Received: 2025-07-28
Accepted: 2025-08-12
Published Online: 2025-09-07
DOI: 10.70454/IJMRE.2025.05035

International Journal of Multidisciplinary Research and Explorer (IJMRE)
E-ISSN: 2833-7298, P-ISSN: 2833-7301

Synthesis and Characterization of Copper (II) Complex Bearing 1-Phenyliminomethyl-Naphthalen-2-ol

Babajide Joseph Adisa[1], Daniel Ayodele Idowu[2], Chidera Godswill Obideje[3], Ejikeme Peter Igwe[4], Okiki Quadri[5], Emmanuel Ogbonnia Oko[6], Gabriel Lagbel[7]

[1,2]Department of Chemistry, Federal University of Agriculture, Abeokuta, Ogun State, Nigeria
[3]Department of Biochemistry, Federal University of Technology, Minna, Niger State, Nigeria
[4]Department of Biochemistry, University of Nigeria, Nsukka, Enugu State, Nigeria
[5]Department of Chemistry and Biochemistry, Northern Illinois University, Illinois, USA
[6]Department of Chemistry, Michael Okpara University of Agriculture, Umudike, Abia State, Nigeria
[7]Department of Physics, Kwame Nkrumah University of Science and Technology, Kumasi, Ghana

[1]babajide20172308@gmail.com, [2]Idowudaniel284@gmail.com
[3]godswillobideje@gmail.com,[4]igweejikeme@gmail.com
[5]quadri.okikiola@gmail.com,[6]okoemmanuel8@gmail.com
[7]mastergabby@gmail.com

Corresponding email: Idowudaniel284@gmail.com

Abstract

A novel Schiff base ligand, 1-phenyliminomethyl-naphthalen-2-ol was synthesized by condensation of 2-hydroxyl-1-naphtaldehyde and aniline, and subsequently complexed with Cu(II) ions to become a stable metal-ligand complex. The synthesized Schiff base and Cu(II) complex have been characterized using UV–Visible spectroscopy and Fourier-transform infrared spectroscopy. The UV-Vis analysis revealed that metal-ligand charge transfer which depicted low band at 406, 350 nm (for the two molar ratio 1:1, 2:1) respectively. The FTIR absorption spectra confirmed the formation of the imine (C=N) bond and coordination of the phenolic oxygen and azomethine nitrogen. NH bonds revealed coordination at 3340 and 3366 cm^{-1} while CN bonds showed no coordination and 1161 cm^{-1}, both at 1:1, 2:1 respectively. In conclusion, these findings supports the successful formation of a bidentate Schiff base to Cu(II) complex with a potential application in biological and catalysis studies.

Keywords: Cu(II) Complex, Schiff Base, UV-Vis and FT-IR Spectroscopy, 2-hydroxyl-1-naphtaldehyde, Aniline.

INTRODUCTION

Schiff bases have been shown to be an important part in the development of coordination chemistry. Their metal complexes have been widespread studied because of their attractive chemical and physical properties and their wide range of applications in numerous scientific areas. Both synthetic and structural research have an important role to play in these bases because of their preparative accessibility and structural diversity (Nagajothi et al., 2012). Their bases have generated a lot of attention due to its synthesis, structure, and reactivity, relating to their ease of preparation and structural flexibility. Also, their potential applications cut across different potential field such as in biological modeling, catalysis, design of molecular magnets, and the synthesis of therapeutic agents (Nica et al., 2015; Xiang et al., 2019; Rangaswamy et al., 2012; McGarrigle and Gilheany, 2019).

Schiff bases are known to be chemical compounds characterized by the presence of the azo-methine group (C=N) (Al-Daffaay., 2022; Al-khazraji., 2023). They are characterized by the formation of reaction of carbonyl groups with primary amines. These compounds are known for their ability to function as ligands, forming metal complexes that stabilize metal ions in various oxidation states. One of their unique properties make them valuable in a range of catalytic and industrial processes. These metal complexes of Schiff bases that contain oxygen and nitrogen as donor atoms are particularly studied because of their flexibility, sensitivity to environmental reactions, and distinctive structural characteristics. Copper-binding Schiff-base ligands, in reference, have gained significant attention for their relevant in optoelectronics, catalysis, and biomolecular studies (Al-Redha et al., 2022). Previous studies has explored the synthesis of various these bases through condensation processes (Alothman and Albaqami., 2020).

These bases were originated by Hugo Schiff in 1864 which are important class of organic compound (Hussain et al., 2014). They are also a type chemical compounds containing carbon-nitrogen double bond as functional group, where the nitrogen atom connected to aryl group or alkyl group (R) but not hydrogen. Since discovery, many scientist shows great interest in the field of transition metal complexes, and it also represent one of the most employed class of ligands in coordination chemistry due convenient synthetic preparation and high versatility. These properties influence their ability to form stable complexes with the large majority of transition metal ions.

Larger number of Schiff bases and their metal complexes has been well studied due to their fascinating properties which include the ability to reversibly bind oxygen, catalytic activity in hydrogenation of Olefins, transfer of an amino group, photochromatic properties and complexing ability toward some toxic metals (Chandra et al., 2007).

The high affinity for the chelation of the Schiff bases towards the transition metal ions is utilized in preparing the solid complex. Their complexes also have been a subject of interest due to growing applications in the field of catalysis. Particularly, in transition metals, they possess distinct characteristics properties like variable in oxidation states, metals offers a wide range of oxidation state and reactivity of the metal complexes depend upon the stability and inter convertibility of these oxidation states. Most widely used organic compounds, have their applications as pigments and dyes, catalyst, intermediate in organic synthesis and as polymer stabilizers. It has shown a broad range of biological activities, which include antifungal, antibacterial, antimalarial, antiviral and antipyretic properties (Da Silva et al., 2011). The current research employs advanced characterization techniques, including 1 H- and 13C-NMR, UV-Vis, and FTIR spectroscopy, to analyze the molecular structure of these compounds. This study is aimed at synthesizing compounds through metal ion coordination from a Schiff base ligand of 2-hydroxyl-1-naphtaldehyde and aniline to synthesize copper metal complex of the Schiff base.

2.0 MATERIALS AND METHODS

2.1 Materials

The Cu(II)chloride dihydrate ($CuCl_2.2H_20$), methanol (CH_3OH), aniline($C_6H_5NH_2$), and 2-hydroxy-1-naphthaldehyde used for this investigation are in Analytical grades and were used without further purification (Idowu et al., 2025). De-ionized water was used for the preparation of different concentration of samples.

2.2 Instrumentation

The UV-visible spectra of the synthesized compounds were measured using a SHIMADZU UV-1800 spectrophotometer in the range of 200–800 nm. FT-IR spectra were recorded with a SHIMADZU 8400 FTIR spectrophotometer for analysis within the 650–4000 cm^{-1} range.

2.3 Synthesis

The Schiff base ligand and its complexes were synthesized according to the general synthetic procedure (Gajendra et al., 2010; Prakasha et al., 2011).

2.3.1 Synthesis of Schiff Base.

According to the method used, aniline and 2-hydroxyl-1-naphthaldehyde were used to synthesize the Schiff base. The Schiff base was prepared by condensation of salicyaldehyde (0.005M) with various aromatic amines (0.005M) in water (10 mL) and the mixture was stirred at ambient temperature. After the production of the Schiff base, the metal salt (Cu(II) chloride dihydrate ($CuCl_2.2H_2O$)) was weighed and dissolved in distilled water and methanol.

2.3.2. Synthesis of Metal Complexes

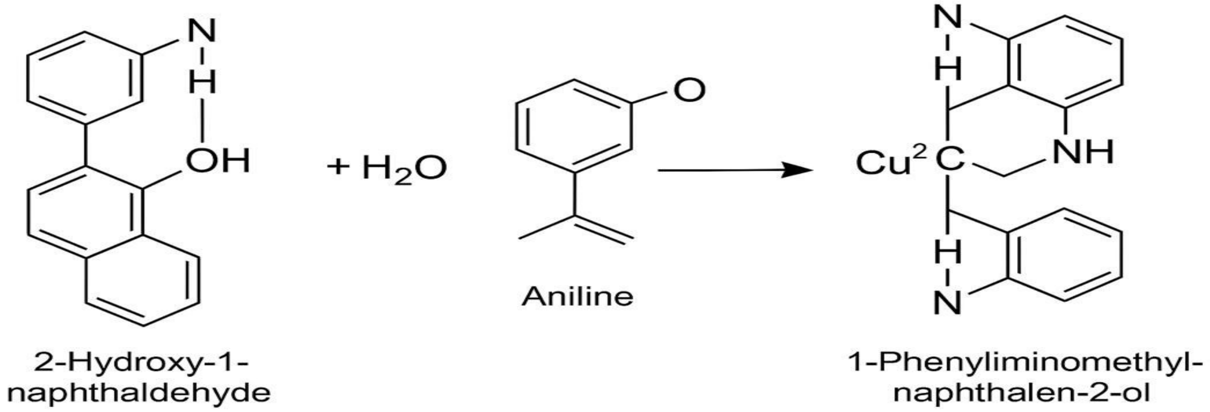
The metal salt was soluble in distilled water and methanol. The solution of metal salt (40 mL), was then poured in a round bottom flask with magnetic stirrer the Schiff base of same volume was poured in the round bottom flask with magnetic stirrer and placed on hot plate, and allowed to run for one and half hours. This process was repeated consecutively for 1:1 and 2:1. After rigorous mixing for about one and half hours, the solution was poured in a beaker, condensed through evaporation by perforating the paper that was used to cover the beaker. After perforation it was allowed to dry for few days. The condensed yield is then scrapped through the help of a spatula into sample bottles for FT-IR and UV-Vis characterization, the sample was put directly into the sample bottles, and for UV-Vis analysis, the sample was first dissolved in an organic solvent (methanol).

2.4 Determination of Number of Coordinated Ligand

The number of the ligands coordinated to the metal ion was determined using Job's method of continuous variation (Angelici, 1971). 3 mL aqueous solution and the metal chlorides were prepared. 16 mL of the total volume was maintained throughout the process and the mole fraction of the ligand was calculated in each mixture. The solutions of the metal chlorides were scanned (as blank) to find the wavelength of maximum absorption for the particular metal ion (Angelici, 1971). The machine was fixed at λmax before taking the absorbance values. A graph showing plot of absorbance against mole fractions was made and by extrapolation, mole fraction at maximum absorbance was recorded, which was the point where the metal ion and the ligand are in stoichiometric ratio.

3.0 RESULTS AND DISCUSSION

The ligand was prepared by condensing equimolar amounts of aniline with 2-hydroxy-1-naphthaldehyde in absolute methanol under reflux (see Scheme 1 below). The Cu(II) complexes were synthesized in 1:1 and 1:2 molar ratio of the Schiff base ligand and the metal(II) ions.



Scheme 1: Reaction equation of synthesis of the Schiff base with Cu(II) ion.

The analyses for the physical properties and melting point of the prepared compounds are given in Table 1 & 2. The yellow color of the ligand observed was used to predict the formation of the Schiff base. The analyses values for the ligand and its complexes in (Table 1 & 2), tallied with the expected values, thus confirming the purity of the compounds and the formation of the Schiff base ligand. A 1:1 and 2:1 reaction ratio for metal ion and the ligand during the formation of the complexes was formed. Thefore, the complexes were of the form [ML], which may thus further dimerize (Mohammed et al., 2014; Chahmana et al., 2017) to form a neutral dimeric four-coordinate complex $[ML]_2$.

Table 1: Physical properties of Ligand and Metal Complexes

Compound	Color	Nature
1-Phenyliminomethyl-naphthalen-2-ol	Golden-yellow	Liquid
Cu(II) complexes (1:1 and 2:1)	Yellow	Powdery upon filteration and air drying

Table 2: Melting point data of Metal Complexes

Sample	Melting point (°C)
Cu(II) complex 1:1	220.3
Cu(II) complex 2:1	222.8

3.1 UV-Visible Spectroscopy

From the result below in figure 1, there was a sample absorbance spectrum to the right. The y-axis represents absorbance and x-axis represents wavelength of light being scanned. The ligand, 2-hydroxyl-l-naphtaldehyde has the highest absorbance in the UV region of light around 225-270 nm, and two slight absorbance peaks around 319-400 nm. The latter peaks are less compared to former peaks due to the electron's transition being forbidden. It should be noted if a lower peak is formed, the transition is forbidden, just a few transition metal electrons will undergo the excitation. The spectrum of H_2L_1 exhibited high-intensity bands at 362 nm (27624 cm^{-1}), assigned to $\pi\longrightarrow\pi*$ transition, and at 465 nm (21505 cm^{-1}) assigned to $n\longrightarrow\pi*$ transition (Kabeer and Khaled, 2015; Chioma and Don-Lawson, 2018). The band at 362 nm the spectrum of the Schiff base experienced a blue shift in the spectra of the complexes. The spectrum of CuL_1 complex shows an absorption band at 586 nm attributed to $^2B_{1g}\longrightarrow{}^2A_{1g}$ transition of square planar geometry around Cu(II) while the band observed at 436 nm was attributed to charge transfer transition (Chioma and Don-Lawson, 2018). Also, Comparing to other metal complexes, the spectrum of NiL_1 exhibited absorption bands at 564 nm, attributed to $^3T_2(F)\longrightarrow{}^3T_1(F)$ transition which corresponds to tetrahedral

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geometry, and the band observed at 453 nm is charge transfer transitions (Kabeer and Khaled, 2015).

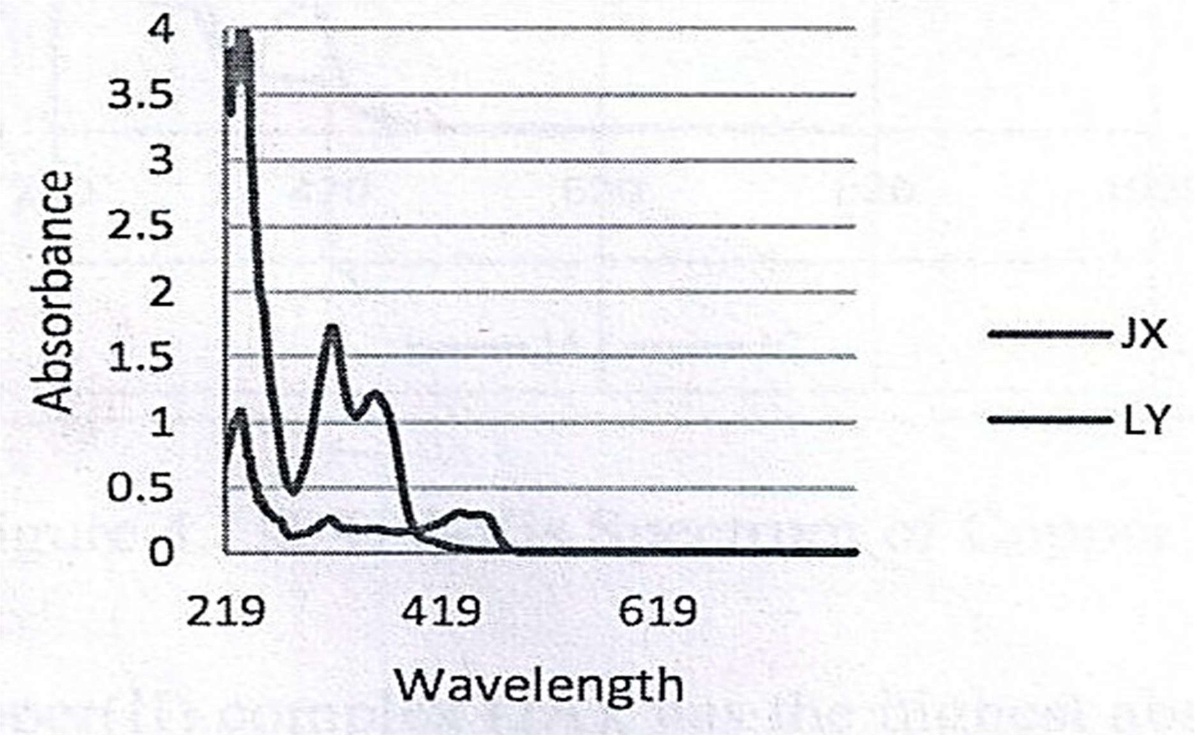

Figure 1. UV-Vis Spectrum of 1-phenyliminomethyl-naphta-2-ol.

From figure 2 below, Cu(II) complex (JA), has the highest absorbance in the UV region of light around 225-270 nm, and two slight absorbance peaks around 320-400 nm. The Cu(Il) complex (JC), has the highest absorbance in the UV region of light around 220-250 nm.

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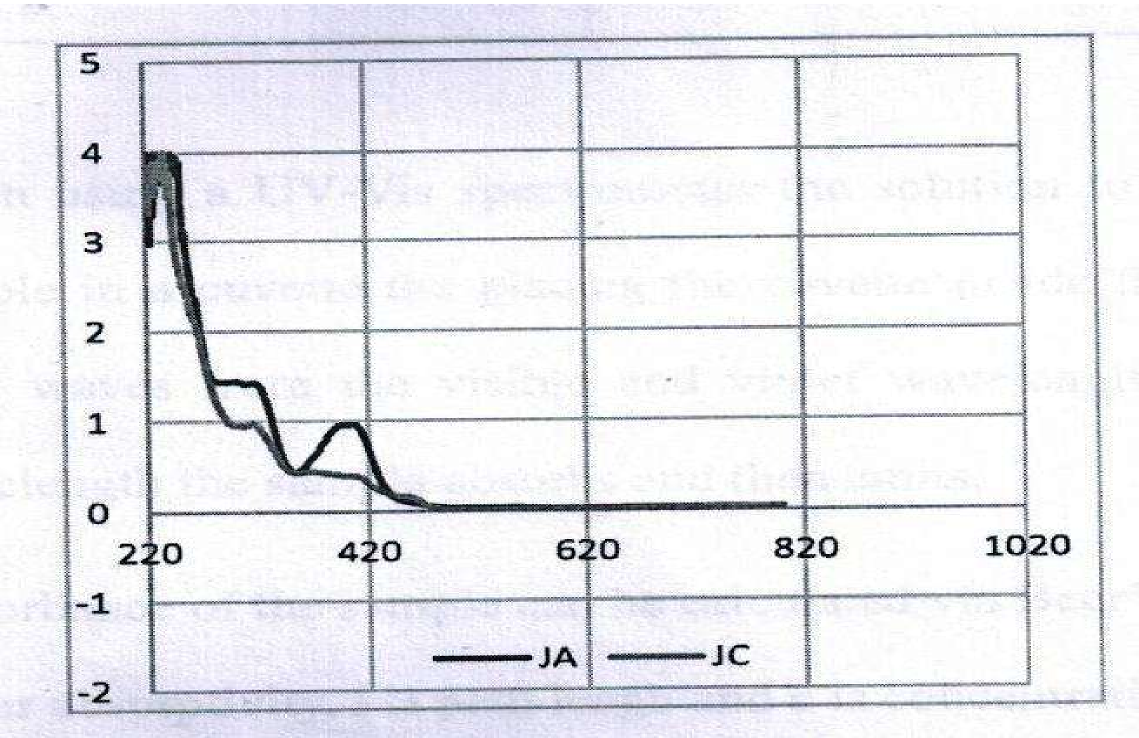

Figure 2: UV- Visible Spectrum of Copper(II) Complex

Table 3: Uv-Vis Spectroscopy showing Ligand and the Metal Complexes

Compound	Wavelength (nm)	Absorbance
1-Phenyliminomethyl-naphthalen-2-ol	230	1.1
Metal complex (2.1)	350	0.5
Metal complex (1.1)	406	1.0

When using a UV-Vis spectrometer, the solution to be analyzed was prepared by placing the sample in a cuvette, thereby placing the cuvette inside the spectrophotometer. The machine shined light waves from the visible and violet wavelengths and measures how much light of each wavelength the sample absorbs and then emits. Absorbance of the sample can be calculated via Beer's law: A=elc, where A is absorbance, e is molar absorptivity, I is path length and c is concentration of the sample.

3.2 FT-IR (Fourier Transform Infrared)

From the result below, comparing the FT-IR results of the Cu(II)complexes and the Schiff base. What interests us in spectra like these is how the bands for the coordinating functional groups change in comparison to the free ligand. To measure the spectrum of the free ligand, a spectrum of complex, the need to find the differences based on what we know about IR spectroscopy. The vibrational frequency stretch of imine CN absorptions in electromagnetic spectrum ranged from 1200 to 1350 cm^{-1}. In table 4, the Schiff base has noticeable peak at 1309 cm^{-1}, while the metal complex (2:1) has a peak at 1161 cm^{-1}, and there's no coordination with (1:1).

The NH group in both primary amines and amides is associated with three characteristic infrared absorption bands with vibrational frequency stretch around 3500cm^{-1} and 3300cm^{-1}. In the table 4, there's no coordination with the Schiff base. While the peaks at 3340cm^{-1} and 3366 cm^{-1} respectively, were noticeable on the electromagnetic spectrum for the Cu(II)complexes.

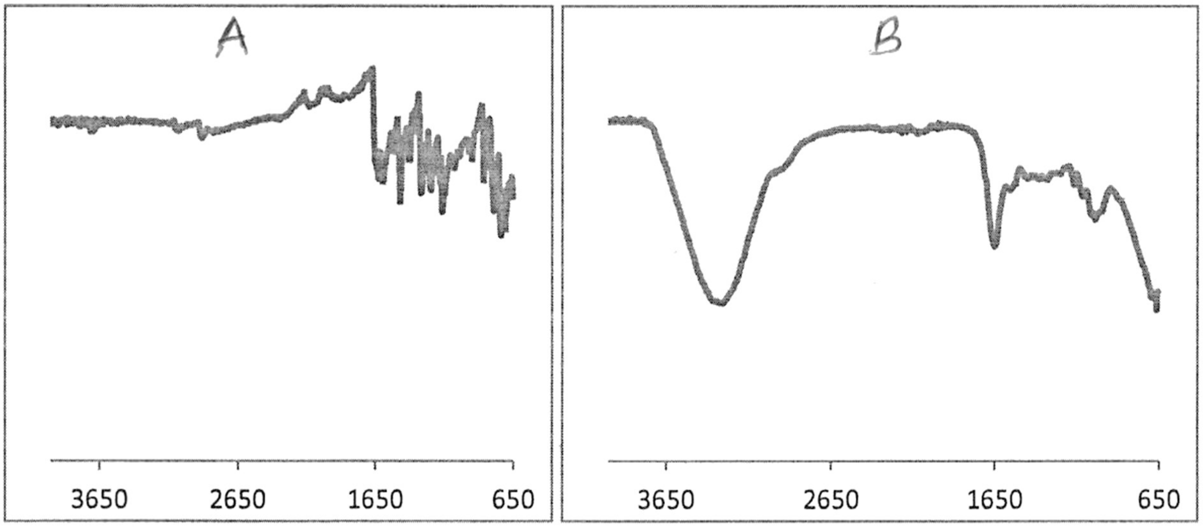

Figure 3: (A) FT-IR Spectrum of the free ligand (B) FT-IR Spectrum of the Cu(II) complex(1:1)

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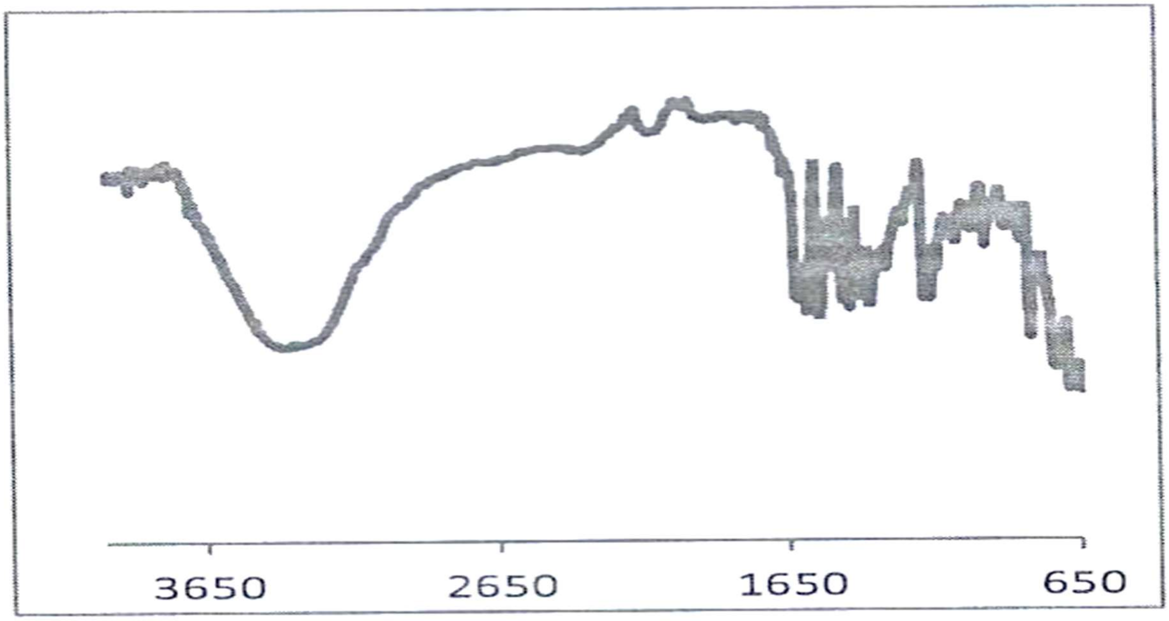

Figure 4: FT-IR Spectrum of Cu(II)complex (2:1)

Azomethine group of the Schiff base undergoes a shift to lower the frequency after complexation, indicating the coordination of azomethine nitrogen to metal atom and this can be explained by the donation of electrons from nitrogen to the empty d-orbital of the metal atom. The nature of metal-ligand bonding is confirmed by the newly formed band at about 440cm^{-1} in the spectra of the complexes which is tentatively assigned to M-N vibration. IR spectra of free ligand shows a band of 334 6cm^{-1} assigned to v(OH) stretching vibrations. A strong peak at 1607 cm^{-1} region were attributed to the azomethine v(C=N) group. (Mahmoud et al., 2016). These relative shifts observed for v(C=N) band in complexes showed the participation of azomethine nitrogen in coordination to the metal ions (Gehad et al., 2016). Two bands at absorptions 717-769 cm^{-1} and that of 441-518 cm^{-1} support the formation of M-N and M-O bands. (Ali, 2014). The common structural feature of Schiff's bases is the presence of the characteristic azomethine functionality, RHC=NR′, which has been shown to account for the observed pharmacological activities (McGarrigle and Gilheany., 2019).

Table 4: FT-IR Spectroscopy showing the Ligand and Metal Complexes

Compounds	NH Bonds (cm^{-1})	CN Bonds (cm^{-1})
1-Phenyliminomethyl-naphthalen-2-ol	No coordination	1309
Metal complex (1.1)	3340	No coordination
Metal complex (2.1)	3366	1161

Comparing the FT-IR result, amide and imine functional groups in the Schiff base and Cu(II) complexes. The infrared spectrum of the ligand exhibits absorption bands around 3255 and 2943 cm^{-1} which are attributed to the hydroxyl and thiol groups respectively. The broad nature of the bands indicates the existence of an intramolecular hydrogen bonding between the O-H and nitrogen atom of -CN groups (Chahmana et al., 2017). The disappearance of the absorption bands around 3255 and 2943 cm^{-1} attributed to O-H and S-H groups in the spectra of the complexes is due to deprotonation and involvement of the oxygen and sulfur atoms in the coordination to metal ion (Usharani et al., 2012; Chahmana et al., 2017), thus confirming the dentate nature of the ligand. It is further supported by the appearance of three new bands at 522–501 cm^{-1}, 575–555 cm^{-1}, and 468–454 cm^{-1} in the far-infrared spectra of the complexes, assigned to (M-N), (M-O) and (M-S), respectively (Usharani et al., 2012; Kanmani et al., 2016; Chahmana et al., 2017). In addition, the broad absorption peak observed at 3452 cm^{-1} in the Co(II) complex is attributed to O-H of the water molecule of crystallization.

According to the result above, there is bathochromic shift (Red Shift) in the NH functional group, while, hypsochromic shift (Blue shift) in CN functional group. The position shift of a peak to longer wavelengh with lower energy is bathochromic, and shorter wavelength with higher energy is called hypsochromic.

3.3 Solubility Test

Solubility test was carried out on Schiff base and metal complexes and it has shown that the complex compound were soluble in methanol, ethanol, distilled water but slightly soluble in acetone. However, in ligand it is soluble in methanol, ethanol, and acetone but insoluble in distilled water as shown in table 5 below:

Table 5: Solubility tests of Ligand and its Cu(II) complex.

Compound	**MeOH**	**EtOH**	**Distilled water**	**Acetone**
Ligand	S	S	IS	S
Cu(II) complex	S	S	S	SS

KEY: S= Soluble, SS= Slightly soluble, IS= Insoluble

4.0 CONCLUSION

The synthesized metal complex and Schiff base were characterized. From the FT-IR, it depicts the Schiff base ligand with amine and hydroxyl as donor atom arising from the coordination. The complex showed a square planer geometry as a result of shift in their structure. The result obtained validate the hypothesis that Schiff bases having substitution with halogens, hydroxyl group and nitro group at phenyl ring are required for the antibacterial ring activity while methoxy group at different positions in the aromatic ring has minimal role in the inhibitory activity. It serves as a precursor for the synthesis of antibacterial, anticancer, antioxidants, antifungal studies and catalysis in organic reactions.

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