 (3.86) | 9.21 (9.02) | 10.40 (10.31) | 9.10 (9.45) | 3.38 | 75% | - |
| [Cu (II) (SbsC) ₂ 2H ₂ O] | 656.69 | 47.68 (47.88) | 3.42 (3.68) | 8.24 (8.59) | 9.68 (9.82) | 9.18 (9.75) | 2.01 | 70% | _ |

 Table 1: Physical Properties & Elemental Analysis Data

| Compounda | E. coli/S. Aureus | | | | | |
|---|-------------------|-------|-------|--------|--|--|
| Compounds | 25 μg | 50 µg | 75µg | 100 µg | | |
| Sulphanilamide | 06/05 | 06/08 | 08/08 | 09/16 | | |
| SbsC | 10/ - | 10/07 | 13/07 | 15/09 | | |
| [Co (II) (SbsC) ₂ 2H ₂ O] | 05/07 | 07/08 | 07/11 | 09/14 | | |
| [Ni (II) (SbsC) ₂ 2H ₂ O] | 10 /8 | 12/09 | 15/09 | 20/12 | | |
| [Cu (II) (SbsC) ₂ 2H ₂ O] | 07/08 | 09/09 | 13/09 | 21/12 | | |

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