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Synthesis, Characterization and Anti-Bacterial Activity of Schiff Base and its Mixed Ligand Complexes of Cr (II) and Co (II) containing Vanillin and 2-Aminophenol

Ayuba Isiyaku[1*], Ibrahim Tajo Siraj[2], Aishatu Umar Maigari[3] and Buhari Magaji[4]

[1] Chemistry Unit,
Department of Science Laboratory Technology,
Federal Polytechnic Kaltungo, Gombe, Nigeria

[2]Department of Chemistry,
Bayero University Kano,
Kano, Nigeria

[3]Department of Chemistry,
Federal University Kashere,
Gombe, Nigeria

[4]Department of Chemistry,
Gombe State University,
Gombe, Nigeria

E-mail: isayuba3283@gmail.com

Abstract

Schiff Base are organic ligand that contained azomethine linkage (-HC=N-) which shows biological importance. Schiff Base from vanillin and 2-aminophenol was synthesized in 1:1 mole ratio. The complexes of Cr (II) and Co (II) from Schiff Base in 1:2 mole ratio metal-ligand (M-L) and the mixed ligand complexes from Schiff Base and 2-aminophenol in 1:1:1 mole ratio ligand-metal-ligand(L-M-L) were synthesized and characterized based on solubility, melting point, conductivity, Fourier transform infrared spectroscopy (FTIR)and ultraviolet(UV). The solubility result shows that, dimethyl sulfoxide(DMSO) dissolved all the complexes. The results obtained from melting point, conductivity indicated purity and non-electrolytic of the complexes respectively. In metal complexes, the infrared data showed the ligands is coordinated to the metal ion through azomethine nitrogen, oxygen in methoxyl group and oxygen in phenolic group. In mixed ligand complexes, the infrared data revealed the ligands is coordinated to the metal ion through azomethine nitrogen, oxygen in methoxyl group, oxygen in phenolic group and nitrogen in amino group. The results showed a six coordinate octahedral geometry for these complexes. The ligands and the metal complexes were examined for their antibacterial activity using agar well diffusion method against Escherichia coli, Staphylococcus aureus, streptococcus pyrogens , Klebsiella pneumonia (gram - bacteria), and Bacillus sutilis, Staphylococcus aureus (gram + bacteria). In comparing the results, the complex of $Co(HL^1)_2$ has greater zone of inhibition against the tested organism than the free ligands as antibacterial agent.

Keywords: Schiff base, Cr (II) Complex, Co (II) Complex, Antibacterial activity, Infrared

**Author for Correspondence*

INTRODUCTION

Schiff Base are organic compound generated as a product of condensation involving primary amine and either aldehyde or ketone with the elimination of water. The Schiff Base formed by condensation reaction is influence by steric, pH of the solution, and amine and electronic effect of the carbonyl compound (Felix, 2016). Schiff Base was first reported by German chemist Novel Prize winner in 1864 (Hugo Schiff) (Wakil *et al.,* 2017). The stability of the Schiff Base depends on the nature of aldehyde. The aliphatic aldehyde of Schiff Base are relatively unstable which can easily polymerized, but aromatic aldehyde having efficient conjugation are more stable (Esther *et al.,* 2019). Schiff Base formation generally takes place under base or acids with heat or catalysis. They contained carbon-nitrogen double bond which are called azomethine group (-HC=N-). The occurrence of lone pair of electron in sp^2 hybridized orbital of nitrogen atom of the azomethine is of significant biological and chemical importance (Sunil, 2014). Schiff Bases play important role in coordination chemistry as they simply form stable complexes with most transition metal ions (Mohammed & Mosab, 2020). Organic bidentate ligands contain donor atom like nitrogen, oxygen and carbonyl group, therefore their interaction with metal ion give complex of different geometries and are capable to show biological activity (Tasmina *et al.,* 2016).

Complex formation between transition metal and the Schiff Base ligands results in foundation of coordinate complexes (Shazia *et al.,* 2010). Rania *et al*. (2019), reported the Co and Cd (II), La and Gd (III) complexes of Benzoin and 2-Amino thiopenol and found the Cd (II) complex was safe to be used as anticancer agent. It's also been reported that, salicyldehyde ethylenediamine Schiff Base in coordination with Cr (III) ion used as a new model of glucose tolerance factor and was shown to reduce the symptoms of diabetes (Pedro *et al.,* 2006). Silver (I) sulphaxin is a complex that used to treat burns to prevent them from bacterial infection (Shazia *et al.,* 2010). Ndahi *et al*. (2012) reported the Ni, Co and Zn(II) complexes from benzenecarboxaldehyde with 2-Aminophenol and 4-bromoaniline have potency against bacterial strain including *Staphylococcus aureus, Pseudomonas aeruginosa, Escherichia coli, Salmonella typhi* and *Bacillus sutili*. Some other researchers reported the synthesis of vanillin-2-aminophenol Schiff Base metal complexes of Co, Mn, Cu, Zn and Ni(II) (Fugu *et al.,* 2013; Padma *et al.,* 2016).However, there are no reports on the synthesis of mixed 2-aminophenol and vanillin-2-aminophenol metal complexes of Schiff Base. The aim of the research was synthesis and investigatesanti-bacterial activity of mixed 2-aminophenol and vanillin-2-aminophenol Schiff Base metal complexes of Cr and Co (II).

MATERIALS AND METHODS

All reagents used were of analytical grade. The used of metal salt were inform of chloride. All the apparatus were washed and rinsed with distilled water. The melting points were taken on electrical thermal apparatus. The infrared (IR) results were obtained on fourier transform infrared spectroscopy (FTIR) in the range of 450-4000 cm^{-1}. The electronic spectra were recorded on ultraviolet (UV) visible spectrophotometer at a wavelength ranging from 200-800 nm using dimethyl sulfoxide (DMSO) solvent. The Measurement of conductivity was carried out in dimethyl sulfoxide (DMSO) solvent at a concentration of 10^{-2} Molar using digital conductivity meter. The antibacterial study were determined against *Escherichia coli*, *Streptococcus pyrogens*, *Klebsella pneumonia* (Gram -) and *Bacillus subtilis, Staphylococcus aureus* (Gram +) using ager well diffusion method.

Synthesis of Schiff Base ligand (HL^1)

Schiff Base was synthesized by slight modification of the method described by (Raman *et al*., 2004; Edward *et al.,* 2006; Ndahi *et al.,* 2012). This was done by the aldehyde condensation with primary amine in1:1 molar ratio. The reaction was achieved by dissolving 1.5 g (0.01 mol) of vanillin and 1.1g (0.01 mol) 2-aminophenol in 30 ml ethanol while heating under refluxed for three hours. The products form were filtered, and washed in ethanol then dried in a desiccator containing calcium chloride.

Synthesis of metal complexes derived from Schiff Base

Metals (II) complexes from Schiff base has been prepared by 1:2 molar ratio of metal-ligand. 1.1 g (0.02 mol) of Schiff base dissolved 20 mL ethanol; this was added in 20 mL hot ethanolic solution of 0.48 g (0.01 mol) $CrCl_2$ and $CoCl_2.6H_2O$. The mixture was heated at 78^{o}C while stirring under refluxed for three hours. The product was filtered and washed in distilled water then ethanol, dried in desiccator over calcium chloride for 1 day.

Synthesis of metal complexes derived from mixed ligand

The two ligands HL^1(vanillin-2-aminophenol Schiff Base) andHL^2 (2-aminophenol)were complexes in 1:1:1(Ligand-Metal-Ligand) ratio. This was done by adding 10 mL ethanolic solution of 0.55 g (0.01mol) 2-aminophenol and 10 mL ethanolic solution of 0.55 g (0.01 mol) Schiff Base in 20 mL ethanolic solution of 0.48 g (0.01mol) of $CrCl_2$ and $CoCl_2.6H_2O$. The reaction was carried out under refluxed while heating and stirring at 80^{o}C for three hours; the product formed was filtered and washed in distilled water then dried in desiccator containing calcium chloride over twenty four hours.

Antibacterial activity

The antibacterial studies of Schiff Base and its mixed ligand metal complexes were done using disc diffusion method as reported elsewhere (Ochie, 2000). The method was employed due to ability to test enormous number of microorganism and ease to interpret results. Itwas carried out against *Escherichia coli*, *Streptococcus pyrogens*, *Klebsella pneumonia* (Gram -) and *Bacillus subtilis, Staphylococcus aureus*, (Gram +). The samples were collected with the help of sterilized wire loop, transferred in to McCartney bottle of saline water for replication, then introduced in to inoculated agarplate, disc of 6 mm in diameter contained the test compound were positioned on the agar surface, the petri dishes ware incubated using an autoclave at 37 ^{o}C for twenty four hours. The antibacterial agent diffuses in to the agar and hinders germination and development of the bacterial species. The activity was determined and recorded by quantify the zone of inhibition in diameter.

RESULTS AND DISCUSSION

Results

The results of Schiff Base and its mixed ligand complexes of Cr (II) and Co (II), are presented in Tables 1 - 4.

Table 1. Physical characteristic of ligands and the metal complexes

Compounds	M F (gmol^{-1})	Color	Yield (%)	Conductivity($\Omega^{-1}cm^{-1}mol^{-1}$)	M.P (^{o}C)
HL^1	$C_{14}H_{13}NO_3$	Black	58	2×10^{-3}	365
$Cr(HL^1)_2$	$Cr(C_{14}H_{13}NO_3)$	Blue	35.7	8×10^{-3}	185
$Cr(HL^1)(HL^2)$	$Cr(C_{20}H_{20}N_2ON_4)$	Blue	57	25×10^{-2}	164
$Co(HL^1)_2$	$Co(C_{14}H_{13}NO_3)$	Brown	56.8	4×10^{-3}	190
$Co(HL^1)(HL^2)$	$Co(C_{20}H_{20}N_2ON_4)$	Brown	52	12×10^{-2}	150

Where M F= molecular formular, M P= melting points

Table 2. Solubility of Schiff Base and its mixed ligand metal complexes

Sample	Distilled H_2O	Chloroform	Acetone	Methanol	Ethanol	DMSO	DMF
HL^1	NS	SS	S	S	S	S	S
$Cr(HL^1)_2$	NS	SS	S	S	S	S	S
$Cr(HL^1)(HL^2)$	NS	SS	SS	S	S	S	S
$Co(HL^1)_2$	NS	SS	S	S	S	S	S
$Co(HL^1)(HL^2)$	NS	SS	S	S	S	S	S

Where NS= not soluble, SS= slightly soluble, S= soluble DMSO= dimethylsulphuroxide, DMF= dimethylformamide

Table 3. Infrared data of Schiff Base and its mixed ligand metal complexes

Compounds	*v*OH	*v*OCH_3	*v*C=N	*v*C-O	*v*C-N	*v*M-N	*v*M-O
HL^1	3376	2827	1581	1353	1498	-	-
$Cr(HL^1)_2$	3380	3229	1556	1361	1511	642	534
$Cr(HL^1)(HL^2)$	3377	-	1574	1367	1512	751	557
$Co(HL^1)_2$	3376	3227	1573	1371	1510	661	535
$Co(HL^1)(HL^2)$	3363	-	1585	1372	1511	747	584

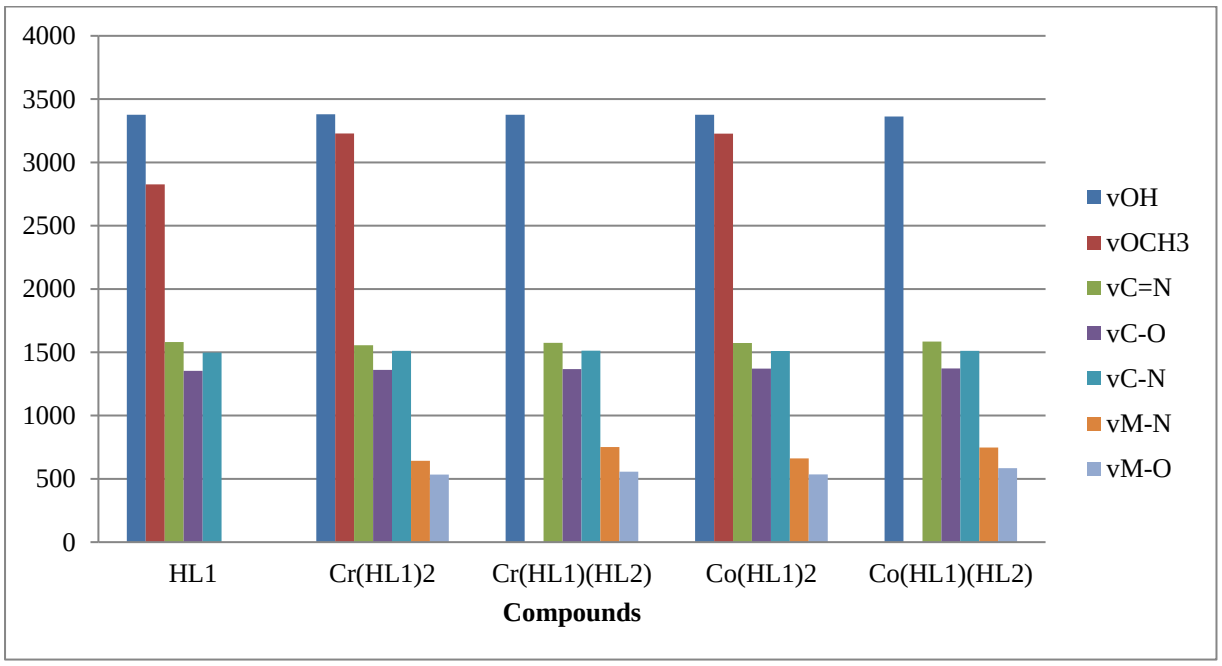

Fig. 1: Infrared data of Schiff Base and its mixed ligand metal complexes

Table 4: Antibacterial activity of Schiff Base and its mixed ligand metal complexes

Compounds	zone of inhibition (mm)				
	E. coli	*S. pyrogene*	*K. pneumonia*	*B. subtilis*	*S. aureus*
HL^1	14	07	12	07	05
$Cr(HL^1)_2$	14	00	07	00	00
$Cr(HL^1)(HL^2)$	15	08	09	10	07
$Co(HL^1)_2$	18	00	20	19	21
$Co(HL^1)(HL^2)$	19	00	08	00	00

Where 00: absence of inhibition, < 9: weak, 9-16: moderate, > 16: significant, size of disc: 6mm

Discussion

The Schiff Base (HL^1) from vanillin and 2-aminophenol was synthesized and produced as black color with the percentage yield of 58 and melting point of 365^{o}C. The interaction of Schiff Base with metal (II) in 2:1 (Metal-Ligand) and mixed ligand derived from Schiff Base and 2-aminophenol on interaction with metal (II) in 1:1:1(Ligand-Metal-Ligand) produced the complexes with a moderate yield between 35.7 - 57% and melting point ranging from 150 - 190^{o}C. The complexes shows range of colors of brown and blue which is typical of transition metal due to d-d electron transition (Lee, 1996). The molar conductivity measurement of the compounds revealed the lower values between 2×10^{-3} - 25×10^{-2} which means the complexes are non-electrolyte (Shelke*et al*., 2011). The solubility of the complexes was examined in dionized water, chloroform, acetone, methanol, ethanol; dimethyl sulfoxide (DMSO) and dimethyl formamide (DMF) are presented in Table 2. The compounds were not soluble in water, slightly soluble in chloroform and soluble in ethanol, dimethyl formamide, methanol and dimethyl sulfoxide. and Cr (HL^1)(HL^2) complex was slightly soluble in Acetone. In general, organic ligands with transition metal complexes are moderately non polar, as a consequence they are not soluble polar solvent like water but soluble in organic solvents as reported elsewhere (Nasiru *et al.*, 2018).

The IR spectroscopy was employed to study the band position of the ligand which may change due to bond formation as a result of complexation with metal are presented (Table 3) and Figure 1. The IR spectrum of Schiff Base appeared at 3376 and 2827 cm^{-1} and was consigned to νOH and νOCH_3 stretching due to the present of phenolic and methoxy group. The band at 1581cm^{-1} was consigned to νC=N stretching, this was shifted to lower frequency at1556 cm^{-1} - 1573 cm^{-1} in the complexes derived from Schiff Base in 2:1 (Ligand-Metal) ratio. This may be due to coordination to the metal in azomethine nitrogen, this values is in close conformity with the work reported by (Fugu *et al.,* 2013). The emergence of new bands at 642 - 661 cm^{-1} and 534 - 535 cm^{-1} was attributed to νM-N and νM-O vibration and were absence in the free ligands, this supported the association of nitrogen and oxygen during complexation and it is comparable to the result reported by (Monika *et al.,* 2010). In IR spectrum of mixed ligand metal complexes derived from Schiff Base and 2-aminophenol in 1:1:1 (LML) ratio, the azomethine bands were shifted to higher frequency at 1574 – 1585cm^{-1}, an increasing bands to higher frequency is due to increase of bond order on coordination (Gupta *et al.,* 2012). The bands at 747 - 751 cm^{-1} and 557 - 584 cm^{-1} were assigned to νM-N and νM-O respectively. This indicates the participation of metal during bond formation. This is in conformity with the work reported by (Jeasmin *et al.,* 2017). Thus, six coordinates octahedral geometry of cobalt and chromium complexes were obtained.

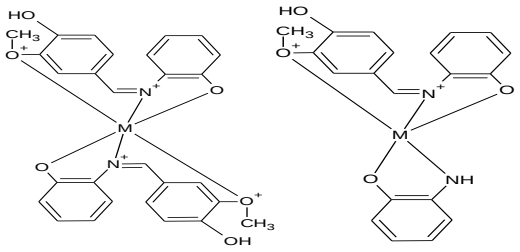

Fig. 2: Propose metal complexes structure

Where M = Cr and Co(II)

Table 4 presents the antibacterial activity of the metal complexes and ligand were tested against *E. coli*, *S. pyrogens*, *K. pneumonia* (Gram -) and *S. aureus*, *B. subtilis* (Gram +) in dimethyl sulfoxide solvent using disc diffusion method. The measure of zone of inhibition shows biological activity of all compounds against *Escherichia coli*, but no inhibitory action against *S. pyrogens, B. subtilis and S. aureus.* Weak inhibition was observed in the Schiff Base

against *K. pneumonia* and more significance inhibition was determined in $Co(HL^1)_2$ complex against the tested bacterial strain with the exception of *S. pyrogens*. Comparatively, the activities of metal complex is higher than the Schiff Base, this may be due to interaction with metal ion. The result agreed with the work reported by (Ndahi *et al*., 2012). This type of interaction increases the activity towards inhibition of bacteria (Sunil *et al*., 2014).

CONCLUSION

The Schiff Base, metal complexes derived from Schiff Base in 2:1 (Ligand-Metal) and the mixed ligand metal complexes derived from Schiff Base and 2-aminophenol in 1:1:1 (Ligand-Metal-Ligand) have been synthesized. The compounds are stable at room temperature and their solubility in dimethyl sulfoxide due to organic ligand in the complexes. The lower values of conductivity result revealed the nature of non electrolytic of the complexes. In metal complexes, the infrared data showed the ligands is coordinated to the metal ion through azomethine nitrogen, oxygen in methoxy group and oxygen in phenolic group, while in mixed ligand complexes, the infrared data showed the ligands is coordinated to the metal ion through azomethine nitrogen, oxygen in methoxy group, oxygen in phenolic group and nitrogen in amino group. A six coordinate octahedral geometry for these complexes have been obtained. The antibacterial screening against bacterial strain shows greater zone of inhibition in the metal complex than in the free ligand.

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