

Synthesis and Characterization of Three New Schiff Base Complexes of Metals (II) Derived from a Mix of Drugs Sulfamethaxole with 2-aminopyridine and Benzaldehyde

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ABSTRACT

In this research, 2-Aminopyridine and Benzaldehyde was synthesized to give a Schiff base (L^1). The Transition Metal Complexes were prepared by the reaction of the Schiff Base and Metal (II) Salt in a 2:1 molar ratio $\{M: 2(L^1)\}$. While the mixed ligand Complexes were synthesized by reaction of Schiff base with Metals (II) salt and Sulfamethoxazole Drug in a ratio of 1:1:1 $\{M: (L^1): (L^2)\}$. Modern and purity of these complexes were determined using melting point, texture, color, solubility and molar conductivity. The Schiff Base, the Metal (II) complexes and the mixed ligand Complexes were characterized using FT-IR and VU-Visible spectroscopic. It was found that in the Metal (II) Complexes bound to the Metal through Nitrogen (N) and azomethine. While in the mixed ligand Complexes both the Schiff base and Sulfamethaxazole drug are bound to the Metal through Nitrogen (N) and Oxygen (O) or azomethine.

Keywords: Synthesis, Schiff base, Complexes, Sulfamethaxole, Benzaldehyde.

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I. INTRODUCTION

Transition elements are metallic elements that have incomplete d or f shells in the neutral or cationic states. They are called also transition metals and make up 56 of the 103 elements; these include the three series of elements that are positioned between the first two groups and the last six groups in the periodic table [1]. First series Transition metals represent the d block element which includes group's IIA - IIIA on the periodic table. Their d shells are incompletely filled. This property of transition metals resulted in the

foundation of coordination complexes [2]. The d -block transition metals have s , p , and d orbitals. And those with n electrons in the d orbitals are termed ions with a d^n configuration. For example, Ti^{3+} is a d^1 ion, and Co^{3+} a d^6 ion. The number of electrons occupying the orbitals split by the ligand field (cf) is denoted by a superscript on the orbital symbol. Molecular compounds which consist of d -block transition metals and ligands are referred to as complexes or coordination compounds. Metal complexes are also known as coordination compounds, which include all metal compounds. Metal complex is a structure

consisting of a central atom or ion (metal) bonded with anions (ligands). Compounds that contain a coordination complex are called coordination compounds [1]. Schiff bases sustain azomethine or imine ($-C=N$) unit. This is the primary condensation of amines with carbonyl compounds and have been reported by Hugo Schiff [3]. Schiff bases have a wide variety of applications in different areas such as biological chemistry, organic and inorganic chemistry. The medicinal uses and applications Schiff bases and their metal complexes are of increasing clinical and commercial importance [3]. Schiff bases have played a significant role in the development of coordination chemistry and have been implicated as an important point in the development of inorganic, biochemistry and optical materials [3]. In the search for novel therapy against resistant organism, the modification of existing drug by combination to a metal center has gained attention in recent years [3]. So, we synthesized

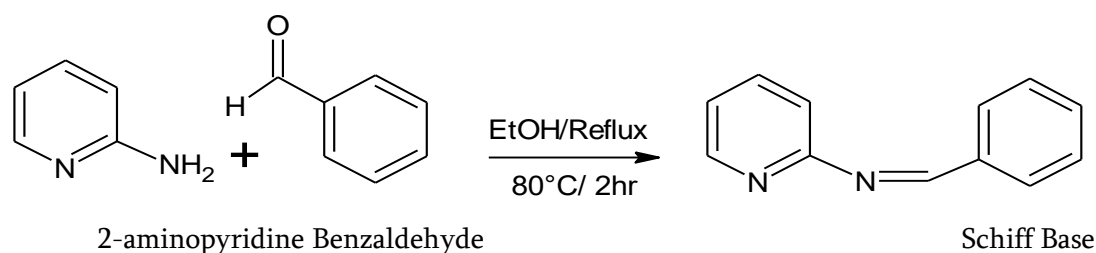
and characterized Metal (II) complexes derived from a mix of drugs of Sulfamethaxazole with 2-aminopyridine and Benzaldehyde

II. MATERIALS AND METHOD

All chemical and solvent used were of analytical grade and were used as supplied. The metal (II) salts were used in the form of their chloride and sulphate [4].

Preparation of the Schiff Base (L^1)

The Schiff Base was prepared with a slightly moderation as describe by Mahmud [5]. A solution of benzaldehyde (0.01mole) was slowly added to a solution of (0.01mole) 2-aminopyridine in 25cm³ of ethanol in the present of HCl as catalyst. The mixture was refluxed for 3hours. The mixture was allowed to cool at room temperature. The product obtained was filtered, washed in ethanol and dried in desiccators.



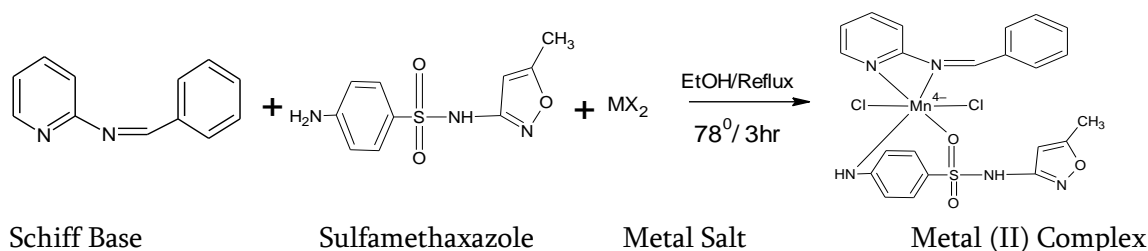
Scheme 1: Schiff Base (L^1).

Preparations of Mixed Ligand with Metal (II) Salt $\{M(L^1)(L^2)\}$

The Metal (II) complexes were prepared with a slightly moderation as describe by Mahmud [5].

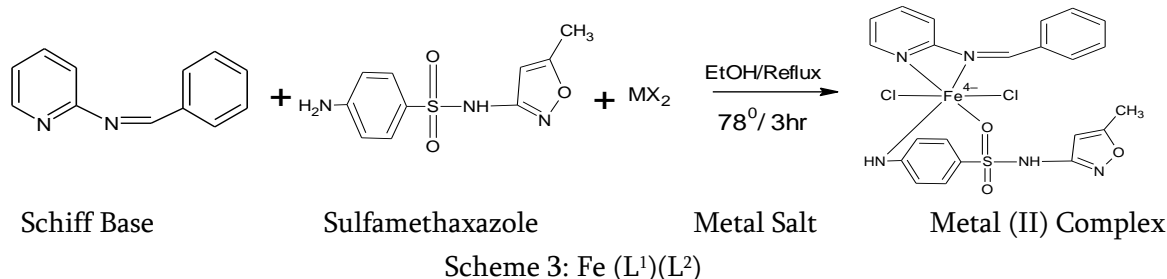
Preparation of Mn (L^1), (L^2)

A solution of $MnCl_2 \cdot 4H_2O$ containing 0.198g (0.01mol) in 15cm³ of ethanol was mixed with 1.82g(0.01mol) of Schiff Base and 2.53g(0.01mol) Sulfamethaxazole in 15cm³ of ethanol in a ratio of 1:1:1. The mixture was refluxed for 3hours and the solution was allowed to cool at room temperature. The product obtained was filtered and dried in desiccators.

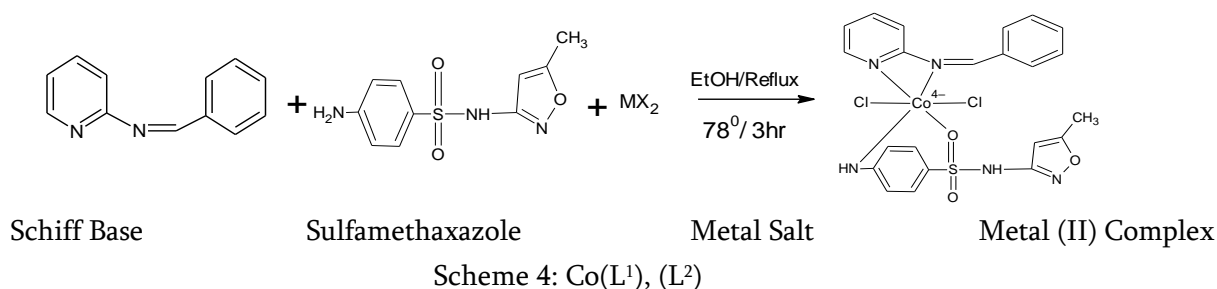


Scheme 2: Mn (L¹)(L²)**Preparation of Fe (L¹), (L²)**

A solution of FeCl₂.6H₂O containing 0.236g (0.01mol) in 15cm³ of ethanol was mixed with 1.83g (0.01mol) Schiff Base and 2.53g(0.01mol) Sulfamethaxazole in 15cm³ of ethanol in a ratio of 1:1:1. The mixture was refluxed for 3hours and the solution was allowed to cool at room temperature. The product obtained was filtered and dried in desiccators.

**Preparation of Co (L¹), (L²)**

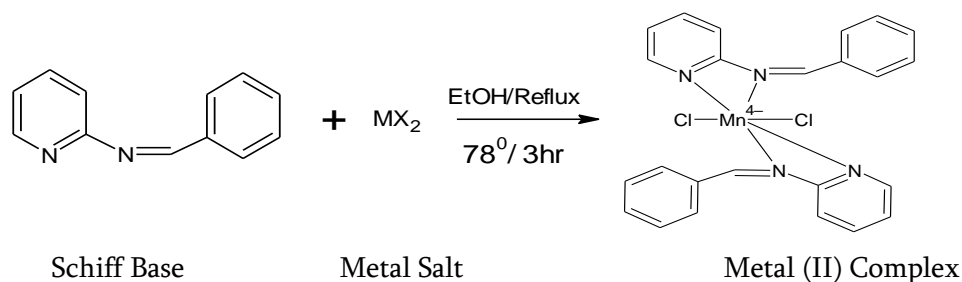
A solution of CoCl₂.6H₂O containing 0.238g (0.01mol) in 15cm³ of ethanol was mixed with 1.82g(0.01mol) of Schiff Base and 2.53g (0.01mol) Sulfamethaxazole in 15cm³ of ethanol in a ratio of 1:1:1. The mixture was refluxed for 3hours and the solution was allowed to cool at room temperature. The product obtained was filtered and dried in desiccators.

**Preparation of Schiff Base with Metal (Ii) Salt {M(L¹)₂}**

The Schiff base with Metal (II) complexes were prepared with a slightly moderation as describe by Raman [6].

Preparation of Mn {M(L¹)₂}

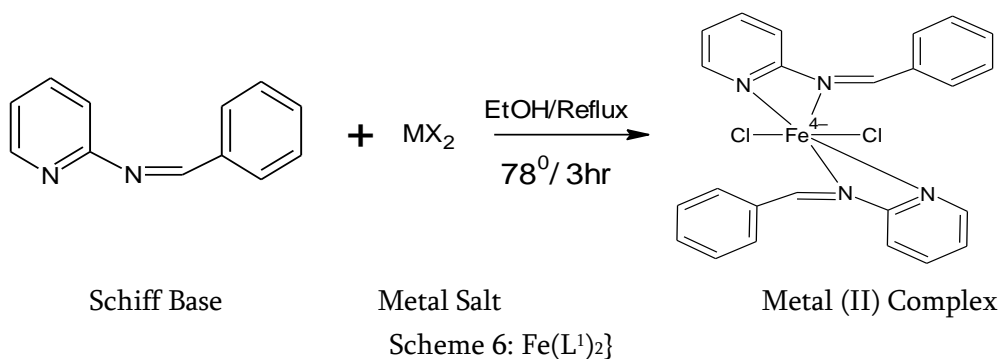
A solution of MnCl₂.4H₂O containing (0.01mol) were dissolve in 25cm³ of hot ethanol and (0.01mol) of the Schiff base was added to the solution, 1:2 molar ratio of metal-ligand. The mixture was heated at 80°C while stirring under refluxed for 3hrs. The product was filtered, washed in distilled in ethanol, dried in desiccator over calcium chloride for 1 day



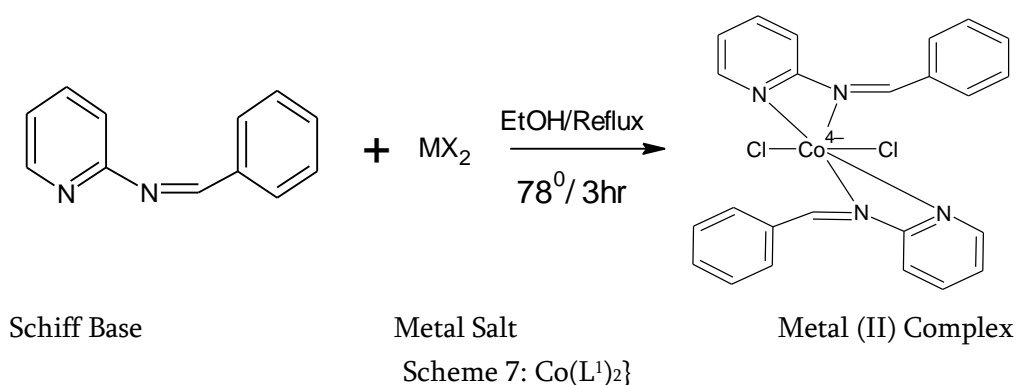
Scheme 5: $Mn(L^1)_2$

Preparation of Fe $\{M(L^1)_2\}$

A solution of $FeCl_2 \cdot 6H_2O$ containing (0.01mol) were dissolve in $25cm^3$ of hot ethanol and (0.01mol) of the Schiff base was added to the solution, 1:2 molar ratio of metal-ligand. The mixture was heated at $80^\circ C$ while stirring under refluxed for 3hrs. The product was filtered, washed in distilled in ethanol, dried in desiccator over calcium chloride for 1 day.

**Preparation of Co $\{M(L^1)_2\}$**

A solution of $CoCl_2 \cdot 6H_2O$ containing (0.01mol) were dissolve in $25cm^3$ of hot ethanol and (0.01mol) of the Schiff base was added to the solution, 1:2 molar ratio of metal-ligand. The mixture was heated at $80^\circ C$ while stirring under refluxed for 3hrs. The product was filtered, washed in distilled in ethanol, dried in desiccator over calcium chloride for 1 day.

**Physical Characterization of the Schiff Base and Metal (II) complexes****Solubility Test**

The Solubility Test was carried out in solvents such as Dimethylsulphoxiden (DMSO), Chloroform, Acetone, Methanol and Distilled water. 6mg of the Schiff Base and the Metal (II) complexes were dissolved in $2cm^3$ of different solvent at room temperature and at elevated temperature separately and the solubility were checked.

Color and Texture

The color and texture of the synthesized Schiff base and its Metal (II) complexes were determine by it physical appearance after bringing out from desiccator.

Melting Point Determination

The melting points were determined on electro thermal melting point apparatus by the procedures; A dry sample was put into the capillarity tube. The open end of the capillarity tube was pushed into the powder and gently

tapping the closed end on a bench. Both the capillarity tube and a thermometer were installed on melting point determination machine and the temperatures were checked and record in the thermometer.

Conductivity Test

The molar conductivity was carried out using conductivity meter model HI9835 of Hanna with a cell constant of at $2 \times 10^{-1} \Omega^{-1} \text{cm}^{-2} \text{mol}^{-1}$ at a concentration of 10^2M solution in DMSO as a solvent [4].

Percentage Yield

The percentage yield was calculated using the formula.

$$\text{percentage yield} = \frac{\text{experimental yield}}{\text{theoretical yield}} \times 100$$

Characterizations

FTIR Analysis

Buck scientific infrared spectrophotometer, Model 530 Buck Sci. USA, within the range of $(4000-400 \text{cm}^{-1})$.

UV-Visible

The UV-visible spectrophotometer was used to determine the wavelength of absorption range.

III. RESULTS AND DISCUSSION

Melting point of the Schiff base and Metal (II) Complexes

The melting points of the mixed ligand $[\text{M}(\text{L}^1)(\text{L}^2)]$ and the Metal (II) $[\text{M}(\text{L}^1)_2]$ Complexes are higher than the Schiff Base (L^1) due, to the different structural arrangements and bond strengths within the $\text{M}(\text{L}^1)(\text{L}^2)$ and $\text{M}(\text{L}^1)_2$ Complexes [4].

Color of the Compound

The color of the complexes were recorded in table 4.1, $\text{Mn}(\text{L}^1)(\text{L}^2)$ complex were found to be Dark green similarly Fe complex was found to be Light green, where $\text{Co}(\text{L}^1)(\text{L}^2)$ complex was found to be Brown, while $\text{Ni}(\text{L}^1)(\text{L}^2)$ complex was found to be Black and finally $\text{Cu}(\text{L}^1)(\text{L}^2)$ complex was found to be Light Brown, $\text{Fe}(\text{L}^1)(\text{L}^2)$ Complex was found to be Light Green will for the $\text{Mn}(\text{L}^1)_2$ is brown, $\text{Fe}(\text{L}^1)_2$ is blue-black, $\text{Co}(\text{L}^1)_2$ is brown, $\text{Ni}(\text{L}^1)_2$ was found to be brown and finally $\text{Cu}(\text{L}^1)_2$ dark blue. The colors of these complexes arise as a result of either from d-d transition, charge transfer transition or imperfection in the crystal structure of the compound [4].

Texture of the Complexes

The textures of the complexes were recorded in the table 1. above. These show that the (L^1) is crystal while all the complexes of the $\text{M}(\text{L}^1)(\text{L}^2)$ and $\text{M}(\text{L}^1)_2$ are powders [4].

Percentage Yield

The percentage yield of the (L^1) is lower than the $\text{M}(\text{L}^1)(\text{L}^2)$ and $\text{M}(\text{L}^1)_2$ complexes, except in $\text{Mn}(\text{L}^1)(\text{L}^2)$ complex which has lower than the (L^1) due to incomplete reaction of the complex during the synthesis [3].

Molar Conductivity

The conductivity value for the (L¹) and the M(L¹L²) and M(L¹)₂ complexes ranged between 21×10⁻² 14×10⁻² Ω⁻¹cm⁻²mol⁻¹. Indicated that both (L¹) and the M(L¹L²) and M(L¹)₂ complexes have low conductivity value showing that they are non-electrolyte [3].

Table I. Physical Characterization of the Schiff Base (L¹) and Metals (II) Complexes. [M (L¹)₂ and M(L¹)(L²)]

Compound	Melting P.(°C)	Conductivity Ω ⁻¹ cm ⁻² mol ⁻¹	Colors	Texture	Estimated (%)
SB	200	21×10 ⁻²	White	Crystal	52
Mn(L ¹) ₂	230	7 10 ⁻²	Brown	Powder	55
Fe(L ¹) ₂	230	6 10 ⁻²	Blue black	Powder	78
Co(L ¹) ₂	250	4.8 10 ⁻²	Brown	Powder	75
Mn(L ¹)(L ²)	270	2.0×10 ⁻²	Dark Green	Powder	32
Fe(L ¹)(L ²)	290	16×10 ⁻²	Light Green	Powder	88
Co(L ¹)(L ²)	250	14×10 ⁻²	Brown	Powder	72

Solubility test

The synthesized Schiff base and its Metal (II) complexes are soluble in water, Acetone and Dimethylsulphoxide (DMSO), insoluble in chloroform and are slightly soluble in methanol.

Table II. Solubility test for Schiff base and the Metal (II) complexes

Compound	H ₂ O		MeOH		Acetone		Cloroform		DMSO	
	R	E	R	E	R	E	R	E	R	E
SB	S	S	SS	SS	S	S	IS	IS	S	S
Mn(L ¹) ₂	S	S	SS	SS	S	S	IS	IS	S	S
Fe(L ¹) ₂	S	S	SS	SS	S	S	IS	IS	S	S
Co(L ¹) ₂	S	S	SS	SS	S	S	IS	IS	S	S
Mn(L ¹)(L ²)	S	S	SS	SS	S	S	IS	IS	S	S
Fe(L ¹)(L ²)	S	S	SS	SS	S	S	IS	IS	S	S
Co(L ¹)(L ²)	S	S	SS	SS	S	S	IS	IS	S	S

R= Room Temperature, E=Elevated Temperature, S=Soluble, SS = Slightly Soluble and IS = Insoluble

FT-IR Analysis

The Metal ligand bond was confirmed by comparing the IR spectra of the Schiff base ligand with metal (II) complexes. The FT-IR spectra predicted all the absorption bands of the Schiff base ligand and some new bands at specific frequency confirmed the modes of absorption and the completion of the ligand with the metal ions

through nitrogen and oxygen [5]. The stretch vibration due to hydroxyl of the Schiff base was observed as a broad and weak at 3307- 3632 cm^{-1} . This is a consequence of strong intramolecular hydrogen bonding between the hydroxyl proton and imine nitrogen OH-NH [1]. The strong band at 1603-1651 cm^{-1} was assigned to the imine $\nu(\text{C}=\text{N})$ band of the ligand, the increase in the $\nu(\text{C}=\text{N})$ values for the 2-aminopyridine and benzaldehyde (Schiff base) may be understood to be due to the presence of the methylene bridge that isolated the pyridine ring from the π conjugation of the rest of the molecule [7]. While the spectra of Mn(II), Fe(II) and Co(II) the band 1640 cm^{-1} , 1615 cm^{-1} and 1629 cm^{-1} due to azomethine linkage in the ligand is shifted to higher frequency than Schiff base 1603 cm^{-1} in the complexes, indicating the participation of the azomethine Nitrogen in coordination with the metal ions [4]. Further evidence of the coordination of the Schiff base and the metal ions was shown by the appearance of weak frequency new bands of M-N and M-O vibration (595 cm^{-1} - 760 cm^{-1}) and (482 cm^{-1} - 618 cm^{-1}) respectively, these bands are absent in the spectra of the Schiff base [7].

Table III. *FTIR data of Mixed Ligand M(L¹)(L²) Complexes*

COMPOUND	V(NH)	V(C=N)	V(M-N)	V(M-O)
SB	3307	1603	----	-----
Mn(L ¹)(L ²)	3632	1640	610	482
Fe(L ¹)(L ²)	3408	1615	668	518
Co(L ¹)(L ²)	5631	1629	595	495

* cm^{-1}

The lattice and coordinated water were observed at (3307-3435). The spectra of the first ligand (L¹) showed a strong band at 3300 cm^{-1} due to stretching vibration of O-H group, this band was shifted to the lower frequency in the spectrum of Metal (II) complexes 3435 cm^{-1} as a result of complexation [7]. The band appeared at 3300 cm^{-1} was attributed to stretching vibration of N-H group for Schiff base (L¹) and which absent in spectra of Metal (II) complexes because of coordinate through the N atom [2]. The spectra of L¹ and Metal (II) Complexes showed bands at 2200, 2256, 2267, 2366, 2366 and 2363 cm^{-1} respectively, these bands assigned to vibrational mode of C-H. The spectrum of L¹ exhibited the moderate band at 1603 cm^{-1} belong to C=N, while this band was shifted to lower frequency at 1635, 1620, 1527, 1635 and 1627 cm^{-1} for Metal (II) Complexes respectively [8]. The strong band at 845, 725, 798, 845 and 749 cm^{-1} is due to M-N, this band is absented in the spectra of the L¹ (Schiff base) as a result of coordinate N atom of M=N with metal ions. The bands appeared at 698, 578, 667, 689 and 564 cm^{-1} is due to stretching vibration of M-Cl [7].

Table IV. *FTIR Data of Schiff Base (L¹)₂ and Metal (II) Complexes*

COMPOUND	V(O-H)	V(C-N)	V(C=H)	V(M-N)	V(M-Cl)
SB	3307	1603	200	----	-----
Mn(L ¹) ₂	3698	1635	2256	845	698
Fe(L ¹) ₂	3417	1620	2267	725	578
Co(L ¹) ₂	3377	1627	2363	749	564

*cm⁻¹

UV-visible spectra of M(L¹)(L²) complexes

The ultraviolet visible electronic spectrum of the Schiff base (L¹) complexes is shown in figure 5 above. The Schiff base showed three weak bands at 500, 600 and 700nm attributed to $\pi \rightarrow p^*$, $\pi \rightarrow p^*$ and $n \rightarrow p^*$ electronic transitions respectively. For all complexes it showed the similar electronic transition as observed in the Schiff base and it also showed the electronic transitions of the metal d orbital (d-d electronic transition) observed located in the visible region as extra information [7].

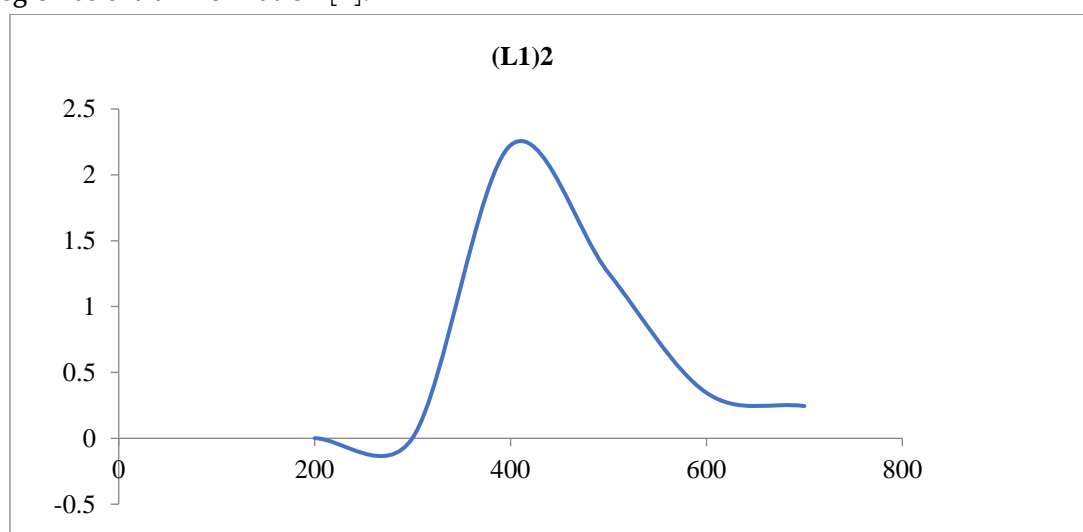


Figure 1: L¹

The electronic spectrum of the Fe (II) complex showed two bands. The band at 400nm may be assigned to the $^5T_{2g} \rightarrow ^5E_g$ transition and the other at 500nm to charge transfer. Similar types of transitions were reported for octahedral Fe(L¹)(L²) complexes [7].

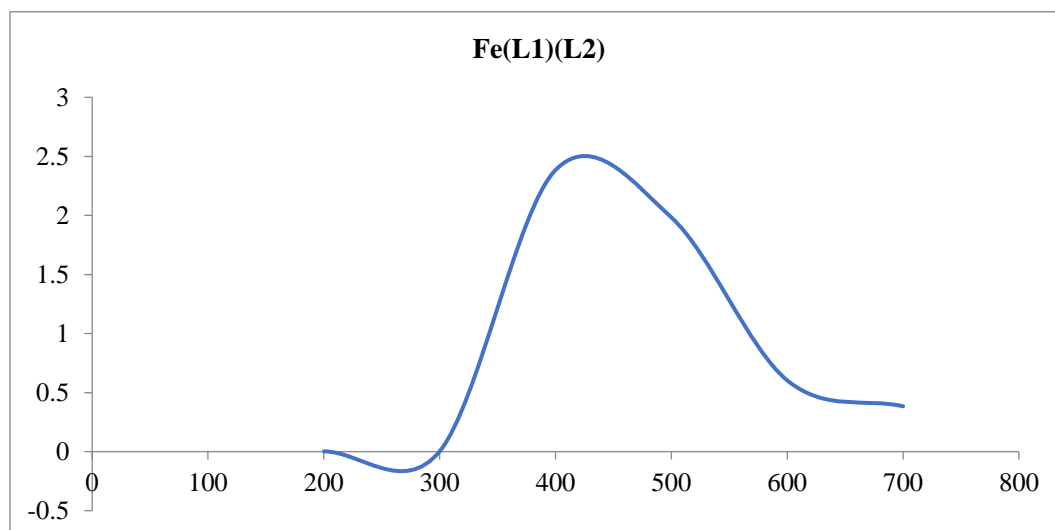


Figure 2: $Fe(L^1)(L^2)$

The electronic spectra of the complexes were recorded in the solution state. The energies of the observed spin allowed bands in all the complexes agreed with the octahedral geometry. The electronic spectrum of $Mn(L^1)(L^2)$ complex shows four weak bands at bands at 400, 500, and 600nm, which can be assigned to $6A_{1g} \rightarrow 6T_{1g}$, $6A_{1g} \rightarrow 4T_{2g}$, $6A_{1g} \rightarrow 4E_g$ and $6A_{1g} \rightarrow 4T_{1g}$, respectively, for an Mn(II) ion in an octahedral field [7].

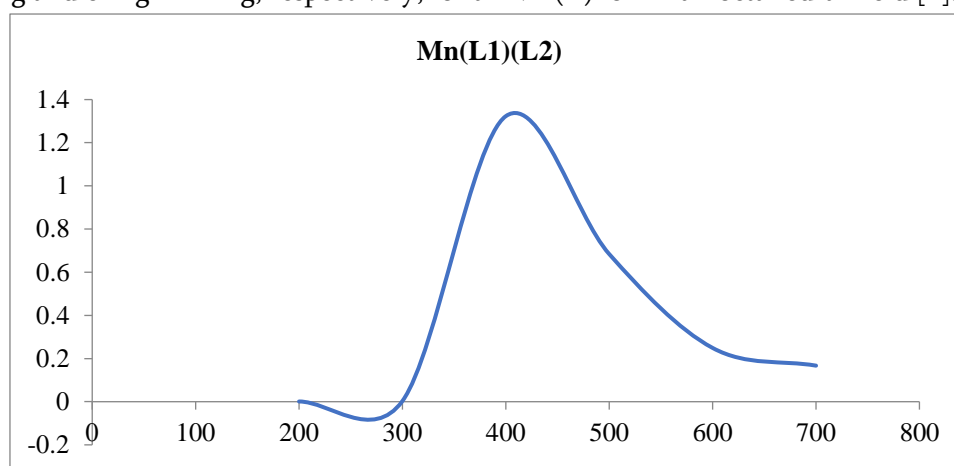


Figure 3: $Mn(L^1)(L^2)$

The electronic spectra of octahedral $Co(L^1)(L^2)$ species are theoretically expected to have three spin-allowed transitions from the ground state: $4T_{1g}(F)$ to $4T_{2g}(F)$, $4A_{2g}(F)$ and $4T_{1g}(P)$, referred to hereafter as ν_1 , ν_2 and ν_3 , respectively. In addition, spin-forbidden transitions to doublet terms may appear. The second spin-allowed transition, ν_2 , in the spectra of oxygen-coordinated octahedral species is usually obscured because of one or more of the following factors. (i) Within the spectral region where ν_2 is expected, weak bands due to spin-forbidden transitions to various levels descending from the free-ion term, $2G$, may also appear; the weak ν_2 may be easily confused with any of them. (ii) In octahedral fields of oxygen-coordinating ligands, the value of the crystal fields splitting parameter, Dq , is such that the energy of $4A_{2g}(F)$ is nearly coincident with that of $4T_{1g}(P)$. This is expected to cause overlapping of ν_2 and ν_3 leading to masking of the weak ν_2 by the intense broad ν_3 . The electronic spectra data of the $Co(L^1)(L^2)$ complex are shown in Fig. 4. The electronic spectral data reveal four bands at 200, 300, 400, 500, 600 and 700 nm. The band appearing in 200-300 nm is attributed to $\pi \rightarrow \pi^*$ transition of the benzene ring. Furthermore, the absorption spectra of complex illustrate bands in the 300-500 nm assigned

to the $n \rightarrow \pi^*$ transitions of the azomethine group and to the presence of charge transfer transition, respectively. Also, band at 600-700 nm assignable to [${}^4T_{1g}(F) \rightarrow {}^4T_{1g}(P)$] transition, characteristic of Octahedral geometry [7].

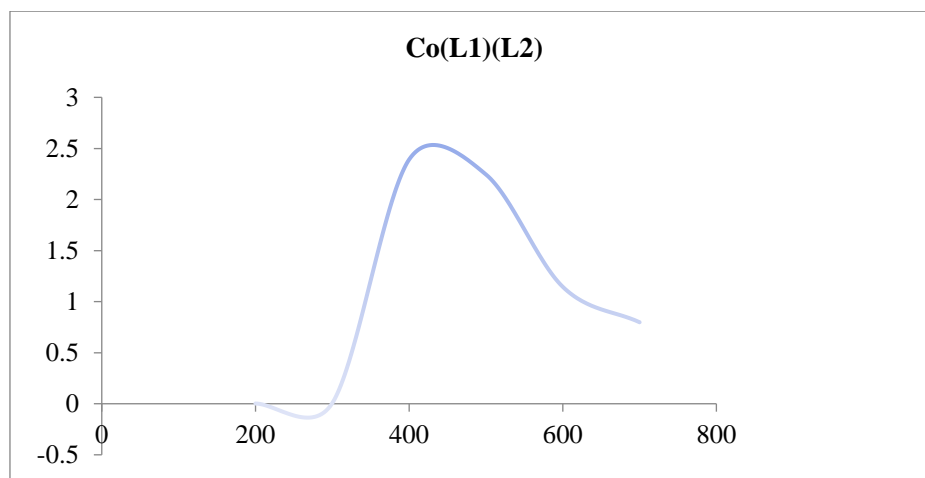


Figure 4: $Co(L^1)(L^2)$

UV-visible spectra of $M(L^1)$ complexes

The Ultraviolet Spectrum of $Mn(L^1)_2$ Complex in Figure above designated the maximum peak around 460nm, indicating $n \rightarrow \pi^*$ Electronic Transition Which fall within the Visible Region, which indicate octahedral geometry of the $Mn(L^1)_2$ complex [7].

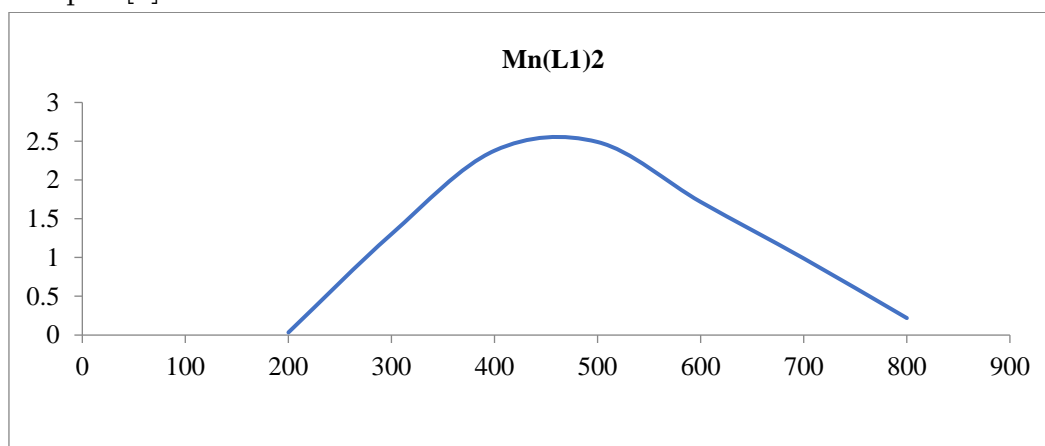


Figure 5: UV Spectrum of $Mn(L^1)_2$ Complex

The figure above represented the UV-Spectrum of $Fe(L^1)_2$ Complex that Shows the λ -max at 410nm, this is due to Electronic Transition that fall in the Visible Region, which indicate octahedral geometry of the $Fe(L^1)_2$ complex [7].

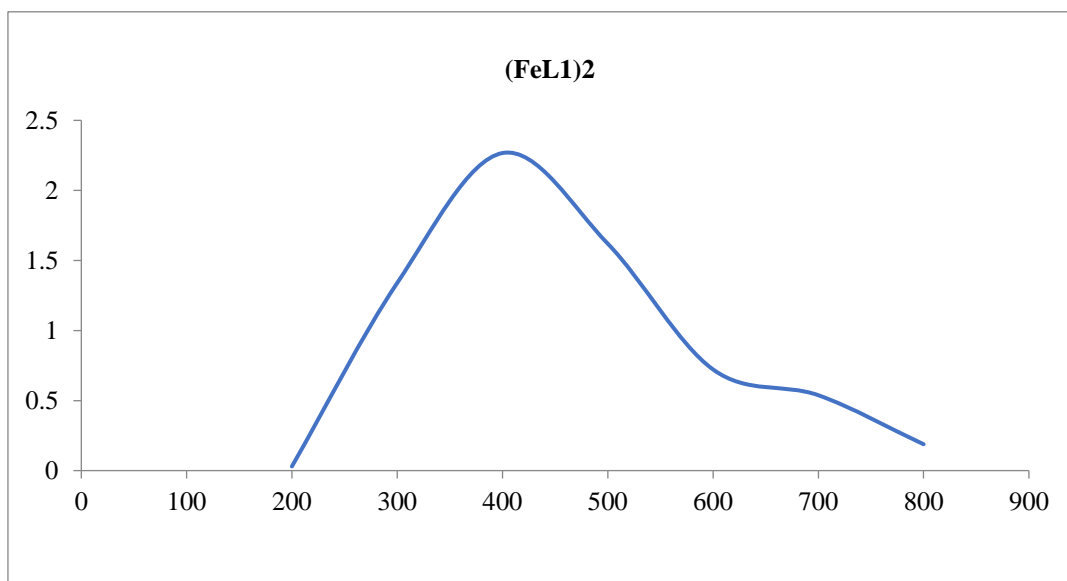


Figure 6: UV Spectrum of $Fe(L^1)_2$ Complex

In $Co(L^1)_2$ Complex of Figure above, the UV-Spectrum Showed a Maximum absorption Peak of the wavelength at 490nm which attributed to $n \rightarrow \pi^*$ Electronic Transition. In which indicate octahedral geometry of the $Co(L^1)_2$ complex [7].

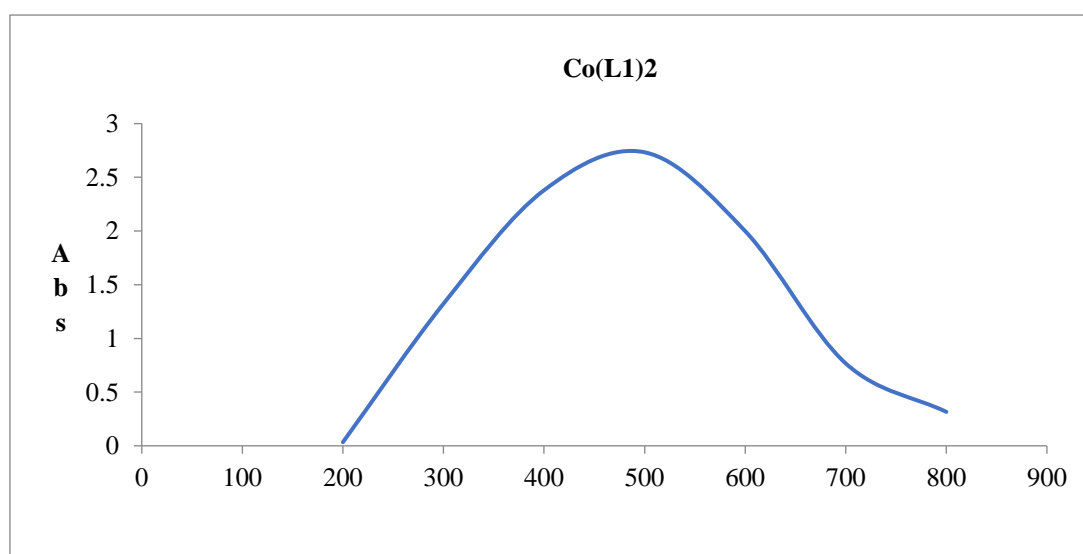


Figure 7: UV Spectrum of $Co(L^1)_2$ Complex

may be because of the change in concentration of constituents of the plant according to different geographical regions.

IV. CONCLUSION

In Conclusion, the synthesis of the Schiff base was obtained from the reaction 2-aminopyridine with benzaldehyde has been described. The Metal (II) (Mn, Fe, and Co) complexes were obtained from the reaction

of the Schiff base and the Metals (II) Salt which have been described. The mixed ligand Complexes of Metal (II) (Mn, Fe, and Co) was obtained from the reaction of Schiff base, Metals (II) Salt and Sulfamethaxazole drugs which have been described. The Schiff base and Metal (II) Complexes were Characterization by FTIR and UV-Visible. In mixed ligand Complexes based on the FTIR data the Schiff Base (Azomethane) and the Sulfamethaxazole drugs (N and O) have been found to

be the coordination sites. While in the Metal (II) Complexes based on the FTIR data the Schiff Base (Azomethane) have been found to be the coordination sites to the Metal Salt. Based on the UV-Visible data both mixed ligand and Metal (II) Complexes have been found that the geometry is octahedral.

Conflict of interest

No conflict of interest declared

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