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Synthesis and Characterization of Co (ii) Complex with a Schiff Base Ligand Derived from Salicylaldehyde and 4-chloroaniline

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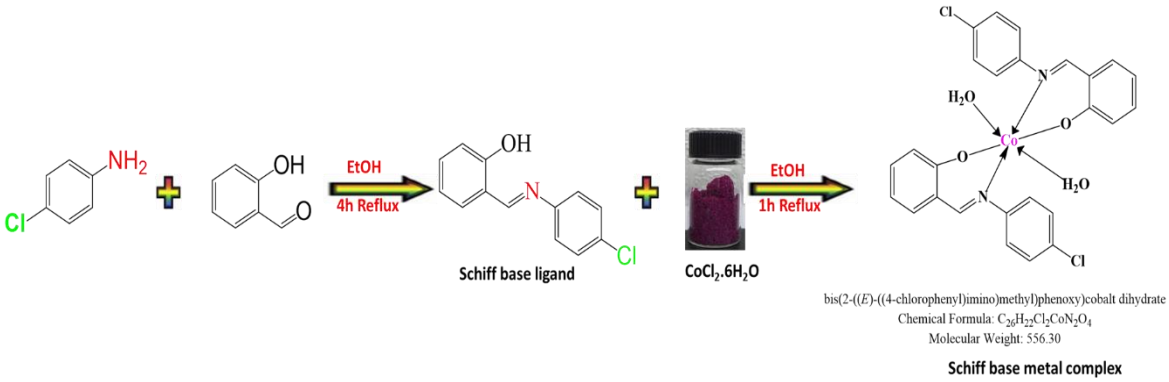
Graphical abstract:



Abstract: **This article presents synthesis of novel Schiff base ligand from N-salicylidene-p-chloroaniline and its Co (II) complex in ratio 1:2 metal-ligands. The complex formed was subjected to elemental analysis and spectroscopic studies (ultraviolet spectroscopy and infra-red spectroscopy) alongside melting point evaluation. From the infrared spectroscopy of the complex, an octahedral arrangement of the ligand around cobalt atom is proposed. The complex formed showed the stability at an ambient condition and it was soluble in most organic solvents.**

Keywords: **Schiff base, Salicylaldehyde, Ligand, Metal complex.**

1. INTRODUCTION

Schiff bases are aldehyde- or ketone like compounds in which the carbonyl group (C=O) is replaced with an imine or azomethine group (-CH=N-) and were first reported by Hugo Schiff in 1864 [1]. Schiff bases derived from an amine and any aldehyde are a class of compound which coordinates to metal ions via the azomethine nitrogen. Schiff bases of the aliphatic aldehydes are stable and are readily polymerizable [2] while those of aromatics aldehydes, having an effective conjugation system, are more stable. Schiff bases have number of applications viz., preparative use, identification, detection and determination of aldehydes or ketones, purification of carbonyl or amino compounds or protection of these groups during complex or sensitive reactions [3].

Metal complexes of Schiff bases derived from substituted salicylaldehyde and various amines have been widely investigated because of their wide applications such as: pigments, dyes, catalysts, intermediates in organic synthesis and as polymer stabilizers [4]. Chelating ligands containing O and N as donor atoms show broad biological activities and are special because

of the variety of ways in which they are bonded to metal ions. Transition metal complexes of Schiff bases have shown considerable attention due to their antifungal, antibacterial, antitumor activities and many other activities because of the azomethine linkages [5]. They have appeared to be important intermediates in a number of enzymatic reactions involving interaction of an enzyme with an amino or carbonyl substrate [6]. Another important role of Schiff bases structure is that it helps to clarify the mechanism of transamination and racemization reaction in biological system [7].

4-chloroaniline is an important building block used in chemical industries for the production of pesticides, drugs and dyestuffs, an important precursor to be widely used in antimicrobial and drug manufacture. Salicylaldehyde is a precursor to a variety of chelating agent and other chemicals. It is well known from the literature that much work have been done on the synthesis and characterization of these compounds with Schiff base ligand formed from salicylaldehyde because of their wide application in many fields such as biochemical, analytical and antimicrobial fields [8].

The rapid development of these ligands resulted in an enhanced research activity in the field of coordination chemistry leading to several interesting conclusions. These conclusions have driven these research efforts towards the synthesis of novel Schiff base ligand. Therefore, the main objective of this study therefore is to prepare a new Schiff base ligand from Salicylaldehyde and 4-chloroaniline and its Co (II) complex.

2. MATERIALS AND METHODS

Sample collection

The materials used include Cobalt (II) chloride ($CoCl_2.6H_2O$, 273.93 g/mol), Salicylaldehyde ($C_7H_6O_2$, 122.12 g/mol) and 4-cholroaniline ($C_6H_4NH_2Cl$, 127.5 g/mol) was collected from Department of Chemistry, University of Ilorin, Ilorin, Nigeria.

Synthesis of the Ligand

Salicylaldehyde (2.12 ml, 20 mmol) and 4-chloroaniline (2.55 g, 20 mmol) were separately dissolved in 10ml of ethanol. The mixture was heated under reflux with constant stirring for 4 hours. The solution was allowed to cool to room temperature and a yellow-colored precipitate was obtained. The precipitate was filtered and washed with ethanol and then dried to obtain a golden yellow powder which is then stored in the desiccator.

Synthesis of the Schiff base metal complex

The ligand (0.46 g, 2 mmol) was dissolved in 10ml of ethanol. The solution of metal salt $CoCl_2.6H_2O$ (0.237 g, 1 mmol) in 10 ml of ethanol was added. The mixture was heated under reflux with constant stirring for 1 hour. A yellow-colored precipitate was obtained. The precipitate was collected by filtration washed with ethanol and dried and stored in a desiccator.

Characterization of the ligand and the complex

Melting point test

The melting point determination and decomposition of the ligand and complex were obtained with Gallen Kamp apparatus using capillary tubes and was carried out in the department of chemistry

Solubility test

The solubility of the ligand and the complex were tested in hot and cold various solvent such as distilled water, methanol, acetone and chloroform. It was observed that virtually the same solvent dissolve both the ligand and the complex

Elemental analysis

Elemental analysis of both the ligand and complex were carried out at MEDAC LTD, U.K

UV spectroscopy

The UV spectroscopy were recorded on a bulk scientific M530 IR spec. machine at the center of research institute, Ladoke Akintola University of Technology, Ogbomoso, Oyo state, Nigeria

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Infrared spectroscopy

The infrared spectroscopy was recorded on a bulk scientific M530 IR spec. machine at the centre of research institute, Ladoke Akintola University of Technology, Ogbomoso, Oyo state, Nigeria

3. RESULTS AND DISCUSSION

As shown in **Table 1,** it was observed that the Schiff base melted at exactly 100 ^{o}C while the metal complex was found to have decomposed between 116 – 118 ^{o}C.

The solubility result of the Schiff base and the complex shown in **Table 2** depicted that the Schiff base is soluble in distilled water, ethanol and chloroform but insoluble in other tested solvents. However, the metal complex was found to be soluble in DMSO, DMF, $CHCl_3$, and acetone but slightly soluble in methanol and ethanol and completely soluble in the mixture of ethanol and methanol.

Table 1: Physical data for the ligand and the complex

Compound	Colour	Melting point (^{o}C)
$C_{13}H_{10}NOCl$ Schiff base	Yellow	100
$C_{26}H_{22}Cl_2CoN_2O_4$ Complex	Pale yellow	116 – 118

Table 2: Result of solubility test

Reagents	Schiff base	Complex
Distilled water	NS	NS
Methanol	S	S
Ethanol	S	NS
Diethly ether	S	S
DMF	S	S
Dichloromethane	S	S
Acetone	S	S

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Elemental analysis of the ligand and the complex

The elemental analysis data for the Schiff base and the complex is presented **Table 3** below. It is observed that the percentage composition of C, H and N are in agreement with the ligand. This indicates that the compound is pure. However, the difference in the percentage of C, H and N values was observed in the complex, this may be attributed to the presence of reactant impurities in the complex.

Table 3: CHN data for the Schiff base and the complex

ELEMENT	C	H	N
Ligand %calculated	67.33	4.31	6.04
Ligand %found	67.43	4.22	6.39
Complex %calculated	56.08	3.95	5.03
Complex %found	66.78	4.45	6.00

The variation observed between the calculated and experimentally determined CHN values of Co (II) complex may be attributed to the presence of unreacted materials in the product. Hence, the compound needs to be further purified to obtain the clean product.

UV-visible spectroscopic analysis of the ligand and the complex

The electron spectra of the ligand and its metal complex are represented in the **Table 4** below:

Table 4: UV data for the Schiff base and the complex

Compound	Wavelength	Transition assignment
Schiff base	400	n-n*
Metal complex	420	π -π*

Result and Interpretation of IR spectroscopic data

The IR spectra of the Schiff base and the complex are presented below in the table

Table 5: IR data for salicylaldehyde, 4-chloroaniline, Schiff base ligand and the complex

Compound	**N-H (cm^{-1})**	**O-H (cm^{-1})**	**C=O (cm^{-1})**	**C=N (cm^{-1})**	**C=C (cm^{-1})**	**M-N (cm^{-1})**	**M-O (cm^{-1})**	**C-N (cm^{-1})**
Salicyaldehyde	-	3100	1680	-	1490	=	-	-
4-chloroaniline	3473	-	-	-	1618	-	-	1289
Schiff base	-	3200	-	1608	1492	-	-	1271
Complex	-	3059	-	1610	1487	514	422	1273

The IR spectrum of salicylaldehyde shows the band around 3100 cm^{-1} which is attributed to O-H stretch. The peak around 1680 cm^{-1} attributed to the C-O, the peak around 1490 cm^{-1} is assigned o he aromatic C=C.

The IR spectrum of 4-chloroaniline shows the peak at 3473 cm^{-1} assigned to the N-H band, the peak at 1289 cm^{-1} is attributed to the C-N band. The peak at 1590 cm^{-1} is assigned to C=C band.

Interpretation of the IR spectra of the Schiff base

The IR spectrum of the ligand showed the band around 2100 cm^{-1} which is attributed to O-H stretching vibration of the Schiff base. The band at 1271.09 cm^{-1} is assigned to C-N of the Schiff base while the peak around 1608 suggest the azomethine attributed to the C=N stretching frequency of the Schiff base ligand. The peaks at 1492 $cm^{-1}$are assigned to the aromatic C=C. The disappearance of the C=O peak in the salicylaldehyde spectra and the appearance of a new band C=N that shows the coordination of the two ligands which confirms the formation of the Schiff base.

In the spectra of the complex, the band at 3200 cm^{-1} is attributed to the O-H stretching which is almost the frequency in the spectra of the ligand and suggesting the in coordination of the group. The C-N at 1271.09 cm^{-1} in the Schiff base was assigned to 1273.02 cm^{-1} in the complex indicating the coordination of the carbonyl nitrogen of the Schiff base. The bad at 1610.56 cm^{-1} suggests the involvement of C=N of the Schiff base in the coordination to the central metal ion. The appearance of two new band s at 422.41 cm^{-1} and 514.99 cm^{-1} the spectra of the M-O ad M-N stretching vibrations respectively, supports the formation of the eta complex.

Based on the results from the elemental analysis, IR, and the melting point determination, the following structures are proposed for the ligand and the complex as shown in **Figure 1** and **Figure 2** respectively.

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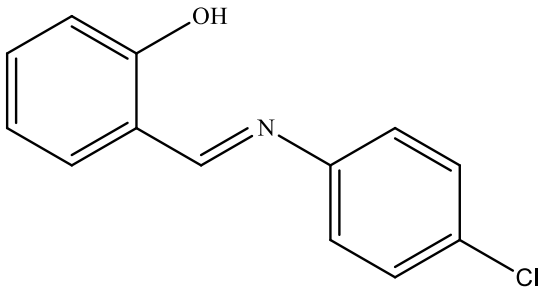
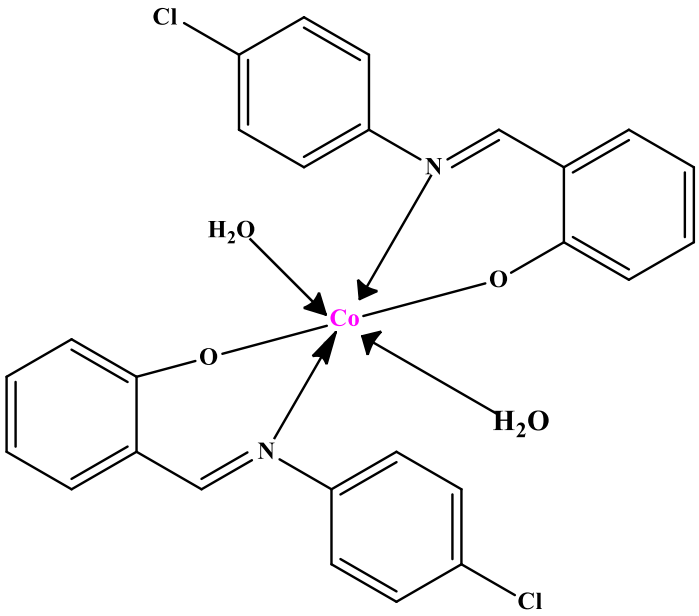


(*E*)-2-(((4-chlorophenyl)imino)methyl)phenol
Chemical Formula: $C_{13}H_{10}ClNO$
Molecular Weight: 231.68

Figure 1: Proposed ligand structure



bis(2-((*E*)-((4-chlorophenyl)imino)methyl)phenoxy)cobalt dihydrate
Chemical Formula: $C_{26}H_{22}Cl_2CoN_2O_4$
Molecular Weight: 556.30

Figure 2: Proposed complex structure

4. CONCLUSION

A Schiff base ligand and Co (II) complex was synthesized from salicylaldehyde and 4-chloroanline and were characterized by elemental analysis, ultraviolet spectroscopy and infrared spectroscopic techniques and an octahedral geometry was suggested f or the complex.

REFERENCES

[1] A. Maher and S. R. Mohammed, "Metal complexes of Schiff base derived from salicylaldehyde-Areview," Int. J. Curr. Res. Rev., vol. 7, no. 2, p. 6, 2015.

[2] S. T. Ha, L. K. Ong, Y. Sivasothy, G. Y. Yeap, P. L. Boey, and H.-C. Lin, "New mesogenic Schiff base esters with polar chloro substituent: synthesis, thermotropic properties and X-ray diffraction studies," Am. J. Appl. Sci., vol. 7, no. 2, pp. 214–220, 2010.

[3] H. Chaudhry, "Synthesis, Characterization and Biological Aspects of O-and N-Donor Schiff Base Ligands and their Co (ll), Cu (ll),...," Synthesis (Stuttg)., vol. 7, no. 2, pp. 29–33, 2020.

[4] D. T. Sakhare, "Synthesis, Characterization of Schiff Bases and Biological Activities of Their Transition Metal Complexes-Review," Int. J. Adv. Sci. Eng., vol. 6, no. 04, pp. 1538–1544, 2020.

[5] I. P. Ejidike and P. A. Ajibade, "Transition metal complexes of symmetrical and asymmetrical Schiff bases as antibacterial, antifungal, antioxidant, and anticancer agents: progress and prospects," Rev. Inorg. Chem., vol. 35, no. 4, pp. 191–224, 2015.

[6] A. Xavier and N. Srividhya, "Synthesis and study of Schiff base ligands," IOSR J. Appl. Chem., vol. 7, no. 11, pp. 6–15, 2014.

[7] E. HASANOĞLU ÖZKAN, N. KURNAZ YETİM, D. NARTOP, and N. SARI, "The importance and applications of Schiff bases."

[8] A. A. Raj, J. Vinnarasi, M. Johnson, and G. Valli, "Synthesis characterization and biological activities of Schiff bases of Isatin," vol. 1, no. 2, pp. 217–222, 2019.