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SYNTHESIS, CHARACTERIZATION AND ANTIMICROBIAL ACTIVITY OF SCHIFF BASE DERIVED FROM GLYCINE AND 4-CHLOROBENZOPHENONE AND ITS METALS COMPLEXES OF Cr(II) and Co(II)

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ABSTRACT

The Schiff base ligand was synthesized using glycine and 4-chlorobenzophenone, and then reacted with metal salt of chromium, and cobalt to form the complex of the metals. The ligand and the complexes were characterized using IR-spectrophotometer, and peaks were analysed which confirmed the formation of (M-N) and (M-O) bond in the complexes at around 503- 847, also solubility test, water of crstallisation, magnetic susceptibility, melting Point and conductivity of the ligand and complexes were carried out in the research. Lastly, the compound were tested against some strain of bacteria (Salmonella typhi, Staphylococcus aureus, Escherichia coli) in which the activity of the ligand and some complex were detected in the bacteria, the cobalt complex showed higher activity than the chromium complex and the ligand.

Keywords: Antimicrobial activity, Metal complex**,** Schiff base ligand

INTRODUCTION

Schiff base is a compound with general structure of $R_2C=NR`$ where R is not hydrogen atom. Schiff bases are condensation products of primary amines and carbonyl compounds and they were discovered by a German chemist, Hugo Schiff who received a Nobel prize winner in 1864 (Brodowska and Elzbieta, 2014). Structurally, Schiff base (also known as imine or azomethine) is an analogue of ketone or aldehyde in which the carbonyl group (C=O) has been replaced by an imine or azomethine group (Figure 1). Schiff base ligands are essential in the field of coordination chemistry, especially in the development of complexes of Schiff bases because these compounds are potentially capable of forming stable complexes with metal ions.

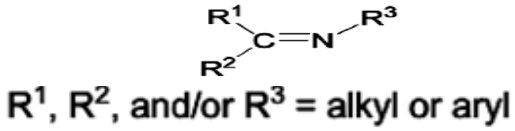


Figure 1: General structure of Schiff bases

Schiff base are the compound containing azomethine group (-HC=N-). They contain condensation products of ketone or aldehydes (aldehyde and ketone) with primary amines. Formation of Schiff base generally takes place under acids or base catalysis or with heat. The common Schiff base are crystalline solids, which are feebly basic but atleast some form insoluble salt with strong acids. Schiff base are used as intermediate for the synthesis of amino acids or as ligands for preparation of metal complexes having a series of different structures (Xavier and Srividhya, 2014). Schiffbase behaves as a flexi-dentate ligand and commonly co-ordinate through the O atom of the de-protonated phenolic group and the N atom of azomethine group. In Schiff base azomethine nitrogen and other donor atoms like oxygen play a vital role in co-ordination chemistry (Xanvier and Srividhya).

Metals complexes [Co(II), Cu(II), Zn(II) ions] of Schiff base having played a central role in the development of co-ordination chemistry, especially in the development of Schiff base complexes, because Schiff base complexes are potentially capable of forming stable complexes with metal ions, many Schiff base complexes show excellent catalytic activity in various reactions at high temperature and in the presence of moisture (Xanvier and Srividhya, 2014).

Schiff bases, also known as azomethines due to they have RC=N group, play important roles in biological systems. They are facing a growing interest due to their various applications, e.g as anticancer, antibacterial, antiviral, antifungal, and about their other biological properties. (In 2010 Aliyu and Adamu) reported the synthesis of Schiff base using semicarbazide hydrochloride solution in ethanol with benzyldehyde, ammonia solution was used as neutralizing agent and morpholine was used as precipitation agent. And three new metal complexes Zr(IV), Pb(II), U(VI), the ligand and the metal complexes have been characterized by various analytical techniques which confirmed the synthesis of the new compounds. The antibacterial activity of the synthesized compound was done against *E. Coli* and *Pacillus sp.* Results proved that the Zr and Pb complexes exhibit significant antibacterial activity over other compound. (Benabdullah *et al*., 2004) reported the synthesis of Schiff base from condensation of ethylenediamine with salicylaldehyde, and another one from 2-hydroxy-1-pyridinecarboxaldehyde, and 2-hydroxy-1-naphthaldehyde which were all prepared by condensation of aldehyde and di or triamine in 2:1 ratio and refluxing in ethanol for two hours. The three complexes of Cu, Ni and Mn exhibited a new absorption band in the visible region which can be associated with d-d transition, their wavelength are 574, 703 and 579nm for the L1, L2 and L3 complexes respectively. The differences among these values are probably due to the change in nature of the metal environment in the three complexes. And antibacterial activity was carried out against the bacterial strain (*e.coli, staphylococcus aureus*,), the antibacterial indicated that Cu is more active against the bacterial while Ni and Mn are moderately active.

In this research, we aimed to synthesize, characterize the series of Cr(II) and Co(II) metal complexes of Schiff base derived from glycine and 4-chlorobenzophenone and investigate their biological activity.

MATERIALS AND METHODS

Reagents used in this study were of analytical grade. The glass wares used were washed thoroughly with detergent, rinsed with distilled water and dried in an oven. Melting point and decomposition temperature were determined using a melting

point apparatus(gallenkamp). Molar conductivity measurement was carried out using Jenway conductivity meter model 4010, while magnetic susceptibility measurement was done on Sherwood (29275) magnetic susceptibility balance at 25ºC. IR spectral analysis was carried out using FTIR Cary 630 (Agilent Technology) model in the range of 4000 – 600 cm^{-1}

Preparation of the Schiff base Ligand

1.5 g (0.02 mmol) of Glycine (white precipitate) was accurately weighed and dissolved in a beaker with 40:50 ratio of water to ethanol so as to dissolved it completely and 4.33 g (0.02 mmol) of 4-Chlorobenzophenone (white crystal) was accurately weighed and dissolved in separate beaker with 25 cm^3 of ethanol, then the two solution were mixed and the solution were refluxed for complete three hours and then allowed to cooled and filtered the white precipitate formed and then dried and weighed The percentage yield of the product was calculated using the equation shown below;

$$\% \text{ Yield} = \frac{Experimental Yield}{Theoretical Yield} * 100 \quad (1)$$

Preparation of Metal (II) Complexes

The metal complexes were prepared by mixing 1:2 molar ratios of $CrCl_2.2H_2O$ or $CoCl_2 .2H_2O$ and the ligand respectively in absolute ethanol and dissolved in separate beaker with 25cm^3 of ethanol, then the two solution were mixed and the solution were refluxed for complete 3 hours and then allowed to cooled and filtered the colored precipitate formed and then dried and weighed.

The percentage yield of the product was calculated using the formula expression (1) above;

Antimcrobial Activity

9.5 g of mullerhinton agar was dissolved in 250ml of sterile distilled water and warmed gently to completely dissolved the agar on hot plate, then the media was sterilized using an autoclaved machine at 121 ^{O}C for 15min, and then the media was allowed to cooled and poured in to petridishes, and then allowed it to solidified. The test organism were standardized using Macfarland turbidity standard, the the media was inoculated with the standardized test organism (E.coli, Salmonellla Typhi and Staphylococcus aureus) using sterile swab stick all over the surface of the media then agar well diffusion was made by drilling wells in the agar media using sterile cork borer, and then the stock solution was separately prepared in the test tubes (i.e for Schiff base, chromium complex and cobalt complex) making varying concentrations using serial dilution, then the Schiff base and the metal complexes solution of varying concentration with their control using ciprofloxacin(cf) were incoorperated in to the wells of different cultures using syringes and allowed it to stand upright in an incubator at 37 ^{0}C for 24hours after which the zone of inhibition was observed.(khan et al.,2014)

RESULTS AND DISCUSSION

Table 1: Presentation of physical properties of the Schiff Base and its Metal (II) Complexes

Compounds/ Complex	Molecular Formula	Colour	Water of crystallization(%)	Percentage yield	Molecular Weight	Melting Point/^{0}C	Decomposition temperature
Ligand	$C_{15}H_{12}O_2Cl$	White	-	95	259.5	224	-
$[CoL_2]$	$C_{30}H_{22}O_3ClCo$	Blue	60	89	577.93	_	263
$[CrL_2]$	$C_{30}H_{22}O_3ClCr$	White	50	90	573.93	_	286

Table 2: Result of solubility test of the ligand and complexes.

Ligand/ Complexe	Distilled water	Ethanol	Methanol	Acetone	n-hexane	Chloroform	DMSO
Ligand	IS	S	SS	SS	SS	S	S
$[CoL_2]$	IS	S	S	SS	SS	S	S
$[CrL_2]$	IS	S	S	SS	SS	S	S

Table 3: Infrared ray result of ligand and its complexes

Compound	V(O-H)	V(C=O)	V(C-O)	V(C-N)	V(C=N)	V(C=C)	V(C-H)	V(M-N)	V(M-O)
Ligands	3283	1689	1149	1089	2113	1652	3089	_	_
$Cocl_2$	3283	1704	1279	1320	2113	1581	3041	847	668
$Crcl_2$	3528	1652	1302	1302	2121	1581	3037	732	698

Table 4: Magnetic susceptibility data of metal(II) schiff base complexes

Complexes	μeff(B.M)	Magnetic Property	Number of unpaired electron
Cocl	6.40	paramagnetic	4
Crcl	2.7	Diamagnetic	0

Table 5: Conductivity result of Schiff base and its complexes

Ligand/Complex	S/cm
Cr complex	$0.09x10^{3}$
Co complex	$0.08\ x10^{3}$

Table 6: The result of anti-bacterial activity of the schiff base and the complexes

Complexes	Concentration (ug/ml)/ Zone of inhibition (mm)											
	Staphylococcus Aureus				*Escherichia Coli*				*Salmonella typhi*			
	4000	2000	1000	500	4000	2000	1000	500	4000	2000	1000	500
Ligand	14	11	9	-	12	9	8	_	10	_	_	_
[CoL_2]	18	16	12	10	17	15	11	8	12	9	7	_
[CrL_2]	13	10	9	_	14	12	9	8	10	8	_	_
Control				*25*				*30*				*32*

Control= ciprofloxacin

Discussion

The Schiff base ligand was prepared by condensation of glycine and 4-chlorobenzophenone to obtain white crystal with high yield (95%) and meltig point 224 °C (table 1). The metal complexes , Cr(II) and Co(II) complexes were synthesized and found to be of various color with percentage yield of 90% and 89%

Physical analysis

Physical analysis was presented in table 1, the Schiff base was prepared from glycine and 4-chlorobenzophenone which obtain white colour crystal with high percentage yield (95%) and melting point of 224 °C. the metal complexes Cr(II) and Co(II) were synthesized and found to be of white and blue colour with percentage yield of 90 % and 89 % , the decomposition temperature of the chromium was 286 °C and cobalt 263 °C, decomposition temperature of the metal complexes is higher than that of the melting point of the ligand indicating that complexation has taken place.

Solubility test

The solubility of the Schiff base and its complexes, the Schiff base was found to be insoluble in water, slightly soluble in methanol, acetone and n-hexane, but soluble in ethanol, chloroform and DMSO. Similarly, the complexes were both found to be insoluble in water, slightly soluble in acetone and n-hexane, but soluble in ethanol, methanol chloroform and DMSO (Table 2)

FTIR Analysis

The FTIR spectra shows a broad peaks around 3283 to 3528 cm^{-1} this is due to presence of water in the compound which initially used to dissolve the amino acid reagent completely, the Ligand shows V(C=N) peak which was found to be 2113.04cm^{-1} this due to formation of azomethine group in the ligand, peak at 1089.9cm^{-1}indicating the presence of V(C-N) bond in the ligand. The complexes spectra shown the peaks around 1689cm^{-1}, 1149cm^{-1} , 2113cm^{-1} , 1089$cm^{-1}$and 3283cm^{-1} which assigned to V(C=O), V(C-O), v(C=N), V(C-N) and V(O-H) respectively. In comparison, some bands are shifted down to lower frequency in complexes as compared ligand peaks band; this confirmed the formation of ligand and its metal complexes. However, new peaks appeared around 732-847cm^{-1} and 668-698cm^{-1} in complexes these are assigned to V(M-N) and V(M-O) respectively, this also confirmed the coordination between metal ion and ligand. These results were in line with reported FTIR metal complexes in literature Thakurata *et al*. (2014). (table3)

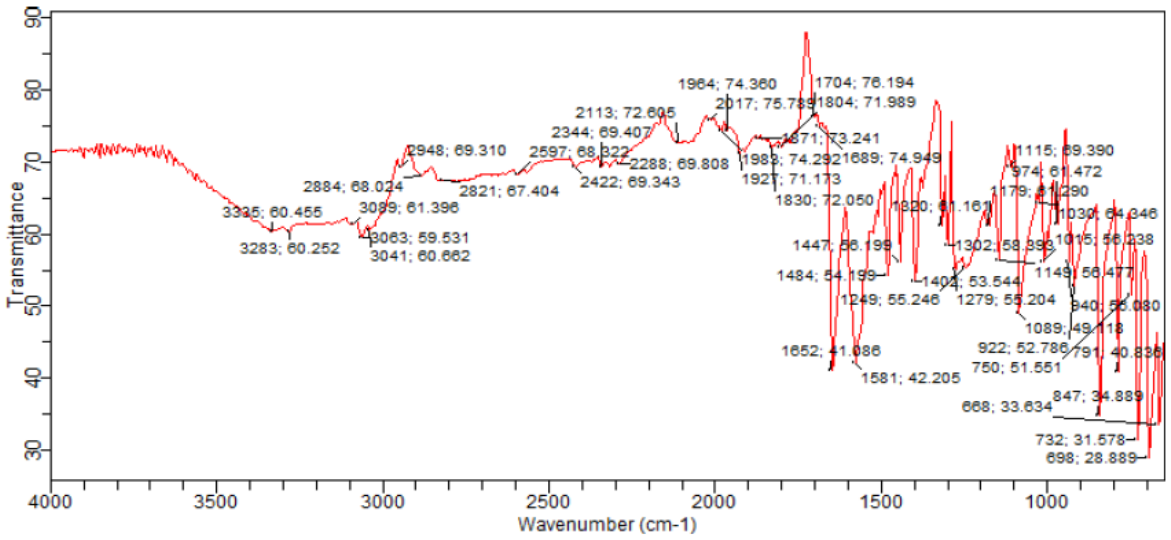


Figure 2: FT-IR of Cobalt complex

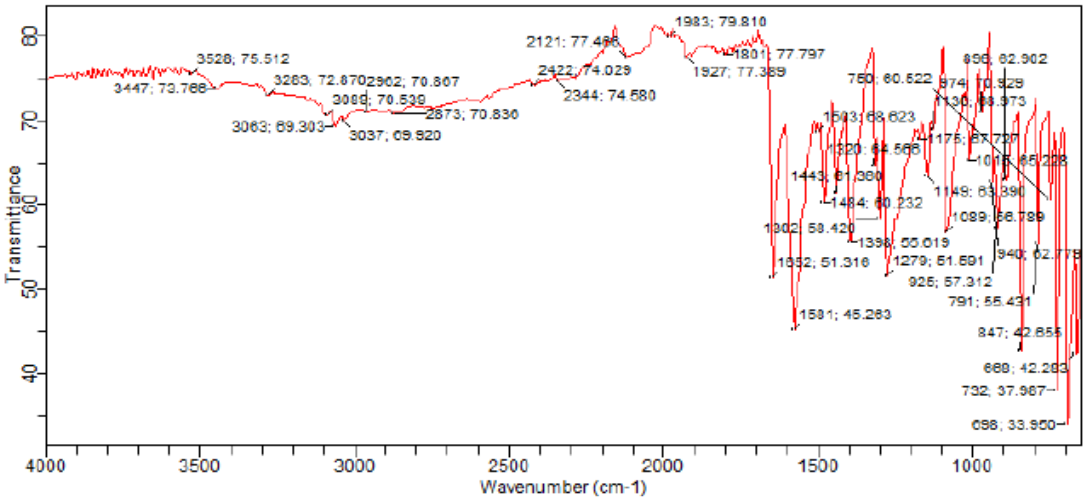


Figure 3: FT-IR of Chromium complex

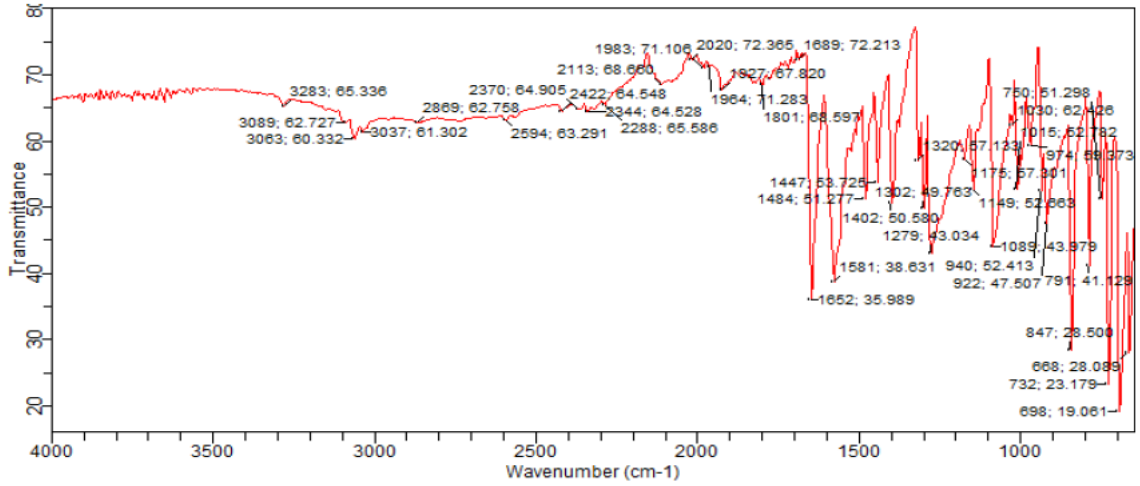


Figure 4: FT-IR of the Schiff base

Magnetic susceptibility

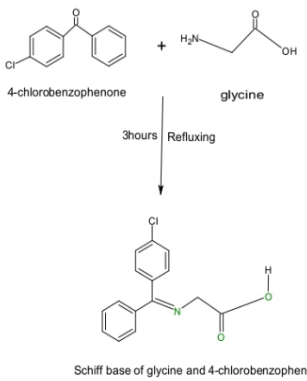
Magnetic susceptibility measurement of the complexes (Table 4), Magnetic susceptibility measurement values for Co(II), and Cr(II) complexes at room temperature. The values for the complexes indicated that they are both paramagnetic wich both shows the present of unpaired electrons. (table 4)

Conductivity Measurement

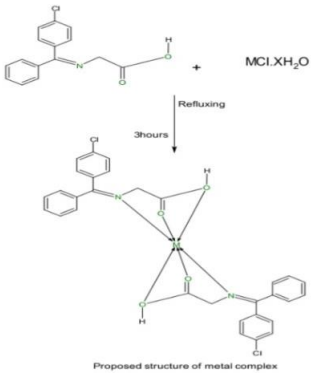
The conductivity of Cr(II) and Cobalt(II) metal complex was measured in DMSO. The values were obtained which shows are non-electrolytic nature . the conductance electrolyte metal complexes in DMSO ranges from 1-50ohm/cm/mol. (table5)

Antimicrobial activity

The antibacterial activity of Schiff base and metal complexes was carried out on three isolate(staphylococcus aureus, salmonella typhi and Escherichia coli) using well diffusion method by using DMSO as a solvent(table 6) . The result showed both ligand and metal complexes are found to be moderately active against all tested bacteria, but metals exhibit higher activity than that of the ligand and activity increase with increase in concentration. This is probably due to chelation in metal complexes.



Scheme 1: proposed structure of the ligand



Scheme 2: proposed structure of the complex

CONCLUSION

The Schiff base and its metal complexes of Cr(II) and Co(II) have been synthesized and characterized by various analytical techniques. The FTIR shows that complexation has taken place due to the presence of OH in the ligand and metal complexes, and presence of M-N and M-O. the electrical conductivity carried out for the metal complexes shows both are non-electrolytic in DMSO solvent, the antibacterial activity of Schiff base and metal complexes against (staphylococcus aureus, salmonella typhi and Escherichia coli)show a very high respond in the metal complexes than that of the Schiff base in which the inhibition zone of the complexes were very high this shows that the metal complexes have high antibacterial activity.

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