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Synthesis, Characterization and Antibacterial Activity of Mixed Ligands of Schiff Base and It's Metal(II) Complexes Derived from Ampicilin, 3-Aminophenol and Benzaldehyde

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Abstract: Schiff base ligands were synthesis via a condensation of the ligands in methanol in 1:1:1 molar ratio reactions. The transition metal(II) complexes were formed by the reaction of Co, Cu, Mn, Fe with the Schiff base and ampicillin and they were characterized via FTIR, electronic spectra, melting points, solubility and molar conductance. The *invitro* antibacterial activities of the complexes were tested using four bacterial strains; gramnegative; (*Escherichiacoli, salmonellatyphi*) and grampositive; (*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 spectrum of the HL (Table4) displays a new at (1651) cm^{-1} which is due to υ(HC=N) group of the azomethine stretching vibrations of the ligand, on complexation these band has been shifted to lower frequencies (1651), (1621), (1651), and (1627)cm^{-1} for complexes [Co(Ampi)(3AMPB)Cl], [Cu(Ampi)(3AMPB)], [Mn(Ampi)(3AMP)Cl] and [Fe(Ampi)(3AMPB)] respectively. The overlap band at (1688)cm^{-1} stretching vibration is due to υ(C=O) for β-Lactam group, these band has been shifted to lower frequency at (1370-1425)cm^{-1} for complexes showing that the coordination is through the Oxygen atom of β-Lactam group. The band sat (525), (526), (524) and (526)cm^{-1} were assigned to υ(M–O) for compounds of Co, Cu, Mn, Fe, indicating that to the carbocyclic oxygen, and oxygen of β-Lactam group of the ligand are involved in coordination with metalions. The bands at (659), (669), (660), and (698)cm^{-1} were assigned to υ(M–N) for compounds Co, Cu, Mn, Fe respectively, indicating that the nitrogen is involved in coordination with metal ions. The electronic spectral data of the complexes suggest an Octahedral and tetrahedral geometry for all the complexes. The molarconductivity 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: SchiffBase, 3-aminophenol, Benzaldehyde, Ampiciln, Metal Complexes, Antibacterial Activity

1. Introduction

Schiff bases and their complexes are versatile compounds synthesized from the condensation of an amino compound with carbonyl compounds and widely used for industrial purposes and also exhibit a broad range of biological activities including antifungal, antibacterial, antimalarial, antiproliferative, anti-inflammatory, antiviral, and antipyretic properties [1] Schiff base are the compound containing azomethine group (-HC=N-). The acid/base catalysis or heating is employed for the synthesis of Schiff bases as their reactions are mostly reversible [2].

The aim of the research is to synthesize, characterize and evaluate the antibacterial activity of the synthesized Schiff base and its metal (II) complexes of Fe(II), Co(II), Cu(II) and Mn(II) ions .

While the specific objectives of the study will be to:

i. Prepare Schiff base using a mixed ligands of 3-aminophenol and Benzaldehyde.
ii. Synthesize Schiff base metal complexes of Fe(II), Co(II), Cu(II) and Mn(II) ions.
iii. Synthesize Schiff base and ampicillin with its metal

complexes of Fe(II), Zn (II), Co(II), Cu(II) Ni(II) and Mn(II) ions.

iv. Determine the physico–chemical properties of Schiff base and its metal complexes using different analytical technique like melting point, FTIR, conductivity, solubility test and UV/Visible spectrophotometer.

v. Study the antibacterial activity of the Schiff base and it's metal(II) complexes.

2. 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

2.1. Synthesis of Schiff Base 3-aminophenol Benzaldehyde HL[1]

The Schiff base was synthesized by a slight modification of literature [3]. 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 $CaCl_2$.

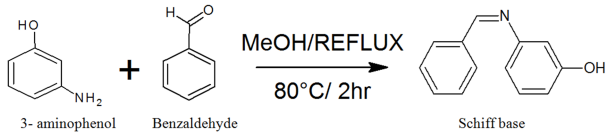


Figure 1. *SynthesizeSchiffbaseof3-aminophenolbenzaldehyde.*

Synthesis of Schiff base, ampicillinandmetal(II)complexes

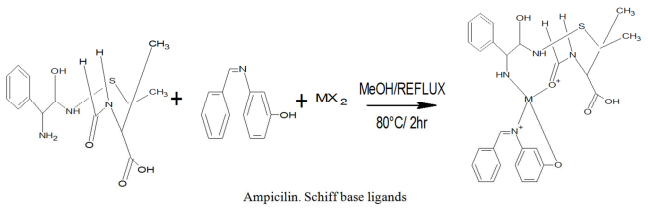


Figure 2. *Synthesize Schiff base and metal(II) complex.*

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

The complexes were synthesized using (1:1:1 molar ratio). A solution of the Schiff base (HL) (0.001mol, 0.43g) in 10ml of methanol and a solution of ampicillin (Ampi) (0.001mol, 0.35g) in 10ml methanol, were added to a stirred solution of (0.001mol, 0.16g of $CuSO_4.5H_20$, 0.001mol, 0.24gof $CoCl_2.6H_20$, 0.001mol, 0.15g of $FeSO_4$, 0.001mol, 0.20g of $MnCl_2.4H_20$)in 5ml of methanol. The resulting mixtures were heated under reflux for 3hr. then the mixture was filtered by suction and the precipitate was washed with excess of ethanol and dried at room temperature for 24hr in a desiccator over $CaCl_2$ [4].

2.2. 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 [5]. 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.

2.3. Antibacterial Activity

The four bacterial species were used two gram-negative (*Escherichia coli*, *Salmonella typhi)* and two gram-positive (*Staphylococcus aureus*, *Streptococcus pyrpgens*), were obtained from pharmaceutical microbiology Department of the Gombe state University. The antibacterial activity of the ligand and the complexes was determined by the disc diffusion technique. The compounds were screened *invitro*. A 1mg/ml solution in DMSO was used. Ceftriaxone and subactam were used as standard reference drug. The bacterium was maintained on nutrient agar and the agar media was incubated for different microorganism culture tests. After 24hrs of incubation at 37^0C for the bacteria, the diameters of zone of inhibition (mm) thus formed around each disc containing the test compound was measured [3].

3. Results and Discussion

3.1. Physical Characteristics and Analytical Data of Ligands/Complexes

In general, the complexes of [Co(Ampi)(3AMPB)Cl], [Cu(Ampi)(3AMPB)], [Mn(Ampi)(3AMP)Cl] and [Fe(Ampi)(3AMPB)] were obtained by heating under reflux of respective metal salts with HL and ampicillin in 1:1:1 molar ratio. The Schiff base and its metal(II) complexes shows various shades of colors ranging from Milk, Milk, Yellow-green, Milk, Grey for The HL and the complexes of Co, Cu, Mn Fe respectively. The HL and the complexes of Co, Cu, Mn, Fe obtained gave a sharp melting point indicating the isolation of fairly pure complexes (<300), (286-288), (283-284), (279-283), (285-287) ^{o}C of HL, Co, Cu, Mn, Fe respectively [6]. The Schiff base and its metal (II) complexes were prepared in good yield, ranging from 50-80% (7). The molar conductance of the complexes was determined in DMSO. It was found to be 4.1, 6.1, 3.1, 13.2 and $5.7\Omega^{-1}cm^{-2}mol^{-1}$ for the Co, Cu, Mn, Fe complexes respectively. These low values suggested their non-electrolytic nature [7, 8].

Table 1. *Physical Characteristic sand Analytical Data of Ligands/Complexes.*

Compounds	MOL. WGT	COLOR	M. P°C	%YIELD	Molarconductivity$\Omega^{-1}cm^{-2}mol^{-1}$
HL	458.393	MILK	<300	75	4.2
Co(Ampi)(3AMPB)Cl	552.823	MILK	286-288	80	6.1
Cu(Ampi)(3AMPB)	522.393	YELLOW-GREEN	283-284	78	3.1
Mn(Ampi)(3AMP)Cl	548.893	MILK	279-283	50	13.2
Fe(Ampi)(3AMPB)	514.393	GREY	285-287	65	5.7

WhereHL[1]=Schiffbaseof3Aminophenolbenzaldehydeandampicillinligands, MOL. WGT=Molecularweight, M. P=meltingpoints.

3.2. Solubility Test of the Complexes

The solubility of the complexes of [Co(Ampi)(3AMPB)Cl], [Cu(Ampi)(3AMPB)], [Mn(Ampi)(3AMP)Cl] and [Fe(Ampi)(3AMPB)] was studied in various solvent in both hot and cool process. The complexes are not soluble in water, pet. ether and diethyl ether (cool and hot), it is sparingly soluble in chloroform. But all the complexes are soluble in MeOH(cool and hot), EtOH (hot) and DMSO(cool and hot). Their solubility is 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 (4) and [9].

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
Co(Ampi)(3AMPB)	NS	NS	S	S	SS	S	S	S	S	S	NS	NS	NS	NS	S	S
Cu(Ampi)(3AMPB)	NS	NS	SS	S	SS	S	SS	S	SS	SS	SS	SS	SS	SS	S	S
Mn(Ampi)(3AMPB)	NS	NS	SS	SS	SS	S	SS	S	NS	NS	NS	NS	NS	NS	SS	S
Fe(Ampi)(3AMPB)	NS	NS	SS	S	SS	SS	SS	S	NS	NS	NS	NS	NS	NS	SS	S

HL=Schiff base of 3Aminophenol benzaldehyde and ampicilin ligands, Where C=Cool, H=hot, S=Soluble, SS=Sparingly Soluble, NS=Not Soluble, MeOH=Methanol, EtOH=Ethanol, Dist.H_2O=Distilledwater, P. Ether=Petroleumether, D. Ether=DiethylEther, DMSO=Dimethylsulphoixed, 3AMPB=Schiffbase of 3Aminophenolbenzaldehyde, Ampi=Ampiciline

3.3. Electronic Spectral Data of Ligands and Complexes

The electronic spectrum of the ligandHL was been measured in DMSO solution between 200-1100 nm at room temperature. In the spectrum of the Schiff base ligand HL, the absorption band observed at (212 nm) (47169 cm^{-1}, which assigned to (π- π transition. The UV-Vis spectrum for the complexes ([Co(Ampi)(3AMPB)Cl], [Cu(Ampi)(3AMPB)], [Mn(Ampi)(3AMP)Cl] and [Fe(Ampi)(3AMPB)]) shows a band at (335nm) (29850 cm^{-1}), (267nm) (37453cm^{-1}), (280nm) (35714cm^{-1}) and (323nm) (30959cm^{-1}) for the complexes of [Co(Ampi)(3AMPB)Cl], [Cu(Ampi)(3AMPB)], [Mn(Ampi)(3AMP)Cl] and [Fe(Ampi)(3AMPB)] which show (T_1g-A_2g), (T_2g-Eg), (A_2g-T_2g) and (T_2g-Eg) transition. The Transitions, similar to those found for distorted octahedral complexes [4, 10, 11].

Table 3. *Electronic Spectral Data of Ligand sand Complexes.*

Compound	Absorptioninnm	Absorptionincm^{-1}	Bandassignment	Geometry
HL	212	47169	n-n*	
Co(Ampi)(3AMPB)Cl	335	29850	T_1g-A_2g	Octahedral
Cu(Ampi)(3AMPB)	267	37453	T_2g-Eg,	Tetrahedral
Mn(Ampi)(3AMP)Cl	280	35714	A_2g-T_2g	Octahedral
Fe(Ampi)(3AMPB)	323	30959	T_2g-Eg	Tetrahedral

* = Transition state

3.4. Major FTIR Spectral Data of Ligands and Complexes

The IR spectrum of the HL (Table 4) displays a new at (1651) cm^{-1}which is due to υ (HC=N-) group of the azomethine stretching vibrations of the ligand, on complexation these band has been shifted to lower frequencies (1651), (1621), (1651), and (1627) cm^{-1} for complexes [Co(Ampi) (3AMPB)Cl], [Cu(Ampi) (3AMPB)], [Mn(Ampi) (3AMP)Cl] and [Fe(Ampi) (3AMPB)] respectively. The overlap band at (1688)cm^{-1} stretching vibration is due to υ (C=O) for β-Lactam group, these band has been shifted to lower frequency at(1370-1425)cm^{-1} for complexes showing that the coordination is through the Oxygen atom of β-Lactam group. The bands at (525), (526), (524) and (526)cm^{-1} were assigned to υ(M–O) for compounds of Co, Cu, Mn, Fe, indicating that to the carbocyclic oxygen, and oxygen of β-Lactam group of the ligand are involved in coordination withmetal ions. The bands at (659), (669), (660), and (698) cm-1 were assigned to υ(M–N) for compounds Co, Cu, Mn, Fe respectively, indicating that the nitrogen is involved in coordination with metal ions [4, 12, 13].

Table 4. *Major FTIR Spectral Data of Ligands and Complexes.*

Compound	v(N-H)P	v(N-H)S	V(C-C)	V(C=C)	V(C=N)	V(C-N)	V(C-S)	V(C-H)	V(M-N)	V(M-O)
HL	3541	3270	2384	1658	1651	1450			-	-
Co(Ampi)(3AMPB)	3572	3491	1135	1370	1651	1211	576	2384	659	525
Cu(Ampi)(3AMPB)	3541	3401	1119	1370	1621	1493	576	-	669	526
Mn(Ampi)(3AMP)	3572	3492	1210	1370	1651	1210	576	2380	660	524
Fe(Ampi)(3AMPB)	3485	3319	1134	1425	1627	1450	576	-	698	526

Ampi=ampicillin, 3AMPB=3Aminophenolbenzaldehyde.

3.4. Antibacterial Activity of the Ligands/Complexes of (3AMPB)

The antibacterial activity of the Metal complexes of Schiff base derived from (HL) and Co, Cu, Mn Fe showed a good antibacterial activity against Gram-negative (*Escherichia coli and Salmonella typh*i,). The Co, Cu, Mn Fe complexes caused strong inhibition for *E. coli* and *Salmonella typhi*. The importance of these lies in the fact that, these complexes could be applied fairly in the treatment of some common diseases caused by *E. coli* and *Salmollatyphi*. However, Co, Cu, Mn Fe complexes were specialized in inhibiting Gram-positive bacterial strains (*Staphylococcus auerus* and*Streptococcus pyrogens*). The importance of this unique property of the investigated Schiff base complexes lies in the fact that, it could be applied safely in the treatment of infections caused by any of these particular strain [1, 14, 15].

Table 5. *Antibacterial Activity of the Ligands and Complexes.*

Compounds	CONCENTRATION(mg/ml)			
	Gram-negative		Gram-positive	
	E.Coil(50/100)	Salm(50/100)	Staphy(50/100)	Strept(50/100)
HL	11/15	8/10	15/16	14/20
Co(Ampi)(3AMPB)	18/20	6/5	17/18	11/12
Cu(Ampi)(3AMPB)	22/25	7/9	21/20	11/15
Mn(Ampi)(3AMP)	19/20	13/12	15/19	14/17
Fe(Ampi)(3AMPB)	15/17	12/14	11/17	8/10

Where; E.coli=Escherichiacoli, SALM=Salmonellatyphi, STAPHY=Staphylococcusauerues, STREPT=Streptococcuspyregens, HL=Schiffbase, 3AMPB=3 aminophenolbenzaldehyde, Ampi=Ampicilin.

4. Conclusion

The complexes are multi-colored, colors, they have good % yields with sharp melting point. The electronic spectral data of the complexes suggest an Octahedral and tetrahedral geometry for 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 Figures 1and 2 and the proposed Structures based on the data of the synthesize schiff base and the metal (II) complexes are shown in figures 3 and 4 below respectively

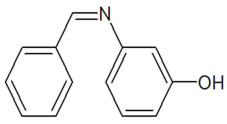
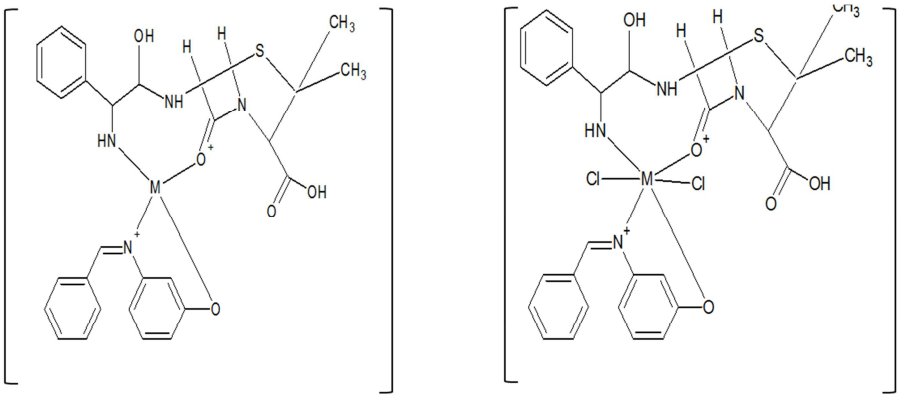


Figure3. *ProposedstructureofSchiffbaseof(3AMPB).*



Where M=$NiCl_2$,$ZnCl_2$,

Figure 4. *Proposed structure of metal (II) complexes.*

References

[1] Ahmed, M., Abu-Dief, I. and Mohamed, M. A. (2015), A Review on Versatile Applications of Transition Metal Complexes Incorporating Schiff Bases. *SUEF University Journal of Basic and Applied Sciences*, (4): 119-133.

[2] Neelima M., Kavitan P and Dinesh K (2013). An overview of biological aspects of Schiff base metal complexes. *International Journal of Advancements in Research & Technology* 2 (8), 52.

[3] 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.

[4] Taghreed, H., Al-Noor, M. R., and Aziz, A. T (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*-2 (4): 187-192.

[5] Cox, P. A., (2004). *Inorganic chemistry laboratory* new college oxford UK scientific publisher 2 (2): 111-230.

[6] Emad Y., Ahmed M., Khulood A., Nadia S., Jumat S., Bashar A (2013). Metal complexes of Schiff base: Preparation, characterization and antibacterial activity. *Arabian Journal of Chemistry* 1 (1) 56-70.

[7] Ahmed, A. A., Umar, H. A, Yunusa, Y. and Adegbemiga, B (2017), Synthesis and Characterization of Cobalt (II) and Nickel (II) Complexes with a Schiff Base Derived from 2-aminophenol and 4-(n,n-dimethylamino)benzaldehyde. *International Journal of Science and Applied Research (IJSAR)* 2 (3) 587-593.

[8] Rehman, W., Baloch, M., and Badshah, A (2008). Synthesis, Spectral Characterization and Bio-Analysis of some Organotin(IV) Complexes. *European Journal of Medicinal Chemistry, 43*: 2380-2385.

[9] Mukhtar, H. U., Sani, M. H., Shagal L and Joseph, J. I (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)* 11 (9):49-53.

[10] Oluwatoosin B., Agbaje, A., Sherifat, M. W., Osowole, A. A (2014). Synthesis, spectroscopic characterisation and antimicrobial activities of some mixed drug metal(II) complexes of Sulfamethoxazole and Paracetamol. *Academic editor JC* 2 (1) 140-143.

[11] Lever A. B. P., (1984)" Inorganic electronic spectroscopy". Elsevier, New york.

[12] Dhivyapriya, D., Akila, E., Usharani M and. Rajavel R (2012). Synthesis, spectral and biological activity of mixed ligand schiff base complexes derived from salicylaldehyde, *Journal of international journal of pharmacy & technology*, (IJPT) 4 (1) 11-12.

[13] Fayad, N. K., Taghreed H. A and Ghanim F. H (2012). Synthesis, characterization and antibacterial activities of Manganese, Cobalt, Iron, Nickel, Zinc, and Cadmium Mixed ligand complexes containing amino acid (L-Valine) and saccharin. *Journal of advances in physics theories and application*. 9 pp12.

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

[15] 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. *Dutse journal of pure and applied science,* 1 (2):584-591.