Synthesis, Characterization and Antibacterial Activity of Schiff Base and its Metal (II) Complexes Derived From 3-Aminophenol and Benzaldehyde

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Abstract: **New Schiff base ligand was synthesis via a condensation of the ligands in methanol. The Schiff base were synthesize in 1:2 molar ratio reactions. The complexes have been characterized on the basis of FTIR, electronic spectra, melting points, solubility and molar conductance. The invitro antibacterial activity of the complexes were tested using four bacteria strain; gram negative (*Escherichia coli, salmonella typhi*) and gram positive (*staphylococcus pyogenes and staphylococcus aureus*). The complexes were formed in good yield and they have various shades of colors and sharp melting points. The IR results for the HL^1 synthesized schiff base revealed bands at 1621cm^{-1} which shifted to higher frequencies in the metal complexes 1622, 1626 and 1621 cm^{-1},. The band at 690, 666, 696 cm^{-1}and 593, 549,579 cm^{-1}(HL^1), were assigned to (M-N) and (M-O) bands in the spectra of the complexes supporting coordination of Schiff base/ligands to respective metals.The electronic spectral data of the complexes suggest an Octahedral and tetrahedral geometry for all the complexes. The molar conductivity indicates that the synthesized complexes are all non-electrolytes and soluble in protic solvent such as methanol and ethanol. The invitro antibacterial screening of schiff base and its metal complexes showed that they are potential antibacterial agents against the tested microorganisms**

Keywords: **Schiff base,3-aminophenol, benzaldehyde, metal complexes, antibacterial activity.**

I. INTRODUCTION

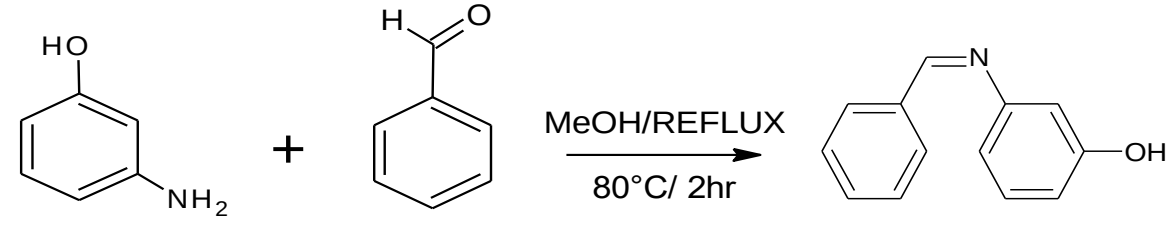
Schiff base was named after the German chemist Hugo Schiff who prepared these compounds from simple condensation between aldehydes and ketones with primary amines. Schiff bases have been known by different names such as imines, azomethine and anils which common for Schiff bases derived from aromatic amines, aldehydes and ketones. In general the Schiff bases which have been derived from aldehydes are called aldimine and from ketones as ketamine and the stability of the final product depend on the nature of aldehyde, ketone and amine (Yaseer 2017).The chemistry of metal complexes including ordinary complexes, chelates and mixed ligand complexes has been extensively studied till date for their bioinorganic relevance as well as a wide range of physicochemical properties (Mukhtar 2018)

II. MATERIALS AND METHOD

All chemicals, reagents and drug that were used in this research are of Analar grade. The metals used are; $NiCl_2.6H_2O$, $ZnCl_2$, $FeSO_4$, 3-aminophenol and Benzaldehyde are the ligands that were used in the research which were obtained from Department of Chemistry,Gombe State University, Nigeria. For the conduct of this research routine laboratory apparatus were used such as; Fourier Transform Spectroscopy (FT-IR), UV/Visible Spectrophotometer, stop watch, conductivity meter, melting point apparatus, Hot plate and magnetic stirrer, Oven, Weighing Balance, Auto clave, pH Meter, Petri Dishes, Pestle and Mortar, Water Bath

Synthesis of Schiff Base 3-aminophenol benzaldehyde HL^1

The Schiff base was synthesized by a slight modification of literature (Fugu*et,.al* 2013) . This is done by the condensation of methanolic solution of benzaldehyde (0.005mol, 0.53g) with 3-aminophenol (0.005mol, 0.55g) in 20ml of methanol (1:1 molar ratio). The resulting mixture was then refluxed for 1hr. the precipitate was separated by a suction filtration. It was recrystallized from ethanol and dried, and preserve in a desiccator for 24hr over $CaCl_2$.

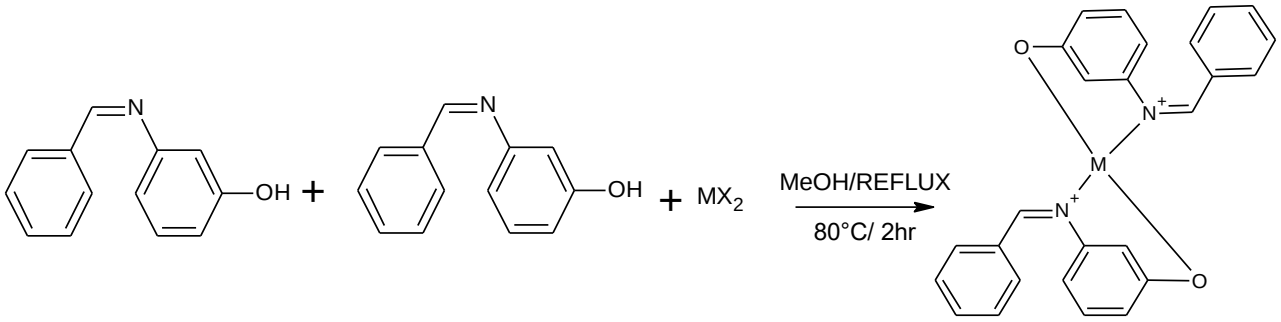


3- aminophenol Benzaldehyde

Scheme 1: synthesize Schiff base of 3- aminophenolbenzaldehyde

Synthesis of Schiff base and metal (II) complexes

The complexes were synthesized using 1:2 molar ratio (metal:ligand), by the condensation of a hot methanolic (40–50^0C) solutions of the HL^1 (1mmol, 0.44g) with aqueous methanolic Solution of (1mmol, 0.30gof $FeSO_4$, 1mmol, 0.44g of $NiCl_2.6H_2O$, 1mmol, 0.27g of $ZnCl_2$), which resulted in the immediate precipitation of metal derivatives. The products formed were filtered, Washed with ethanol, dried and preserved in desiccator over $CaCl_2$ (Singh *et al*., 2017)



Schiff base ligands

Scheme 2: synthesize Schiff base and metal (II) complex.

Where M= $FeSO_4$, $NiCl_2.6H_2O$, $ZnCl_2$.

Characterization of Schiff Base and its Metal (II) Complexes

Characterization involve simple fingerprint of compounds already known, or more extensive investigation designed to establish the formula and structure of a new compound (Cox, 2004). The Schiff base and its metal complexes was characterized by using different physico-chemical techniques like Melting point, U/Visible spectrophotometer, FTIR, Conductivity, Solubility test.

III. RESULTS AND DISCUSSION

Physical Characteristics And Analytical Data Of Ligands /Complexes

The physical properties of the Schiff base and the complexes for the complexes of $[Zn(3AMPB)_2Cl]$, $[Fe(3AMPB)_2]$, and $[NI(3AMPB)_2Cl]$ are presented in the table 1 below. The Schiff base and the complexes shows various shades of colors ranges from Yellow-brown, Yellow, Brown, and Red-brown for the Schiff base Zn, Fe, Ni complexes respectively (Ndahi 2012).. The color of the complexes are typical of transition metal complexes which are attributed to d-d transitions of electron between energy levels because of partially filled d orbital or charge transfer transitions (Nasiru 2018). The Schiff base and the metal complexes shows a sharp melting points ranges from (140-160), (243-246), (241-244), (241-245), the high melting point of the complexes indicates that the compounds are stable and not easily decomposed (Nasiru*et., al* 2018). The percentage yield of the compounds ranges from 70-95%. The molar conductance of the soluble Schiff base and the complexes in methanol show values (7.5-17.3 $\Omega^{-1}cm^{-2}mol^{-1}$) indicating that the compounds are all non-electrolyte in nature (Al-Noor 2014).

Solubility Test of the Complexes

Generally, the Schiff base and complexes (HL^1 and $[Zn(3AMPB)_2Cl]$, $[Fe(3AMPB)_2]$, and $[NI(3AMPB)_2Cl]$) were prepared by reacting the ligands and the metals ion in 1:1 and 1:2 molar ratio respectively. The solubility test for the complexes reveals that the complexes are soluble in some polar and nonpolar solvents as stated in the table2 below. The complexes of Zn, Fe, Ni are soluble in methanol (Hot and Cool), ethanol (hot and cool), acetone (hot and cool), diethyl ether(hot) and DMSO(hot). And sparingly soluble in chloroform (hot and cool), pet ether (hot), diethyl ether (cool), DMSO (cool) and they are not soluble in water (hot and cool), and pet. Ether (cool). Their solubility may be as results of the interaction between the hydrogen ion in the complexes and the oxygen atom in the solvent which results in the formation of hydrogen bond (Al-Noor 2014)

Electronic Spectral Data of Ligands and Complexes

The UV–Vis spectra of the free ligands and their metal (II) complexes (HL^1 and $[Zn(3AMPB)_2Cl]$, $[Fe(3AMPB)_2]$, and $[NI(3AMPB)_2Cl]$) are recorded in DMSO solution in the wavelength range (200–700) nm. The UV–Vis spectrum of the free Schiff base showed absorption band in the UV region at 240 nm, 41666cm^{-1}. It follows from the literature that the bands at 240nm for schiff base is related to the π–π* transitions of the 3-aminophenol and benzaldehyde which corresponds to the n–π* transition of the C= N and NH-CO groups (Mahmoud 2014). In the metal complexes, the π–π* transition bands are shifted to 254–286 nm which can be attributed to the binding of these coordination centers to the central metal ions. The absorption band at (254nm)(39370cm^{-1}), (286nm)(34965cm^{-1}) and (267 nm) (37453 cm^{-1}) were attributed to the (Eg-T_2g), (T_2g-Eg), (A_2g-T_2g), which reveals that the complex of $Zn(3AMPB)_2Cl$ shows an octahedral geometry, $Fe(3AMPB)_2$ show tetrahedral geometry and $Ni(3AMPB)_2Cl$ shows an octahedral geometry respectively (Mahmoud2014)

Major FTIR Spectral Data of Ligands and Complexes

The IR spectra analysis of the Schiff base ($HL^{1)}$ shows broad band at 3435cm^{-1} assigned to v(O-H) stretching vibration, The strong peak at 1621cm^{-1} is attributed to azomethine (C=N) group for the Schiff base and for the metal complexes of Zn, Fe, Ni shows a band at 1622, 1626, 1621 respectively. The peaks at that region indicate the interaction between the aldehyde and the amine group of the benzaldehyde and the 3aminophenol which ascertain the formation of the azomethine bond, and the shift of the peaks to higher frequency shows that the azomethine bond have participated in the coordination of the schiff base and the central metal ion. For the metal complexes the band at 3435 disappeared which indicate that it participates in coordination. A new two absorption band in the complexes at 690, 666, 696 cm^{-1} and 593, 549,579 cm^{-1} in the metal (II) chelates of Zn, Fe, Ni, respectively, show the coordination through nitrogen of azomethine and the oxygen of the v(CO) group of the schiff base which indicate the formation of M-N and M-O bonds confirming coordination of the ligand to the metal(II) ions as shown in Table 4 (Mukhtar 2018)

Antibacterial activity of the ligands/complexes of (3AMPB)

The antibacterial activity of the schiff base and the metal complexes were screened against two Gram- negative (*Escherichia coli and Salmonella typhi*) and two Gram-positivite (*Staphylococcus aureus and Streptococcus pyrogens*) bacterial strains to assess their potential antibacterial agents by disc diffusion method. The activity was determined by measuring the diameter of zone of inhibition and it was recorded in the table 5 below. It was observed that the metal complexes show high antibacterial activity than the free ligands; this is as a result of coordination between the central metal and the ligands. Because biologically inactive compounds become active and less biologically inactive become more active upon coordination as it is stated in previous literature (Ndahi, 2012). Hence chelation increases the antibacterial activity.

Table 1: Physical Characteristics And Analytical Data Of Ligands /Complexes

Complexes	MOL.WGT	COLOR	M.P ^{O}C	%YIELD	Molar conductivity $\Omega^{-1}cm^{-2}mol^{-1}$
HL^1	215.32	Yello-brown	140-160	90	7.5
$[Zn(HL^1)_2Cl]$	531.53	Yellow	243-246	95	15.2
$[Fe(HL^1)_2]$	486.64	Brown	241-244	87	17.3
$[Ni(HL^1)_2Cl]$	525.14	Red-brown	241-245	70	12.7
Where HL^1= Schiff base of 3Aminophenolbenzaldehyde, MOL.WGT= Molecular weight, M.P= melting points.					

Table 2: Solubility Test of the Complexes

Compound	Dist. H_2O		MeOH		EtOH		Acetone		Chloroform		P.Ether		D. Ether		DMSO	
	C	H	C	H	C	H	C	H	C	H	C	H	C	H	C	H
$Zn(HL^1)_2Cl$	NS	NS	S	S	S	S	S	S	SS	SS	NS	SS	SS	S	SS	S
$Fe(HL^1)_2$	NS	NS	S	S	S	S	S	S	SS	SS	SS	SS	NS	NS	S	S
$NI(HL^1)_2Cl$	NS	NS	S	S	S	S	S	S	S	S	NS	NS	SS	SS	S	S
Where C= Cool, H= hot, S= Soluble, SS= Sparingly Soluble, NS= Not Soluble, MeOH= Methanol, EtOH= Ethanol, Dist. H_2O= Distilled water, P.Ether= Petroleum ether, D.Ether= Diethyl Ether, DMSO= Dimethylsulphoixed, HL^1= Schiff base of 3Aminophenolbenzaldehyde																

Table 4: Major FTIR Spectral Data of Ligands and Complexes

Compound	V(C=C)	V(C=N)	V(O-H)	V(M-N)	V(M-O)
HL^1	1590	1621	3435	-	-
$Zn(HL^1)_2Cl$	1603	1622	-	690	593
$Fe(HL^1)_2$	1578	1626	-	669	549
$NI(HL^1)_2Cl$	1598	1621	-	699	579
Where HL^1= Schiff base of 3Aminophenolbenzaldehyde					

Table 3: Electronic Spectral Data of Ligands and Complexes

Compound	Absorption In nm	Absorption in cm^{-1}	Band assignment	Geometry
HL^1	240	41666	n-n*	
$[Zn(HL^1)_2Cl]$	254	39370	Eg-T_2g	Octahedral
$[Fe(HL^1)_2]$	286	34965	T_2g-Eg	Tetrahedral
$[NI(HL^1)_2Cl]$	267	37453	A_2g-T_2g	Octahedral
HL^1= Schiff base of 3Aminophenolbenzaldehyde,				

Table 5: Antibacterial Activity of the Ligands and Complexes				
Compuond	CONCENTRATION (mg/ml)			
	Gram-negative		Gram-positive	
	E. Coil	Salm	Staphy	Strept
HL^1	9.97	13.91	8.29	8.81
$[Zn(HL^1)_2Cl]$	20.10	7.12	12.45	9.90
$[Fe(HL^1)_2]$	15.2	14.25	14.76	14.24
$[NI(HL^1)_2Cl]$	13.14	17.26	7.10	13.12
Where *E. coli* = *Escherichia coli*, SALM= *Salmonella typhi*, STAPHY= *Staphylococcus auerues*, STREPT= *Streptococcus pyregens*, HL= Schiff base, 3AMPB= 3Aminophenolbenzaldehyde				

IV. CONCLUSION

The Schiff base and its respective metal complexes have shades varieties of colors, good %yields with sharp melting point. The electronic spectral data of the complexes suggest an Octahedral and tetrahedral geometry for all the complexes.The Results of the molar conductivity indicate that the synthesized complexes are non-electrolytes and 1:2 electrolytes as compared with the electrolytic value of $CaCl_2$.The invitro antibacterial screening of schiff base and its metal complexes showed that they are potential antibacterial agents against the tested microorganisms.

the formation of the schiff base and its respective metal(II) complexes are shown in scheme 1and 2 and the proposed data of the synthesize schiff base and the metal (II) complexes are shown in figure 1 and 2 below respectively

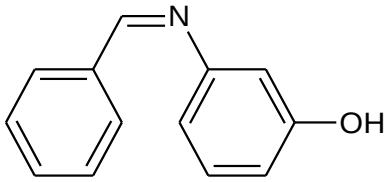
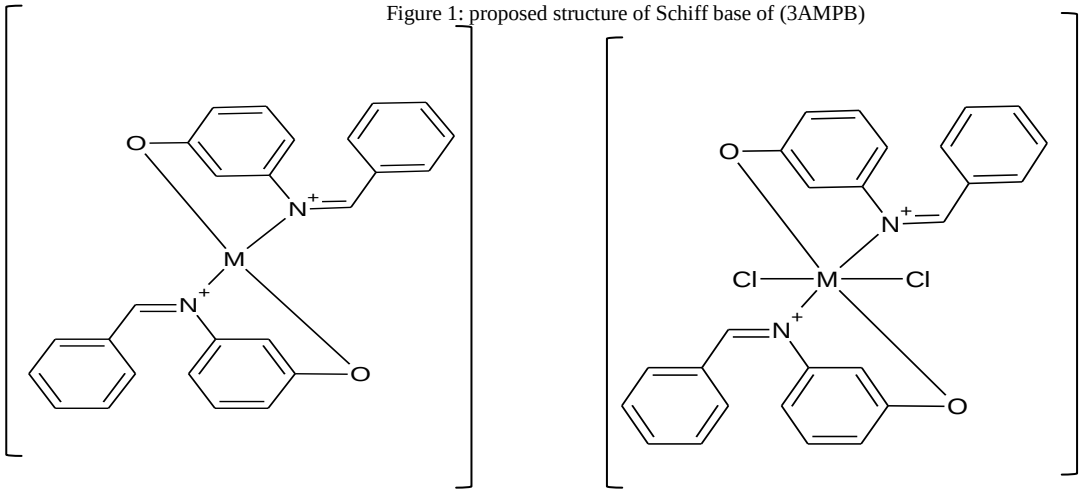


Figure 1: proposed structure of Schiff base of (3AMPB)



Where M= $FeSO_4$, Where M=$NiCl_2$,$ZnCl_2$,

Figure 2: proposed structure of Schiff base of (3AMPB) and its metal two complexes.

REFERENCES

[1] Fugu, M. B., Ndahi, N. P., Paul, B. B. and Mustapha, A. N., (2013). Synthesis, characterization, and antimicrobial studies of some vanillin Schiff base metal (II) complexes Journal of Chemical and Pharmaceutical Research, **5**(4):22-28

[2] Kiran Singh, Yogender Kumar, ParveshPuri, Chetan Sharma, Kamal RaiAneja (2017). Antimicrobial, spectral and thermal studies of divalent cobalt, nickel, copper and zinc complexes with triazole Schiff bases. Arabian Journal of Chemistry www.ksu.edu.sa

[3] Mukhtar,H. U. Sani, M. H. Shagal and J. I. Joseph (2018). Synthesis, Characterization and Antimicrobial Studies of Schiff base Derived from Salicyldehyde and 2,4-dinitrophenyl hydrazine and its Metal (II) Complexes. IOSR Journal of Applied Chemistry (IOSR-JAC)e-ISSN: 2278-5736.**11**(9):49-53

[4] Nasiru, P.Y Ndahi, N.P., Bako, L., Mohamad, L.M, Abdullahi,.Z. and Yusuf, M. (2018). Synthesis and partial characterization of two schiff base ligands with (2 and 4-nitroaniline) and their transition metal (II) (Co and Cu) complexes. Dutsejournal of pure and applied science, **1**(2):584-591.

[5] Ndahi, N.P. and Nasiru, Y.P. (2012). Complexes of Cobalt(ii), Nickel(ii) and Zinc(ii) with schiff bases derived from 4-anisaldehyde. International Journal of Science and Applied Research ,**3**(12): 278-283.

[6] TaghreedHashim Al-Noor, ManhelReemon Aziz, Ahmed.T.AL-Jeboori (2014). synthesis, characterization and antimicrobial activities of[Fe(ii), Co(ii), Ni(ii), Cu(ii) ,and Zn(ii)}mixed ligand complexes schiff base derived from ampicillin drug and 4(dimethylamino)benzaldehyde with nicotinamide. International Journal of Technical Research and Applications e-ISSN:2320-8163, www.ijtra.com **2**(4): 187-192

[7] Walaa H. Mahmoud, Gehad G. Mohamed, Maher M.I. El-Dessouky (2014). Synthesis, Characterization and in vitro Biological Activity of Mixed Transition Metal Complexes of Lornoxicam with 1,10phenanthrolineInt. J. Electrochem. Sci., (**9**) 1415 – 1438.

[8] Yaseer hussam sanbah (2017). Synthesis, characterization and biological evaluation of penicilin derivative complexes with some metal ions. Republic of irag ministry of higher education and scientific research al-Nahrain university college of science department of chemistry.