

SYNTHESIS, CHARACTERIZATION AND ANTIMICROBIAL STUDIES OF Cu (II), and Ni (II) COMPLEXES DERIVED FROM 2-HYDROXYBENZALDEHYDE AND 2-AMINOPYRIDINE

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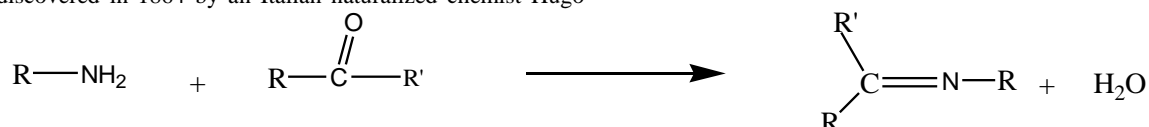
ABSTRACT

Schiff base ligand derived from condensation of 2-hydroxybenzaldehyde and 2-aminopyridine have been prepared and used to synthesized Cu (II) and Ni (II) complexes, characterized by spectroscopic method (IR spectra). Conductivity, solubility test were determined. On the basis of the infrared studies, appearing of new band around 721 -762 cm^{-1} and 553- 443 indicate formation of $\nu(\text{M}-\text{N})$ and $\nu(\text{M}-\text{O})$ confirming a coordination of ligand to the metal ion through oxygen and nitrogen atoms. The antimicrobial activities of the complexes was studied against the *Staphylococcus aureus*, *Escherichia coli*, *Candida albican*. The copper complex showed higher activity than the Nickel complex and the ligand.

Keywords: Antimicrobial activities, ligand, metal complexes, Schiff base

INTRODUCTION

Schiff bases are compounds which contains carbon-nitrogen double bond generally known as azomithine or imine linkage ($-\text{CH}=\text{N}$) formed by the condensation of a primary amine with carbonyl compound according to the following reaction (Scheme1) (Otuokere et.al. 2010). Schiff base was first discovered in 1864 by an Italian naturalized chemist Hugo



Primary amine Aldehyde $\text{R}' = \text{H}$ or Ketone $\text{R}' = \text{R}$

Schiff base

Scheme 1: General synthetic method of Schiff base

Schiff bases plays an important role in coordination with metal ions for the formations of Schiff base metal complexes. Currently Schiff base metal complexes attracting the attention of researchers due to their notable antibacterial, antitumor and antifungal activities. Aliphatic aldehydes are unstable and readily polymerize whereas Aromatic aldehydes form stable Schiff bases (Paul, 2002). In this research, we aimed to synthesize, characterize the series of Cu (II) and Ni (II) metal complexes of Schiff base derived from 2-Hydroxybenzaldehyde and 2-aminopyridine and investigates their biological activities for the drugs development.

MATERIAL AND METHOD

All the reagent used were purchased from zayo sigma (ZAS) chemical limited. The glass wares used was washed thoroughly with detergent, rinse with distilled water and allowed to dried in an oven. Melting point and decomposition temperature was determined using Gallenkamp 4947 melting

Joseph Schiff, since then many researches had followed due same procedure to the stability of the compound. The Schiff base was structure-based compound in which the oxygen atom of the carbonyl group ($\text{C}=\text{O}$) of an aldehyde or ketone replaced by the nitrogen of primary amine as shown in Scheme1 (Cinerman Z. et al.2000).

point apparatus, conductivity measurement were carried out using Jenway 4510 conductivity meter. The FTIR were carried out on a perkins Elmer Uv visible spectrophotometer in this range $4000\text{-}650 \text{ CM}^{-1}$ wavelength. The biological activity was carried out on two bacteria (*Staphylococcus aureus* and *Escherichia coli*) and one fungal (*Candida albican*) at Kano University of Science and Technology Wudil Department of microbiology.

Synthesis of Schiff Base Ligand

The method as reported by lekha et.al; (2013) was used, 0.94g (10mmol) of 2-aminopyridine mixed with 40ml ethanol and 0.88g (10mmol) of 2-hydroxybenzaldehyde, the solution refluxed for 4 hours, the product obtained after cooling in ice block for 20 minutes was left for 7days, the bright yellow precipitate obtained filtered off and washed with Diethylether and dried in desiccators over anhydrous calcium chloride salt.

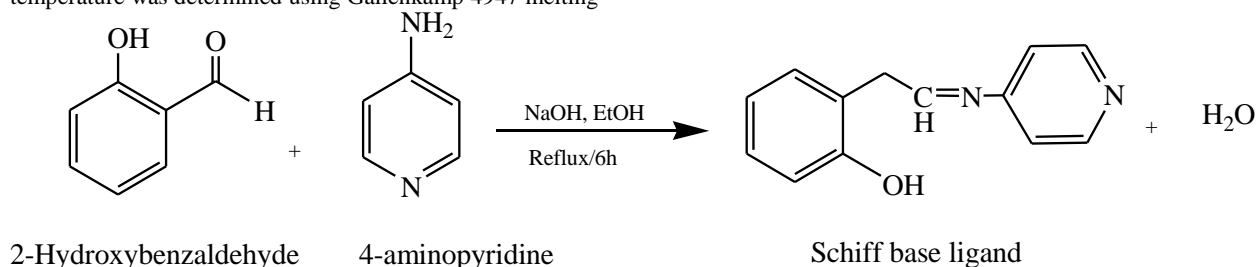
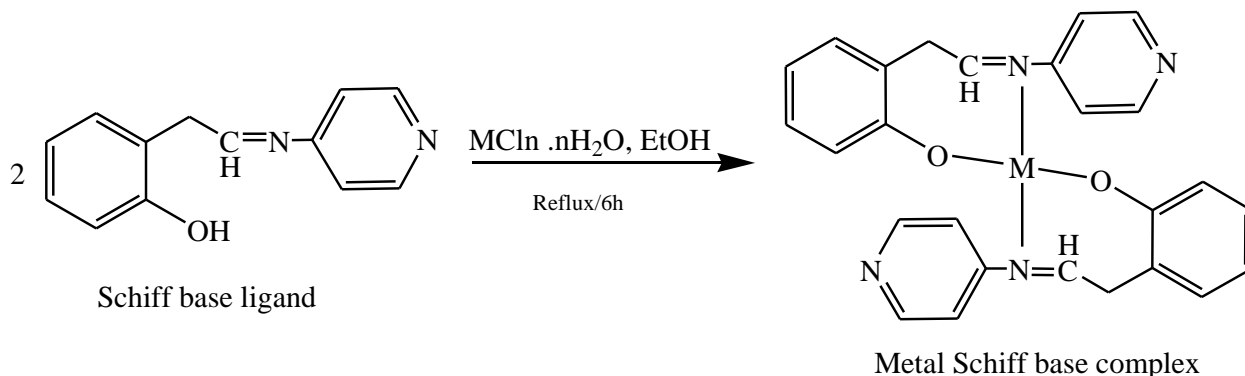


Figure 1: Preparation of Schiff base ligand

Preparation of Schiff Base Metal (II) Complexes

1g (5XZ.00 mmol) of Schiff base derived was dissolved in 10ml ethanol, 1.5g (9.00mmol) of metal (II) chloride was mixup with constant stirring of 20-30 minutes. The resulting

mixture was refluxed for 6 hours and allowed to cooled in ice block, after refluxing the mixture was left for 4 days, the product was filtered off, and wash with Diethylether and dried in desiccators over anhydrous calcium chloride salt.



M = Cr(II), Mn(II), Fe(II), Co(II), Ni(II) or Cu(II).

Figure 2: Preparation of Schiff base metal (II) complexes

RESULT AND DISCUSSION

Table 1: Presentation of Physical analysis

| Compounds | Colour | Melting point and Decomposition temperature (°C) | Water of crystallization (%) | Percentage yield (%) | Conductance (s/cm) | Molecular mass (amu) | Molecular formula |
|-------------------|--------------|--|------------------------------|----------------------|--------------------|----------------------|---|
| Ligand | Light yellow | 125 | | 96 | | 211.00 | [C ₁₃ H ₁₁ ON ₂] |
| Cu (II) complexes | Black | 155 | 10 | 85 | 0.92 | 553.49 | [M(C ₂₆ H ₁₉ O ₂ N ₄)] |
| Ni (II) complexes | Brown | 175 | 12 | 68 | 1.98 | 548.60 | [M(C ₂₆ H ₁₉ O ₂ N ₄)] |

Table2: Solubility test analysis

| Compounds | Distilled Water | Methanol | Ethanol | Diethylether | Chloroform | DMSO |
|-------------------|-----------------|----------|---------|--------------|------------|------|
| Ligand | IS | S | S | IS | S | S |
| Cu (II) complexes | IS | IS | IS | IS | IS | S |
| Ni (II) complexes | IS | SS | SS | IS | S | S |

Table 3: Result of FTIR analytical data

| Compounds | $\nu(\text{O-H})$ | $\nu(\text{C=N})$ | $\nu(\text{M-N})$ | $\nu(\text{M-O})$ cm ⁻¹ |
|-------------------|-------------------|-------------------|-------------------|------------------------------------|
| Ligand | 3283 | 1662 | - | - |
| Cu (II) complexes | - | 1614 | 721 | 453 |
| Ni (II) complexes | - | 1611 | 762 | 443 |

Table 4: showing zone of inhibitory concentration (ZIC) on different microorganisms

| Compounds | Inhibition zone (mm) | | | | | | | | |
|-----------------------|------------------------------|----|-----|-------------------------|----|-----|------------------------|----|----|
| | <i>Staphylococcus aureus</i> | | | <i>Escherichia coli</i> | | | <i>Candida albican</i> | | |
| Organisms | | | | | | | | | |
| Concentration (mg/mL) | 100 | 50 | 25 | 100 | 50 | 25 | 100 | 50 | 25 |
| Ligand | 27 | 25 | NZI | 20 | 40 | NZI | 37 | 38 | 36 |
| Cu (II) complexes | 49 | 48 | 48 | 26 | 29 | 24 | 19 | 18 | 16 |
| Ni (II) complexes | 17 | 37 | 24 | 25 | 19 | NZI | 9 | 9 | 8 |

Physical analysis

Physical properties was presented in table1, the colour of the crystalline ligand is light yellow, copper is black and nickel is brown, indicating their polymeric nature. The melting point

as presented indicated that the metal (II) complexes showed higher melting point compare to that of the ligand showing their stability and purity.

Conductivity measurement of complexes

The conductivity measurement values shows are non-electrolyte and is a significance that show a good coordination of the metal complexes and ligand (Cezar, et al; 2000)

Solubility test

Solubility test of the synthesized metal complexes indicates that both Cu (II) and Ni (II) complex are insoluble in most of the organic solvent except in DMSO and chloroform which are soluble and ethanol and methanol which are slightly soluble. The ligand is soluble in all organic solvent except in distilled water and diethylether.

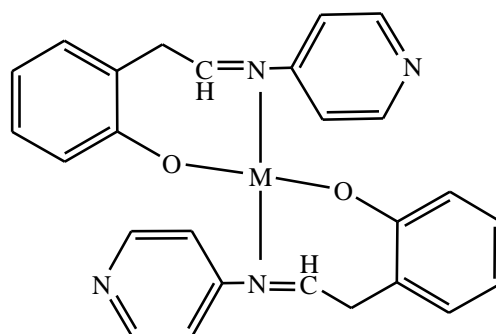
FTIR Analysis

The band around 3283 was observed due the presence of OH group in 2-hydroxybenzaldehyde which was disappear in

metal complexes due coordination of metal ions with phenolic oxygen. Presence of $\nu(\text{C}=\text{N})$ stretching was due to azomethine around $1662 - 1611 \text{ cm}^{-1}$, appearing of new band around $721 - 762 \text{ cm}^{-1}$ and $553 - 443$ indicate formation of $\nu(\text{M}-\text{N})$ and $\nu(\text{M}-\text{O})$ confirming a coordination of ligand to the metal ion through oxygen and nitrogen atoms.

ANTIBACTERIAL AND ANTIFUNGAL ACTIVITIES

Table 4 show s the result of antibacterial and anti-fungal activity taking the *Staphylococcus aureus*, *Escherichia coli*, *Candida albican* as test microorganisms showing their zone of inhibition after measuring the diameter (mm) of each compounds. The metal (II) complexes was found to inhibit more activity then the ligand, copper complexes is more active than the nickel complexes in all the microorganisms .



M = Ni(II) or Cu(II)

Figure 3: Suggested proposed Structure of the Complexes of Ni (II) and Cu (II)

CONCLUSION

In conclusion, metal complexes of Cu (II) and Ni (II) was synthesized successfully from condensation of 2-hydroxybenzaldehyde and 2-aminopyridine. The metals was found to be monometallic. Molar conductivity result of the complexes in chloroform showed that the complexes are nonelectrolytes due to low values obtained. Their activity against three microorganism *Staphylococcus aureus*, *Escherichia coli* and *Candida albican* reveals that the compounds are active against these organism.

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