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Synthesis, Analysis and Antimicrobial Screening of N-Salicyl-O-Hydroxyphenyleneiminato Co (II) Complex

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ABSTRACT

A Schiff base ligand derived from salicylaldehyde and 2-aminophenol and its complex of Co(II) were synthesized. The ligand and the complex were characterized by elemental analysis, conductivity measurement, FT-IR and melting point or decomposition temperature measurements. The elemental analysis data show the formation of 1:1 metal to ligand ratio. The experimental data show that the ligand act as a tridentate ligand (ONO). The Schiff base and the complex were screened for antibacterial and antifungal activity. The complex shows higher activity than the free ligand.

Introduction

Because of the wide variety of possible structure of ligands, the field of Schiff base complexes is fast developing, especially those involving aldehydes and amines. Many Schiff bases and their complexes have been widely studied because of their industrial and biological application, while some Schiff bases were tested and found with good antibacterial and antifungal activity, which is related to their chemical structure. Imine ligands otherwise known as Schiff base compounds (-RC=N-) after a German scientist Hugo Schiff (Hobday and Smith, 1972) are usually formed by the condensation of primary amine with an active carbonyl compound. Schiff bases and their transition metal complexes have been known, the complexes are formed by the interaction between metal ions and ligands (Schriver *et al.,* 1994). This is done by the donation of one or more lone pair(s) of electrons in the ligand to the metal empty d-orbital of suitable energy. The coordination process results in formation of neutral, cationic and anionic complexes (Hridia *et al.,* 1995). Transition metal complexes play a very important role in medicine and in the manufacture of so many important drugs. The cobalt coordinated complex has a variety of metabolic and biosynthetic function it is essential for proper maturation of blood cells and for treatment of panacea's anemia. It plays a role in certain neurological disorder and it's a growth factor for children, Cobalt (II) Schiff base complexes, are potential antiviral agent, cisdichlorodiamine platinum (II) complex is an anti-cancer agent while copper (II) Schiff base complex is an anti tubular agent (Lippard *et al.,* 1994).

The aim of the present study was to prepare, characterize and determine the antimicrobial activity of Schiff base complex derived from salicylaldehyde and o-aminophenol.

Material and Methods

All the chemicals used in this work were of analytical grade purity, obtained from Sigma Aldrich, United Kingdom. All weighings were carried out using Mettler Toledo Balance AB54, pH measurements were performed using a Jenway pH meter model 3320, conductivity values were measured using conductivity meter (Jenway 4010). Two pathogenic bacteria viz. *Escherichia coli* and staphylococcus aurous and two fungi *Aspergillums niger* and Candida albican, were collected from microbiology unit of the department of biological science Bayero university Kano Nigeria. Nutrient agar were used as bacterial media and potato Dextrose agar as fungi media.

Synthesis of the Schiff base ligand

o-hydroxybenzaldehyde (1.7g) in 20 cm^3 of absolute ethanol was added to 1.5g of o-aminophenol in 30cm^3absolute ethanol. The mixture was concentrated by heating to 25$cm^3$and then cooled in an ice-bath. An orange crystalline product was formed, washed with hot ethanol before recrystallizing from ethanol and drying in vacuum desiccators over (Islam *et al.,* 2001).

Synthesis of the complex

Ethanolic solution of Co(II) chloride (20cm^3) was added to 30$cm^3$of ethanolic solution of the Schiff base. The resulting mixture was boiled on a water-bath for five min. and cooled to room temperature. The complex was separated, washed with hot ethanol and dried in vacuum desiccators over phosphorus pentoxide for two days. (Islam *et al.,* 2001).

Analysis of the complex

Analysis of cobalt metal

0.2g of the complex in 20cm^3 distilled water was dissolved with 5cm^3 nitric acid and heated to dryness and allowed to cool followed by addition of 25cm^3 of distilled water and filtered. The filtrate was diluted to 80cm^3, 0.7g ammonium thiocyanate was added after which the source of heat removed immediately. The solution was stirred for 5-second and allowed to cool to room temperature. Shiny red crystals of the complex separated. The precipitate was filtered, washed, dried and weighed as

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dipyridine cobalt (II) thiocynate. The weight of the precipitate was obtained by drying in an oven at 110^0C to constant weight (Vogel, 1972).

Analysis of water of crystallization

0.2g of complex was placed in an oven at 110^oC until constant weight was obtained. The lost in weight was taken as amount of water of crystallization.

Determination of amount of ligand in the complex

The percentage of the ligand in the complex was determined by subtracting the sum of percentages of metal and water of crystallization from 100%.

Antibacterial and Antifungal activity test

The ligand and the complex were dissolved separately in dimethylsulfoxide to produce three different concentrations (1000µg 2000µg and 3000µg) per disc, they were placed on the surface of the culture and incubated at 37^oC for two days. The invitro antibacterial activity was carried out by disc diffusion method. The diameter of zone of inhibition produced by the ligand and complex were compared with Augumentin (30µg) for bacteria standard and Ketoconozole (600µg) for fungal standard (Ramon *et al*., 2003; Yeamin *et al*., 2003).

Results and Discussion

Tables 1 to 7 reported the key results obtained from the various experiments.

Table 1: Some physical properties of Schiff base and its complex

Component.	Colour	% yield	Melting point(^{o}C)	Decomposition temperature(^{o}C)
Ligand	Orange	81.38	96	-
Complex	Greenish	67.61	-	115

Table 2: Solubility test of the schiff base and its complex

Ligand/Complex	Distilled water	Acetone	Benzene	Toluene	Methanol	Ether
Ligand	IS	S	IS	SS	S	IS
Complex	IS	S	IS	SS	S	IS

KEY: S= Soluble SS=slightly soluble
IS=Insoluble.

Table 3: Infrared spectral data of the ligand and its complex

Ligand/complex	*v*(O-H) (cm^{-1})	*v*(C=N) (cm^{-1})	*v*(M-O) (cm^{-1})	*v*(M-N) (cm^{-1})
Ligand	3510	1600	-	-
Complex	3540	1632	746	630

Table 4: Percentage composition

Co (%)	Ligand (%)	H_2O (%)
15.55	77.93	6.50

Table 5: Stability Constant and Gibb's free Energy

Complex	Stability constant	Gibb's free energy
[Co (L)].$2H_2O$	1.1482×10^7	$-4.0257\ 10^{-1}$ KJmol^{-1}

Table 6: Antibacterial effect of ligand and its complex

Compound	*Escherichia coli*			*Staphylococcus aureus*		
	1000µg/ml	2000µg/ml	3000µg/ml	1000µg/ml	2000µg/ml	3000µg/ml
Schiff base	-	-	+	+	++	++
[Co (L)]. $2H_2O$	+++	+++	+++	+++	+++	+++

Table 7: Antifungal effect of ligand and its complex

Compound	*Aspergillus niger*			*Candida albican*		
	1000µg/ml	2000 µg/ml	3000 µg/ml	1000µg/ml	2000µg/ml	3000µg/ml
Schiff base	+	++	++	-	++	++
[Co (L)]. $2H_2O$	++	++	+++	++	++	+++

Key:
High = +++ (Inhibition zone > 12mm)
Moderately active = ++ (Inhibition zone > 9 – 12mm)
Slightly active = + (Inhibition zone > 6 – 9mm)
In active = - (Inhibition zone < 6mm)

The ligand was obtained as orange crystalline solid with 81.3% yield (Table 1). Solubility test carried out on the ligand and complex in some common solvent show that, they are soluble in acetone, DMSO and methanol, but insoluble in benzene water and ether while slightly soluble in toluene (Table 2). The result of IR spectroscopic analysis (Table 3). Indicates a strong band in the spectra of free ligand occurring at $1600cm^{-1}$ which is assigned to *v* (C=N). It is shifted to lower frequency region $1510cm^{-1}$ in the complex indicating the involvement of nitrogen atom of azomethine group in the coordination. The band at 3510 cm^{-1} in the free ligand is assigned to the phenolic group however it disappears on coordination due to the diprotonation. The band in the metal Schiff based complex in the 3540 cm^{-1} are due to *v* (0-H) stretching vibration indicating the presence of coordinated water (Byeong-Goo *et al*., 1996). The bands within $746cm^{-1}$ and 630 cm^{-1} are assigned to *v* (M-O) and *v* (M-N) stretching vibrations respectively (Silverstein *et al.*, 1967; Koji, 1977). The absence of characteristics signals of carbonyl group indicated that condensation of salicylaldehyde was successful (Martinez *et al*.,). The molar ratio of Co: Ligand: H_2O is 1: 1: 2 obtained from Table 4.

The stability constant of Co(II) complex are found to be relatively high showing good stability because the inner d-orbital is used in accommodating the ligand electron pair and low spin confirming the result obtained from decomposition temperature. The Gibb's free energies of the complexes are found to be low and negative sign is an indication of stability which suggests that the complex is stable, as can be seen in Table 5.

Antibacterial activity of Schiff base and its Co(II) metal complex against *Escherichia coli* and *Staphylococcus aureus*, showed a very high responds especially in the complex in which inhibition zones were more than 9mm wide as indicated in Table 5. Antifungal activity of the Schiff base and complex against *Aspergillus niger* and *Candida albican* showed respond at higher concentration (3000ug/m) as the result indicated in Table 6. The result indicate that the complex show more activity than the ligand under similar experimental condition. This will suggest that the chelation could facilitate the ability of a complex to cross a cell membrane and can be explained by Tweedy's chelation theory (Cotton and Wilkinson, 1972).From the results of the various analyses carried out on the complex compound, the general molecular formula below is suggested

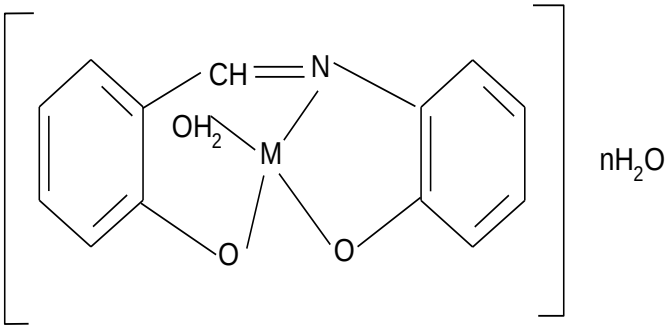

Figure 1:Proposed structure of the complex.

Where M is Co.

Conclusion

The Schiff base was successfully synthesized by condensation of 2-hydroxybenzaldehyde with 2-aminophenol. The Schiff base Co(II) complex was synthesized by the reaction of prepared Schiff base with Co(II) salt. The complex was found to be insoluble in water but soluble in some organic solvents. Similarly, further analysis of the complex by elemental analysis establishes 1:1 metal to ligand ratio. Decomposition temperature was found to be 115^{o}C which indicates that the complex is appreciably stable. The ability of this complex compound to show antibacterial and antifungal properties suggests that it can potentially be used to control bacterial and fungal diseases.

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