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Synthesis, Physicochemical and Antimicrobial Studies of Schiffbase Ligand derived from Benzaldehyde and Urea and its Ni(II) and Cu(II) Metal Complexes

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ABSTRACT

Metal complexes of Ni (II) and Cu (II) with Schiff-base were successfully synthesized, characterized by solubility, melting point/decomposition temperature, molar conductivity, FT-IR, magnetic susceptibility, and elemental analysis. The complexes have a 1:2 metal-ligand ratio and low molar conductance values, indicating nonelectrolytes. They were tested for antibacterial activity against pseudomonas and *Escherichia coli* and *Candida albicans* and *Aspergillus flavus*. Both the free ligand and the complexes showed antibacterial activity, with higher activity at high concentrations. The complexes showed better antibacterial and fungal activity than the free ligand.

Keywords: Urea, Synthesis, Benzaldedyde, Microorganisms, Azomethine.

INTRODUCTION

Schiff bases metal complexes have occupied a central place of importance in the development of coordination Chemistry. The literature on these complexes range from the purely synthetic to modern physico-chemical as well as biochemically relevant studies. Many Schiff base complexes are found to have antibacterial, antifungal, anti-inflammatory and antileukaemic. [1]. Since then, a large number of Schiff base complexes have been prepared and even now they comprise the major portion of the current literature on coordination chemistry. The chemistry of Schiff bases and their complexes has been repeatedly reviewed by several workers [2].

Schiff Base

Hugo Schiff, a German chemist, isolated the first condensed product of amines with carbonyl compounds, which he referred to as Schiff base. Thus the Schiff bases are the organic compounds containing azomethine (–R-C=N-) group and are usually formed by the condensation of a primary amine with an active carbonyl compound. A Schiff base (azomethine), is a functional group that contains a carbon-nitrogen double bond with the nitrogen atom connected to an aryl or alkyl group but not hydrogen. Schiff bases have the general formula of $R_1R_2C=NR_3$, where R_3 is an aryl or alkyl group that makes the Schiff base a stable imine [3]. Generally Schiff bases are prepared under acid or base catalysis or with heat [4]. Schiff bases can be synthesized from a reaction of an aromatic amine and a carbonyl compound by a nucleophilic addition forming a hemiaminal, followed by a dehydration to generate an imine. The Schiff base formation is really a sequence of two types of reactions, i.e. addition followed by elimination. A large number of aldehyde and ketone have been condensed with various amines to give Schiff base Ligand, a metal surrounded by a cluster of ions or molecule is used for preparation of complex compounds named as Schiff base [5].

Materials and Methods

All the reagents used were of analar grade and therefore no further purification was required. The electric meter balance model AB54 was used for weighing all the reagents. The infrared spectral analysis data were recorded using Fourier transform IR (FT-IR) model in Nujol, within 400-4000cm^{-1}. The UV/Visible spectra were obtained from Perkin Elmer Lambda 25 Spectrophotometer. The molar conductance measurement was carried out on Jenway 4010 conductivity meter. The sterilization of plates and bottles was done using autoclave.

Preparation of the ligand

10 mmols (0.6g) of urea and 20mmols (2.06cm^3) of Benzaldehyde were dissolved in round bottom flask containing 50cm^3 of methanol and refluxed for 3hrs with vigorous stirring .The reaction mixture was cooled and white

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crystalline solid obtained was filtered, washed with small amount of methanol and dried in desiccator for three days [6,7].

Preparation of the complexes

A solution of Schiff base was prepared in a round bottom flask by dissolving 10mmols (2.66g) of the Schiff base in 30cm^3 of methanol. A hot methanolic solution of 5mmols of the respective metal (II) chloride was mixed with the Schiff base solution and refluxed for 4hrs with continuous magnetic stirring. On cooling, a coloured complex was separated, filtered, washed with methanol and diethyl ether and then dried in desiccator for three days [6,8].

Molar Conductivity Measurement of the Complex

Electrolytic properties of the complexes are due to the ability of the complexes to form ions in solution. Complexes that produce ions in solution are said to be electrolytic and those that don't are termed non- electrolyte. Molar conductance measurement of the complexes were carried out in 10^{-3}M dimethyl sulfoxide solution at room temperature using procedure reported by [9].

Solubility test for the Schiff base ligand and the complexes

0.1g of each of the metal complexes and Schiff base were taken and transferred into test-tube containing 10cm^3 of the corresponding solvents and their solubility were determined. The solvent used were, distilled water, ethanol, methanol, dimethyl sulfoxide, diethyl ether, carbon tetrachloride and toluene [10].

Magnetic Susceptibility Measurement

Each prepared metal complex was introduced into a capillary tube up to a given mark and the reading recorded using the magnetic susceptibility balance. All measurements were conducted at room temperature [11].
In order to calculate for the magnetic properties of the compounds, below is the equation to be used

$$Xg = \frac{CxL(R-R0)}{109M}$$

Determination of Melting point/Decomposition Temperature

Melting point of the ligand and decomposition temperature of the respective metal complexes were determined by introducing each complex compounds into a capillary tube and then inserted into Gallenkamp melting point apparatus, the temperature at which the ligand melt and the complexes decomposed were recorded [12].

Determination of Metal ions in the Complexes

0.01g of the powdered sample was added into 10cm^3 digestion flask containing 1cm^3 concentrated H_2SO_4 and about 0.7cm^3 of 60-62% of nitric acid. The flask was then heated in a fume cupboard until dense white fume appeared. The solution was then allowed to cool and then filter into 10cm^3 volumetric flasks. Deionize water was then added up to the mark. The blank was also prepared. The digested sample and the blank were then analyse using AAS.

Antibacterial Activity

The ligand and their metal complexes were screened for antimicrobial activity against two bacteria; Escherichia coli and *klebsella pneumoniae* and fungal isolates; *Aspergillus fumigatus and candida albicans by* the agar well diffusion method. The bacteria strains and fungal isolates were spread on the Muller Hilton and potato dextrose agar using the sterile cotton swab. The recommended concentration of the test sample in DMSO was introduced in the respective wells of ciprofloxacin and ketoconazole respectively, to serve as positive controls. The plates were incubated at 37^{o}C for 24h in the case of bacteria and 37^{o}C for 48h in the case of fungi. The activity was determined by measuring the diameter of zones showing complete inhibition in (mm) [13].

RESULTS AND DISCUSSION

The Schiff base and metal complexes prepared by condensation of benzaldehyde and urea was successful and were found to have different colors and the ligand have amelting point of 170^{0}C. The decomposition temperatures complexes range from 197-220^{0}C (table 1). These high temperatures suggested good chelating effect of the ligand, which resulted in formation of stable complexes than do an equivalent number of ligands. These figures are relatively high indicating that they are stable compounds [12]. The molar conductance measurement, were carried out in 10^{-3} moldm^{-3}in DMSO at room temperature. The measured molar conductivity are (18-32) Ω^{-1} cm^2 mol^{-1}for Ni(II), and Cu(II) complexes respectively as showed in Table 3. These low values suggested that the complexes are non-electrolyte, which are in agreement with the report by [14]. Table 4 showed the result of magnetic susceptibility of the Qur-complexes taken at room temperature. The values for the magnetic moment for all the metal (II) complexes conformed to a tetrahedral geometry. The compounds have high magnetic moments values suggesting high spin complexes. This is in agreement with report by [15]. Elemental analysis of the complexes were determined using Perkin Elmer Series II CHNS/O Analyzer 2400 241N9022721. The elemental analyses of the metal(II) chelates for C, H, and N showed that the observed and the calculated percentages of the elements are in good agreement and supported 1:2 metal to ligand in all the quercetin complexes. These are shown in Table 6 respectively. The metal analysis of the complex compounds were determined using Atomic Absorption Spectrophotometer (AAS), used to calculate the percentage of metal (II) ion in the complexes. The results obtained were in agreement with the theoretical value (Table 6) which is supported by the report of [16]. The FT-IR spectral information of the Ligand and the prepared complexes were recorded in (Table 5). The absorption around 1659 cm^{-1} is assign for ν(C=N) in

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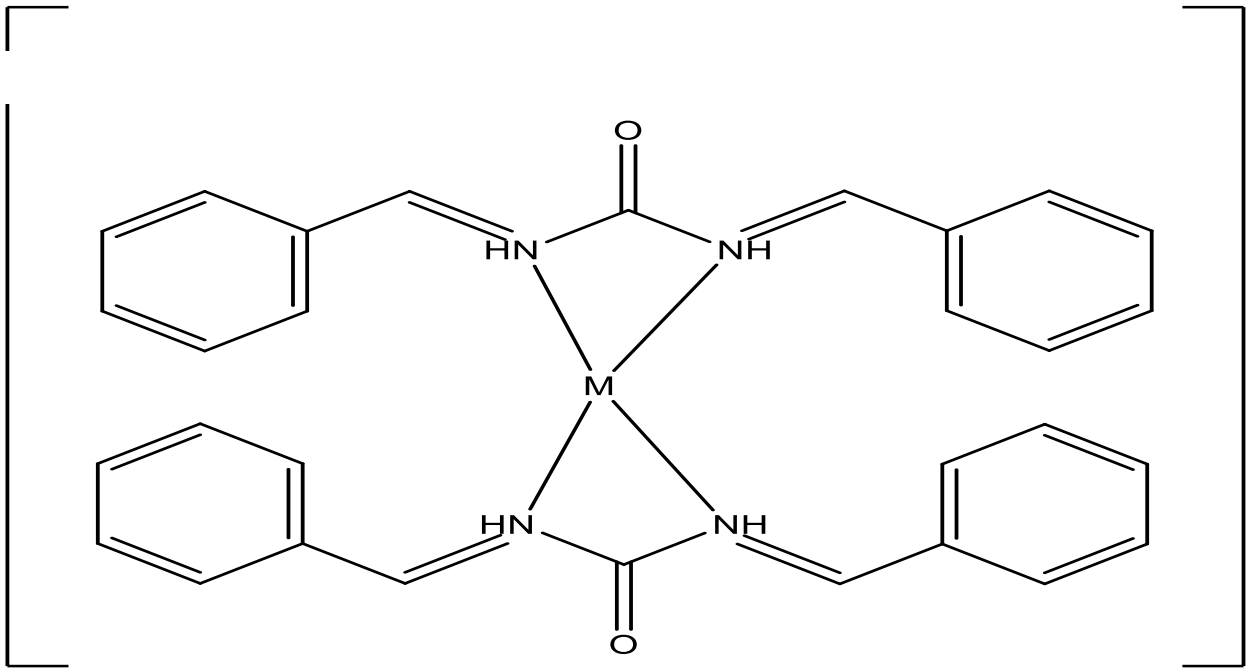
the ligand were found to shifted to higherr frequencies and overlapped with the broad bands at the range 1664-1665 cm^{-1} for the complexes suggesting coordination [15]. The absorption band at 1524 cm^{-1} assign for ν(C=O) were all shifted in the complexes to 1540-1544 cm^{-1} respectively and the carbonyl did not participates in complexes formation [14 and 15]. Moreover, a new band in the complexes appeared at 695-703 cm^{-1}, this band was not found in the free ligands and was assigned to M-O bond [17]. Antimicrobial sensitivity test of the quercetin and its complexes against bacteria carried out in three bacterial isolates: *Escherichia coli pseudomonas* are supplied in the [7] using Ciprofloxacin 500mg/ml as control during the experiment. Zone of inhibition based upon size around each of the disc were measured in millimeter and recorded. The result shows that both the free ligand and the complexes are active against the tested organisms. The data suggest that the antibacterial activity is more at high concentrations. The data also suggest that the complexes have better antibacterial activity than the free ligand. Antifungal sensitivity test of the ligand and its complexes against fungi's carried out in three fungal isolates: *Aspergillusflavus and Candida albicans* are supplied in the (Table 8) using Grisofulvin 500mg/ml as control during the experiment. Zone of inhibition based upon size around each of the disc were measured in millimeter and recorded. The result shows that both the free ligand and the complexes are active against the tested organisms. The data suggest that the antibacterial activity is more at high concentrations. The data also suggest that the complexes have better antibacterial activity than the free ligand. From the analyses of the complexes the general molecular structure has been proposed below:



M= Ni and Cd

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Table 1: Physical properties of the ligand and its metal (II) complexes

Compound	**% Yield**	**Colour**	**Melting/Decomposition Temperature (°C)**
$C_{15}H_{12}N_2O$	**77**	White	170
$[Ni(C_{15}H_{12}N_2O)_2]$	**72**	Dull green	197
$[Cu(C_{15}H_{12}N_2O)_2]$	**71**	Yellow	220

Table 2: Solubility of Schiff base and its Metal (II) Complexes

Compound	Water	Methanol	Ethanol	Toluene	Diethyl ether	Carbon tetrachloride	DMSO
$C_{15}H_{12}N_2O$	**SS**	**SS**	**SS**	**SS**	**SS**	**S**	**S**
$[Ni(C_{15}H_{12}N_2O)_2]$	**SS**	**S**	**SS**	**SS**	**SS**	**SS**	**S**
$[Cu(C_{15}H_{12}N_2O)_2]$	**SS**	**S**	**S**	**SS**	**SS**	**S**	**S**

SS = Slightly Soluble S = Soluble

Table 3: Molar Conductance of the Metal (II) and (III) Complexes

Complexes	Concentration	Electric Conductance ($Ohm^{-1}cm^2$)	Molar Conductance ($Ohm^{-1}cm^2mol^{-1}$)
$[Ni(C_{15}H_{12}N_2O)_2]$	$1x10^{-3}$	$34x10^{-6}$	34
$[Cu(C_{15}H_{12}N_2O)_2]$	$1x10^{-3}$	$18x10^{-6}$	18

Table 4: Magnetic moment values of the Metal (II) Complexes

Complexes	$Xg(g^{-1})$	$Xm(mol^{-1})$	$\mu_{eff}(BM)$	Property
$[Ni(C_{15}H_{12}N_2O)_2]$	$1x10^{-3}$	$4.64x10^{-3}$	$2.41x10^{-3}$	2.43
$[Cu(C_{15}H_{12}N_2O)_2]$	$1x10^{-3}$	$2.36x10^{-3}$	$1.26x10^{-3}$	1.73

Table 5: The Infrared Spectral Data of The Ligand and It's Complexes

Compounds	$\nu(C=N)\ cm^{-1}$	$\nu(M-N)\ cm^{-1}$	$\nu(C=O)\ cm^{-1}$
$C_{15}H_{12}N_2O$	1659	-	1524
$[Ni(C_{15}H_{12}N_2O)_2]$	1665	703	1540
$[Cu(C_{15}H_{12}N_2O)_2]$	1665	695	1544

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Table 6: Elemental and Metal Analysis of the Ni (II) and Cu(II) complexes

Compounds	% Calculated (Found)			
	C	H	N	M
$[Ni(C_{15}H_{12}N_2O)_2]$	67.84(67.01)	4.52(5.21)	10.55(9.87)	11.06(10.9)
$[Cu(C_{15}H_{12}N_2O)_2]$	67.22(68.23)	4.48(4.98)	10.46(10.06)	11.87(11.14)

Table 7: Antimicrobial Activities of Schiff Base Ligand and It's Ni (II) and Cd(III) Complexes

Compounds	Concentration (mg/ml)	Escherichia coli	*klebsella Pseudomonas*
Ligand	60	12	10
	30	9	8
	15	7	6
$[Ni(C_{15}H_{12}N_2O)_2]$	60	16	17
	30	14	15
	15	12	12
$[Cu(C_{15}H_{12}N_2O)]$	60	18	14
	30	16	12
	15	14	10

Table 8: Antifungal Activities of Schiff Base Ligand and It's Ni (II) and Cu (II) Complexes

Compounds	Concentration (mg/ml)	Escherichia coli	*Pseudomonas*
Ligand	60	10	11
	30	8	9
	15	6	8
$[Ni(C_{15}H_{12}N_2O)_2Cl_2]$	60	16	17
	30	15	14
	15	12	12
$[Cu(C_{15}H_{12}N_2O)_2]$	60	18	16
	30	17	14
	15	14	11

CONCLUSION

This study reports the successful synthesis of Schiff base from Benzaldehyde and urea, and its Ni(II) and Cu(II) metal complexes by reaction of methanolic solution of the Schiff base and the respective metal(II) and (III) chloride. From the spectral data it was possible to determine the type of coordination of the ligand and its metal complexes. In the complexes, it is concluded that the ligand is bidentate, coordinates through azomethine nitrogen atoms and the geometry of the complex is octahedral. All the compounds have good antibacterial activity against the tested organism.

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PLATE 1 SHOWING THE ZONE OF INHITION OF SHIFFBASE

PLATE 2 SHOWING THE ZONE OF INHIBITION OF NICKEL COMPLEX

PLATE 3 SHOWING THE ZONE OF INHITION OF CADMIUMCOMPLEX.

REFERENCES

1. Dincer S. (1996): Synthesis of some cytosine Schiff bases. Indian Journal Chem. 33B 1335-1336.
2. Katyal, M. Dutt, J. (1975) Analytical Application of Hydrazones. Talanta. 22, 151.
3. Kamat .P and Yangar R. (2005) Preparation of zinc metal complex (DMAPIMP) Zn and development of HPLC chromatographic method for its analysis. J. chem. Res; 3(1): 188-198
4. Sheikh A.A and Muhammad M. (2010). Synthesis and analytical studies of sulfadimididne-imine Schiff base complexes with Ni(II) and Co(II). The Biol (E. journal of life science; 1(2): 37-40
5. Kumar S. and Dhar D.N. (2009). Application of metal complexs of Schiff base. Journal of scientific and industrial research; 68: 181-187

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ONLINE ISSN: 2992-5479
PRINT ISSN:2992-605X

6. Salawu O.W and Abdilsalam A.O. (2011) Synthesis, characterization and biological activities of Cd(II) complexes with hydrazine ligands; Scholars Research Library Der PharmaChemica. 3(4), 298-304.
7. Monfareda, H.H Omid P, and Christophe J.(2006): Synthesis and Spectral Characterization of Hydrazone Schiff bases Derived from 2,4-Dinitrophenylhydrazine. Crystal Structure of Salicylaldehyde-2,4-Dinitrophenylhydrazone. Z.Naturforsch. 62b, 717–720;
8. Hassan Hosseini Monfareda, Zahra Kalantaria, Mohammad-Ali Kamyabia, and Christoph Janiak (2007) Synthesis, Structural Characterization and Electrochemical Studies of a Nicotinamide-bridged Dinuclear Copper Complex derived from a Tridentate Hydrazone Schiff Base Ligand. Z. Anorg. Allg. Chem.10.1002, 1945-1948
9. Mishara A. and Sari M. (2008). Synthesis, structural and biological studies of some Schiff base and their metal complexes. Metal base drugs; 27; 875-878.
10. Bhatt V.D. Ray A, (2001). Synthesis, characterization and electrical conductivity of polyestercontaining azomethine linkages. International journal of Polymeric Materials, 49: 355-366.
11. Ahmed, A and Akhtar F (1983): Cu(II) and Ni(II) Complexes with a tetradentate Schiff base derived from 2-hydroxyl-1-napthaldehyde and ethylenediamine; Indian Jour Chem. 20A, 737-758q1
12. Ahmed A, Akhtar F. Cu(II) and Ni(II) complexes with a tetradentate schiff base derived from 2-hydroxyl-1-naphthaldehyde and ethylenediamine. Indian Jour Chem. 1983;20A:737-758.
13. Pooja, S., Rajshree, K., Renuka, C., (2020). *Complexes of Pyrimidine Thiones: Mechanochemical Synthesis and Biological Evaluation. Asian Journal of Chemistry; Vol. 32, No. 10 (2020), 2594-2600.*
14. Ezalden J. Alobaidi and Omar H. Al-obaidi, (2023). Synthesis and Spectroscopic Mixed Ligands Complexes of flavonoid and their Biological Study in Antitumor Activity for Treatment Breast Cancer Cell Line (MCF-7), Inhibitory Effect on Xanthine Oxidase and Antimicrobial. *Journal of Survey in Fisheries Sciences*, 10(3S) 2865-2889
15. Safana Ahmed Farhan (2013). Study on the Interaction of Copper (II) Complex of Morin and its antimicrobial effect. *Int. J. Chem. Sci.: 11(3), , 1247-1255*
16. Hassan, R., Ali, H., Gamai, A., Saleh S., Hussein, A., and Hassan, A. I. (2013). Preparation , Characterization and Atomic Absorption Spectroscopic Determination of Some Metal Complexes. Glipizide Scholar Research Library. Der Pherma Chemica 5(6): - 163
17. Emad M. Atta, Khaled H. Hegab, Ahmed A.M. Abdelgawad, Abdelghany A, Youssef (2019). Synthesis, characterization and cytotoxic activity of naturally isolated naringin-metal complexes. Saudi Pharmaceutical Journal 27, 584–592.

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