BAJOPAS Volume 11 Number 2 December, 2018 http://dx.doi.org/10.4314/bajopas.v11i2.22

Bayero Journal of Pure and Applied Sciences, 11(2): 176 - 179
Received: September, 2018
Accepted: December, 2018
ISSN 2006 – 6996

SYNTHESIS, CHARACTERIZATION AND ANTIBACTERIAL STUDIES OF TETRADENTATE SCHIFF BASE AND THEIR METAL (II) COMPLEXES DERIVED FROM 4-(BENZENEAZO) SALICYLALDEHYDE AND ETHYLENEDIAMINE

Sani, M.[1] and Nuraddeen, A[2].
[1]Department of Applied Chemistry, Federal University Dutsin-ma, P.M.B 5001, Katsina, Katsina State,
[2]Department of Pure and Industrial Chemistry, Umaru Musa Yar'adua University, Katsina P.M.B 2218 Dutsinma Road Katsina State
Correspondence Author: smuhammad4@fudutsinma.edu.ng: 08063962618

ABSTRACT
A tetradentate Schiff base was synthesized by the reaction of 4-(Benzeneazo) Salicylaldehyde and Ethylenediamine in their ethanolic solutions. The corresponding metal (II) complexes of the synthesized Schiff base were obtained by refluxing the ethanolic solutions of $CoCl_2$, $CrCl_2$, and $MnCl_2$ salts. The physical properties of the Schiff base and its corresponding metal (II) complexes were investigated. The melting point temperature of the Schiff base was 200°C, while the decomposition temperatures of the complexes were 230 °C, 210 °C and 220 °C for Co (II), Cr (II), and Mn (II) complexes respectively. The magnetic susceptibility measurement reveals that all the complexes are paramagnetic in nature; the conductivity measurement shows that all the complexes are non-electrolytic in nature. The formation of the Schiff base and its metal (II) complexes were confirmed using FTIR spectroscopy. Both the Schiff base and its metal (II) complexes were found active against Staphylococcus aureus and Escherichia coli.
Key words: Schiff base, metal complexes, magnetic susceptibility, conductivity, antibacterial.

INTRODUCTION

Schiff bases are important class of organic compounds which were discovered by Hugo Schiff in 1864 (Hussain *et al*., 2014). They are functional groups that contains a carbon-nitrogen double bond with the nitrogen atom connected to an aryl or alkyl group (da Silvaa *et al*., 2011; Sahu *et al*., 2012).

A biologically important aldehydes pyridoxal *phosphate* is the active form of vitamin B6. Vitamin B6, which serves as a coenzyme by forming an imine with an amino acid group of enzymes (Muhammad *et al*., 2011). The Schiff bases are widely studied due to their synthetic flexibility, selectivity and sensitivity towards the central metal atom, structural similarities with natural biological compounds as well as the presence of azomethine group (-N=CH-) which is important in explaining the mechanism of their transformation biologically (Shargi and Nasseri, 2003).

Aim of the research

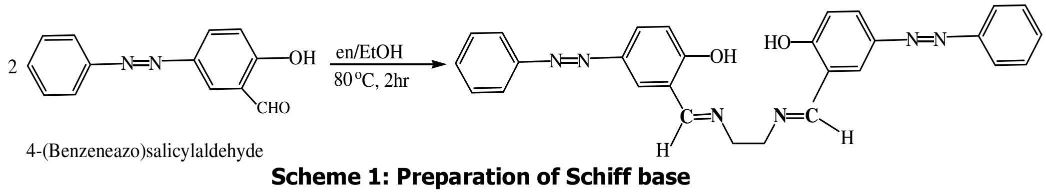
The aim of this research is to prepare, characterize and study the antibacterial potency of the 4-(Benzeneazo) salicylaldehyde and ethylenediamine (en) Schiff base and its Cr (II), Co (II) and Mn (II) complexes.

MATERIALS AND METHODS

All chemicals and solvents used in this research were of Analytical grade and were used as purchased without further purification. The glass wares used were washed with detergent and then rinsed with distilled water and dried in an oven at 110°C. Infrared spectral analysis was carried out using FTIR Cary-630(Agilent Technology model) spectrophotometer in the range of 500- 4000cm^{-1}, Melting point and decomposition temperatures were determined using BUCHI-510 melting point machine. Electrical conductivity measurements were recorded using conductivity meter jenway 4010 model, Magnetic susceptibility of the complexes was determined using MBS MK1 Magnetic susceptibility balance at room temperature. The antibacterial activity was studied using two bacterial isolate (*Staphylococcus aureus* and *Escherichia coli*)

Synthesis of Schiff Base:

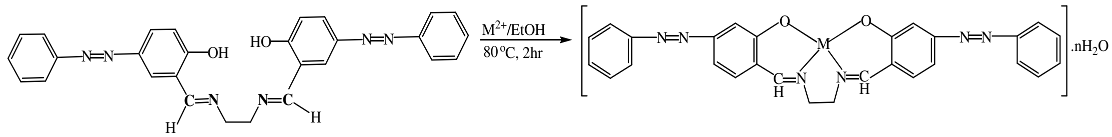
0.01mol of Ethylenediamine (en) was slowly added to a solution of 0.02mol 4-(Benzeneazo) Salicylaldehyde in 30cm^3 ethanol.After refluxing the reaction mixture for 2hrs the precipitate was washed several times with ethanol and dried at 50°C overnight.(Ajibade *et al.*,2015).



Scheme 1: Preparation of Schiff base

Synthesis of Schiff Base Metal Complexes:

The complexes were prepared by the addition of 1mmol of the metal salt $CuCl_2$ into $10cm^3$ of distilled water in which a hot ethanolic solution of 1mmol of the Ligand was added. The mixture was then refluxed for two hours. The precipitated Solid was filtered and washed with ethanol diethyl ether and finally dried at 50°C over night (Ajibade *et al.,* 2015). Same procedure was repeated for Co (II) and Mn (II).



Scheme 2: Preparation of metal complexes
Where M = Cr (II), Co (II), and Mn (II) and n = number of moles

RESULTS AND DISCUSSIONS

The results in Table 1 indicate that the Schiff base and its corresponding metal (II) complexes are colored. The color orange yellow is for the Schiff base (Archana*, 2013* and Hassan, *et al*; 2006) while green, dark brown, and Indian red colors are for Cr(II), Mn(II), and Co(II) complexes respectively. The change in color of the Schiff base from orange yellow to green, dark brown, and Indian red was due to complexation which resulted into the formation of coordination compounds. The melting point/decomposition temperatures for the Schiff base and its corresponding metal (II) complexes range between 200°C – 230°C (Table 1). The results compared physical properties of both Schiff base and its corresponding metal (II) complexes. There were similarities with a report by Mustapha *et al*., 2009 and it indicated the high stability of the compounds.

Table 1: Physical properties of Schiff base and its Metal (II) Complexes

Compound	Molecular formula	Colour	Decomposition Temp. (°C)	M P (°C)
Schiff base	$[C_{28}H_{24}N_6O_2]$	Orange yellow		200
Cr (II) complex	$[Cr(C_{28}H_{22}N_6O_2)].H_2O$	Green	210	
Mn (II) complex	$[Mn(C_{28}H_{22}N_6O_2)].2H_2O$	Dark brown	220	
Co (II) complex	$[Co(C_{28}H_{22}N_6O_2)].4H_2O$	Indian red	230	

The FTIR results were reported in Table 2 below, a band appeared at $1633cm^{-1}$ assigned to ʋ (C=N) stretching vibration which is an important feature of Schiff base and it was reported in the literature (Hassan *et al.,* 2006, Imtiyaz and Athar*.,*2015). Absorption band also appeared in the free ligand at $1283cm^{-1}$ can be assigned to ʋ (C-O) stretching vibration.

In all the metal (II) complexes, there were an observable shift in ʋ (C=N) from $1633cm^{-1}$ to $1644cm^{-1}$, $1629cm^{-1}$ and $1614cm^{-1}$ this indicate the stretching vibration of the azomethine groups and possible formation of the complexes (Yustina, 2009). The appearance of weak absorption bands in all the metal complexes at the range of 592 – 574 cm^{-1} can be attributed to the stretching vibration of Metal – Nitrogen (M – N) and also appearance of weak absorption bands at all the metal complexes at the range of 481- $422cm^{-1}$ can be attributed to the stretching vibration of Metal-Oxygen (M-O) which is similar to what was obtained in the literatures (Deoghoria *et al.,* 2004 Bagihalli and Avaji, 2008 and Shahabadi and Kashanian, 2010).

Table 2: The Infrared Spectral Data Schiff base and its Metal (II) Complexes.

Molecular formula	ʋ C=N (cm^{-1})	ʋC-O (cm^{-1})	ʋM-N (cm^{-1})	ʋM-O (cm^{-1})
$[C_{28}H_{24}N_6O_2]$	1633	1283	–	–
$[Cr(C_{28}H_{22}N_6O_2)].H_2O$	1614	1201	574	481
$[Mn(C_{28}H_{22}N_6O_2)].2H_2O$	1629	1287	589	422
$[Co(C_{28}H_{22}N_6O_2)].4H_2O$	1644	1205	592	452

The Molar conductance measurement in DMSO are in the range 7.5-10.1 $ohm^{-1}cm^2mol^{-1}$ (Table 3).These values are relatively low showed that the complexes are non electrolytes (Raju and Balasubramanian, 2011). The low values indicate that the compounds are neutral. This implies low ions in the solution of the compounds and the conductivity meter measures ion in solution.

Table 3: Conductivity Measurement of the Metal (II) Complexes

Compounds	Electrical Conductance ($Ohm^{-1} cm^2$)	Molar Conductance ($Ohm^{-1}cm^2mol^{-1}$)
$[Cr(C_{28}H_{22}N_6O_2)].H_2O$	7.5×10^{-6}	7.5
$[Mn(C_{28}H_{22}N_6O_2)].2H_2O$	12.2×10^{-6}	12.2
$[Co(C_{28}H_{22}N_6O_2)].4H_2O$	10.1×10^{-6}	10.1

The values of Magnetic moments for the metal complexes confirmed tetrahedral geometry for the transition metal ions and their magnetic moments values suggested high spin complexes and paramagnetic in nature as shown in table 4.

Table 4: Effective Magnetic Moments (μeff) of the Complexes

Compounds	X_g ($erg\ G^{-2}\ g^{-1}$)	X_M ($ergG^{-2}\ mol^{-1}$)	μ_{eff} (B.M)	Property
$[Cr(C_{28}H_{22}N_6O_2)].H_2O$	118.71×10^{-07}	6.25×10^{-03}	3.86	Paramagnetic
$[Mn(C_{28}H_{22}N_6O_2)].2H_2O$	293.76×10^{-07}	15.55×10^{-03}	5.90	Paramagnetic
$[Co(C_{28}H_{22}N_6O_2)].4H_2O$	159.90×10^{-07}	8.53×10^{-03}	4.51	Paramagnetic

The *in vitro* antibacterial activity of the ligand and its metal (II) complexes on the bacterial isolates (*Staphylococcus aureus* and *Escherichia coli)* using DMSO as a solvent was carried out. The complexes of Mn (II) showed no activity at 15 µg/disc against *Staphylococcus aureus* and *Escherichia coli* while Co (II) compound showed no activity against *Staphylococcus aureus.* The results revealed that both the ligand and the metal (II) complexes are relatively good antibacterial agents as shown in Table 5. Highest inhibition zone (23mm) was observed with Cr (II) complex against *Staphylococcus aureus.* The compounds generally showed lower activities in comparison to the standards (Ceftriaxone).

Table 5: Antibacterial Profile of the Ligand and Complexes

Compounds	Zone of Inhibition (mm)/Concentration (µg)					
	Staphylococcus ***aureus***			***Escherichia. Coli***		
	15	**30**	**60**	**15**	**30**	**60**
$[C_{28}H_{24}N_6O_2]$	9	8	10	06	10	12
$[Cr(C_{28}H_{22}N_6O_2)].H_2O$	18	15	23	10	12	16
$[Mn(C_{28}H_{22}N_6O_2)].2H_2O$	06	12	14	06	12	14
$[Co(C_{28}H_{22}N_6O_2)].4H_2O$	06	12	16	10	12	14
Standard(Ceftriaxone)	25	28	23	24	30	27

CONCLUSION

Based on the results obtained, it can be concluded that 4-(Benzeneazo) Salicylaldehyde and Ethylenediamine were successfully used as the starting materials for the preparation of Schiff base and its metal (II) complexes. Antibacterial evaluation of the ligand and metal complexes showed the potency against *Staphylococcus aureus* and *Escherichia coli*

REFERENCES

Ajibade Peter A. and Ikechukwu P. Ejidike.,(2015). Synthesis, Characterization and Biological Studies of Metal (II) Complexes of (3E)-3-[(2-{(E)-[1-(2,4-Dihydroxyphenyl) thylidene] amino}ethyl)imino]-1-phenylbutan-1-one Schiff Base. *Journal of Chemical and Pharmaceutical Research*, 20(6):9788-802

Archana S. (2013): Synthesis and characterization of Schiff base derived from salicylaldehyde and thiohydrazones and its metal complexes. *Advances in Applied Science Research*: 4(4) 152-154

Bagihalli**,** G.B. and Avaji, P.G. (2008): Synthesis, spectral characterization, in vitro antibacterial, antifungal and cytotoxic activities of Co (II), Ni(II) and Cu(II) complexes with 1,2,4-triazole Schiff bases .*European Journal of Medicinal Chemistry* 4(3) 2639-2649

da Silvaa, C. M., da Silvaa, D. L., Modolob, L. V. A., Rosemeire, B. M. C., Fátima, V.B. and de, A. (2011). Schiff bases: A short review of their antimicrobial activities. *Journal of Advanced Research*: 2(1) 1–8.

Deoghoria, S., Mustafa, G., Lu, T.H. and Chandra, S.K. (2004): Synthesis, characterization and X-ray crystal structure of copper (II) complexes with unsymmetrical tetradentate Schiff base ligands: first evidence of Cu (II) catalyzed rearrangement of unsymmetrical to symmetrical complex. *Indian Journal of Chemistry*: 43(A) 329-332

Hassan, H. M. Omid, P. and Christoph J. (2006): Synthesis and Spectral Characterization of Hydrazone Schiff Bases Derived from 2, 4-Dinitrophenylhydrazine and Crystal Structure of Salicylaldehyde-2, 4 Dinitrophenylhydrazone. *Naturforsch*: 64(b) 717-720

Hussain, Z., Emad, Z. Y. A. and Ali, A. A. (2014). Synthesis and characterization of Schiff's bases of sulfamethoxazole. *Organic and Medicinal ChemistryLetter* 4(1):1–4.

Imtiyaz, R. P. and Athar A. H. (2015): Synthesis of Schiff Base Complexes of Mn (II) and Co (II) and their Catalytic Oxidation towards Olefins and Alcohols. *Canadian Chemical transaction:* 3(1) 65-71

Muhammad, A. A., Karamat, M. and Abdul W. (2011). Synthesis, characterization andbiological Applications of Schiff bases. International Conference on Chemistry and Chemical Process 10(A): 1-7.

Mustapha, A., Reglinski, J. and Kennedy, A.R. (2009): The use of Hydrogenated Schiff Baseligands in the synthesis of Multi-metallic compounds. *Inorganic Chemistry Acta:* 36(2):1267-1274

Raju, V.V. a nd Balasubramanian, K.P. (2011). Synthesis , spectral characterization , catalytic and biological studies of new Ru (II) carbonyl Schiff base complexes of active amines. *Natural Science* 3(7): 542–550.

Sahu, R., Thakur, D.S. and Kashyap, P., (2012). Schiff Base: *An Overview of its Medicinal.*Chemistry Potential for New Drug Molecules. *International Journal of Pharmaceutical Sciences and Nanotechnology* 5(30: 1757–1764.

Shahabadi, N.; Kashanian, S. (2010): DNA binding and DNA cleavage studies of a water soluble cobalt (II) complex contains dinitrogen Schiff base ligand: The effect of metal on the mode of binding. *European Journal Medicinal Chemistry:*4(5): 4239 – 4245

Sharghi, H. and Nasseri, M. A. (2003): Schiff-Base Metal (II) Complexes as New Catalysts in the Efficient, Mild and Regioselective Conversion of 1, 2-Epoxyethanes to 2 Hydroxyethyl Thiocyanates with Ammonium Thiocyanate.*Bull. Chemical Society Japan.* 7(6): 137-142.