Algerian Journal of Engineering and Technology

Journal homepage: https://jetjournal.org/index.php/ajet

Original Article

Synthesis and characterization of schiff base of 3-[(2-Hydroxy-phenylimino)-methyl]-6-methoxy-quinolin-2-ol and its metal complexes and their evaluation for antibacterial and antifungal activity

Abubakar Muhammad Jabbi*, Musa Husaini, Habu Nuhu Aliyu

Department of Pure and Industrial Chemistry, Faculty of Physical Sciences Bayero University Kano (BUK), P.M.B. 3011, Kano, Nigeria.

ARTICLE INFO

Article history:
Received 23 November 2019
Revised 10 January 2020
Accepted 29 January 2020

Keywords:
Schiff base;
metal complex of schiff base;
synthesis;
characterization;
antimicrobial activity;
antifungal activity.

ABSTRACT

A Schiff base of 3-[(2-Hydroxy-phenylimino)-methyl]-6-methoxy-quinolin-2-ol was synthesized by a 1:1 molar condensation of 2-Hydroxy-6-methoxy-3-quinolinecarboxaldehyde and 2-aminophenol. The metal (II) complexes were synthesized by refluxing the ethanolic solutions of the Schiff base and the chloride salts of the metals. Melting point, decomposition temperature, solubility, elemental analysis, fourier transform infrared spectroscopy, magnetic susceptibility and molar conductivity measurements were used to characterized the Schiff base and its metal complexes. The Schiff base is yellow and it has a melting point of 251 ^{O}C. The decomposition temperature of the Cd (II) and Cu (II) complexes were 282 and 270 ^{O}C respectively. The elemental analysis of the complexes established the formation of 1:1 metal - ligand ratio. The non-electrolytic natures of the complexes were revealed by the molar conductivity values. The behavior of the Schiff base and its coordination with the metal ions was suggested by the infrared spectral data via the azomethine nitrogen and hydroxyl oxygen after deprotonation. The solubility test of the Schiff base and its metal complexes were carried out by using different solvents. The antibacterial and antifungal activity were performed and discussed.

1. Introduction

Schiff bases are some of the most widely used organic compounds obtained as a result of condensation of primary amines and carbonyl compounds. Schiff bases were first discovered by a German chemist, Nobel Prize winner, called Hugo Schiff in the year 1864. Schiff bases are very important structures for synthetic organic chemistry. Schiff bases are usually characterized by an imine group (–N=CH-), that helps to clarify the mechanism, racemization and transamination interaction in biological system [1]. These compounds play an important role in medicinal fields because of their wide spectrum of biological activities. Many of them have antibacterial, antifungal and antitumor effect as biologically important molecules. A part from their biological effect, they are used in many fields such as pigments, dyes, fungicidal, corrosion inhibitors, analytical chemistry, polymer stabilizers, agrochemical, ion exchange, electrical conductivity catalysis, nonlinear optics and magnetism [2-5]. Schiff bases are changeable molecules and are generally bi- or tridentate ligands which are capable of producing a very stable complexes with transition metals such as nickel, cadmium, cobalt and copper etc. Most of the metal chelates have higher antimicrobial activity than the individual ligands. The biological implementations and chelating ability of metal complexes have attracted a significant concern [6, 7]. Metal complexes have been widely investigated due to their various biological applications in pharmacological areas. Some of the transition metal

* *Corresponding author.*
E-mail address abubakarmuhammadjabbi@gmail.com.
Peer review under responsibility of University of Echahid Hamma Lakhdar.
http://dx.doi.org/10.5281/zenodo.3945251

complexes have been reported to have antimicrobial activities. For example, complexes of copper showed various biological activities such as antifungal, antitumor, antimicrobial activities [8, 9]. Metal complexes having N, O as their donor atoms are very noticeable because of their important biological activities such as anticancer, and herbicidal activity. In this study the schiff base of 3-[(2-hydroxy-phenylimino)-methyl]-6-methoxy -quinolin-2- ol was synthesized from 2-aminophenol and 2-hydroxy-6-methoxy-3-quinolinecarboxaldehyde and its metal complexes where prepared. The schiff base and metal complexes were further characterized and their antibacterial and antifungal effect was also determined.

2. Materials and Methods

2.1. Chemicals, Reagents and Apparatus

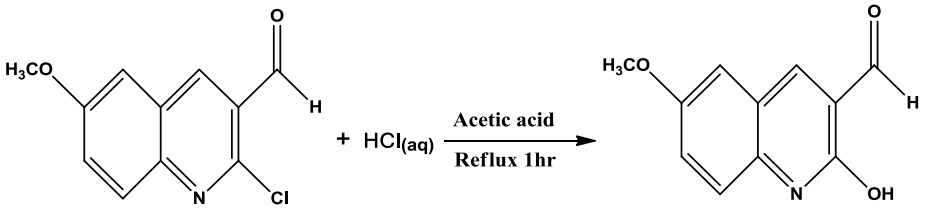
All the chemicals and reagents used in this research were of analytical grade and were used without any further purification. All the glass wares used were washed with detergent and were rinsed with distilled water and were dried in an oven. Electric weighing balance with model AB54 was used in measuring the masses of the reagents.

2.2. Synthesis of the Intermediate

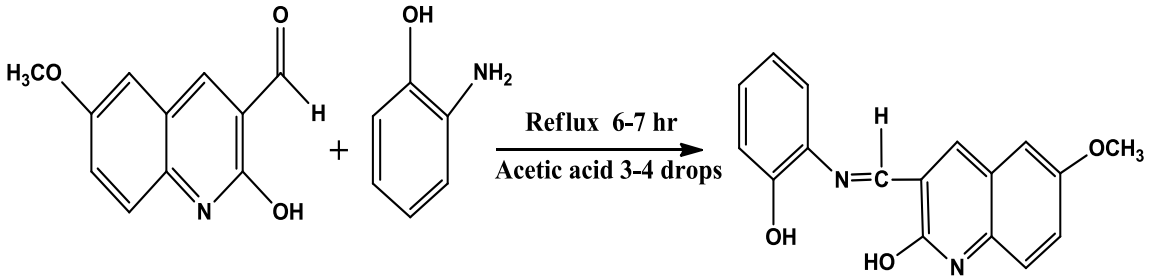
The method followed by Siddappa and Chandrakant [10], it was adopted here on which a mixture of 2-chloro-6-methoxy-3-quinolinecarboxaldehyde (2.4g, 0.01mol) and aqueous hydrochloric acid (35cm^3, 4mol) was heated under reflux on water bath at room temperature for about 1 hour and then it was cooled to room temperature. 2-Hydroxy-6-methoxy-3-quinolinecarboxaldehyde separated as solid was collected by filtration and recrystallized from aqueous acetic acid

2.3. Synthesis of the Schiff base Ligand

The ligand was prepared by the reaction of 2-Hydroxy-6-methoxy-3-quinolinecarboxaldehyde (0.01mol) with 2-aminophenol (0.01mol) in ethanol (35cm^3) by refluxing on water bath at room temperature for 6 – 7 hours in the presence of a few drops of acetic acid. The reaction mixture was cooled to room temperature; the separated ligand was filtered and washed from alcohol, after that it was recrystallized from into yellow silky cotton-like.



Scheme 1. Synthesis of 2-Hydroxy-6-methoxy-3-quinolinecarboxaldehyde



Scheme 2. Formation of 3-[(2-Hydroxy-phenylimino)-methyl]-6-methoxy -quinolin-2- ol

2.4. Synthesis of the metal (II) Schiff Base Complexes

According to Siddappa and Chandrakant [10] the hot solution of the Schiff base ligand (0.001mol) in ethanol (35cm^3) was added to a hot ethanolic solution of respective metal (II) chlorides (0.001mol) in ethanol (15cm^3). The reaction mixture was refluxed on a water bath for 4 to 5 hours to get clear solution. 0.5g of sodium acetate, CH_3COONa, was added to the reaction mixture to adjust the pH of the solution and the refluxing was continued further for 2 hours. The resulting reaction mixture was then decomposed by pouring into distilled water (90-100cm^3), the colored complex separated out was collected by filtration, washed with distilled water, then with hot ethanol and dried in vacuum over phosphorus pentoxide. Two metals (cadmium and copper) were used for the study, and they were obtained from their corresponding salt ($CdCl_2$ and $CuCl_2$).

2.5. Physical measurements

IR spectra of the Schiff base and its metal complexes were recorded on FTIR Carry Agilent Technologies model 630 spectrophotometer at 4000 - 650 cm^{-1} region in KBr powder. C, H and N were estimated by using elemental analyzer Perkin-Elmer model 240c. Jenway 4010 conductivity meter was used in conductivity measurement using DMSO as solvent. SMP10 STUART melting point apparatus was used to obtained Melting point and decomposition temperature. Magnetic measurements of the complexes were performed on Gouy's balance at room temperature.

2.6. Antibacterial and Antifungal Test

Jorgensen's method [11] was used in antibacterial test in which the schiff base and its metal (II) complexes were assayed by ager disc-diffusion method using *staphylococcus aureus*, *Escherichia coli*, and *salmonella typhi*. The samples were separately dissolved in dimethylsulfoxide (DMSO) to have three different concentrations (1000, 2000 and 3000) µg/disc. Each of these was separately placed on the surface and incubated at 37°C for 24 hours. The diameter of zone of inhibition produced by the Schiff base and its metal (II) complexes were taken and recorded. Similar procedure was applied in antifungal by using *Aspergillus flarus*, *Aspergillus niger* and *Mucor indicus* fungal isolates.

3. Result and Discussion

3.1 Physical Properties of the Schiff base and its Metal (II) Complexes

The Schiff base and its metal (II) complexes were prepared in good yield, the physical properties of the synthesized schiff base and its metal complexes were analyzed and presented in table 1. The percentage yield of the Schiff base was 75 % while that of the complexes were 61 and 66 %. The Schiff base was yellow cotton-like silky needles while the Cd (II), and Cu (II) complexes were deep-yellow and black respectively. It was found that the melting point of the Schiff base is 251^{O}C and the decomposition temperature of the metal (II) complexes are 282 and 270^{O}C, this is an indication of thermal stability.

The molar conductance of the metal complexes are 11.69 and 10.76 $\Omega^{-1}cm^2mol^{-1}$ for Cd (II) and Cu (II) complexes. These low values of their molar conductance due to their non-electrolytic nature as reported by Eman [12]. The effective magnetic moments of the complexes were also calculated. The magnetic moment of Cu (II) complex is 1.75 B.M, indicate that this complex is paramagnetic in nature, while the Cd (II) complex with 0 B.M indicate that this complex is diamagnetic in nature.

Table 1: Physical Properties of the Schiff base and its Metal (II) Complexes

Compounds	Colour	% yield	M.P (^{O}C)	D. Temp (^{O}C)	μ_{ff} (B.M.)	M.L($\Omega^{-1}cm^2$ mol^{-1})
L	Yellow	75	251	-	-	-
$[CdL(H_2O)_3].H_2O$	Deep - yellow	61	-	282	0	11.69
$[CuL(H_2O)_3].H_2O$	Black	66	-	270	1.75	13.07

3.2 Solubility Test

Water and some common organic solvents were used to determine the solubility of the Schiff base and its metal (II) complexes. From the result of solubility test presented in table 2, it can be seen that, the Schiff base and its metal complexes were soluble in dimethylsulfoxide (DMSO) and dimethylformamide (DMF), slightly soluble in ethanol and insoluble in n-hexane and water.

Table 2: Solubility test of the Schiff base and its metal (II) complexes

Compound	Solvent						
	Acetone	n-hexane	CCl4	DMF	DMSO	Ethanol	water
L	SS	IS	S	S	S	SS	IS
$[CdL(H_2O)_3].H_2O$	SS	IS	SS	S	S	SS	IS
$[CuL(H_2O)_3].H_2O$	SS	IS	SS	S	S	SS	IS

3.3 Elemental Analysis

The elemental analysis of the Schiff base and its metal (II) complexes were determined and presented in table 3. The calculated and observed values were found to be in good agreement. The elemental analysis data of the Schiff base suggested the formation of $C_{17}H_{14}N_2O_3$ while that of the complexes revealed the formation of $[CdL(H_2O)_3].H_2O$ and $[CuL(H_2O)_3].H_2O$. The complexes are formed in 1:1 metal-ligand ratio. This is similar to the work done by Abubakar [13]

Table 3. Elemental Analysis Data of the Schiff base and its Metal (II) Complexes

Compound	Percentage Found (Calculated)			
	% N	% C	% H	% M
L	8.90	64.39	4.18	-
$[CdL(H_2O)_3].H_2O$	5.81 (5.88)	42.59 (42.83	3.73 (4.23	23.58
$[CuL(H_2O)_3].H_2O$	5.97 (6.04)	39.44 (40.01)	4.78 (5.21)	13.70

3.4 FTIR Analysis

The FTIR results of the Schiff base and metal complexes together with their spectral data are presented in figures 4, 5 and 6 and table 4. There is a band in the infrared spectrum of the Schiff base due to the phenolic *v*(OH) stretching vibration at 3339 cm^{-1}. This band disappeared in the spectra of the complexes due to deprotonation and involvement of the oxygen atom in complexation. Similar result was reported by Abdullahi and Gareth [14]. The broad band at 3394 and 3320 cm^{-1} in the spectra of the complexes are attributed to water of hydration as reported by El-ajaily *et al* [15]. The band at 1622 cm^{-1} in the free schiff base is assigned to the *v*(C=N) stretching vibration. This band shifted towards lower frequencies of 1581 and 1562 cm^{-1} in the complexes, and this suggested the participation of the nitrogen atom of the azomethine in coordination. The *v*(C-O) phenolic stretching of the Schiff base is observed at 1383 cm^{-1} which shifted to lower frequencies of 1264 and 1272 cm^{-1} in the complexes. This is an indication of coordination through the phenolic oxygen as reported by Mounika *et al* [16]. The Schiff base coordination with the metals is further evidenced by the appearance of weak low frequency non ligand bands at 736 and 851 cm^{-1} due to *v*(M-N) stretching vibration, and at 687 and 762 cm^{-1} due to *v*(M-O) stretching vibration. Similar result was reported by Rasha and Farah [17].

Table 4. Infrared spectral data of the Schiff base and its metal (II) complexes

Compound	*v*OH (cm^{-1})	*v*H_2O(cm^{-1})	*v*C=N (cm^{-1})	*v*C-O(cm^{-1})	*v*M-N (cm	*v*M-O (cm^{-1}
L	3339	-	1622	1383	-	-
$[CdL(H_2O)_3].H_2O$	-	3394	1581	1264	736	687
$[CuL(H_2O)_3].H_2O$	-	3320	1562	1272	851	762

Key: L = $C_{17}H_{14}N_2O_3$

3.5 Antimicrobial and antifungal Activity

The result of antimicrobial activity of the Schiff base and its metal complexes is presented in table5 . The Schiff base and its metal (II) complexes were screened for their antibacterial activities against the selected bacteria isolates of *Staphylococcus aureus, Eschirichia coli,* and *Salmonella typhi* by disc diffusion method. It was found that metal complexes have more effect in inhibiting the microbial growth. This may be due to the interaction of the metal complexes with lipoproteins of the cell. Therefore the metal complexes may restrict the normal functioning of the microbial cell. Similar result was reported by Yahyazadeh and Azimi [18]. Furthermore, their higher stability at higher temperature may also allow to use them as a potential antimicrobial agent. Similar result was also recorded in table 6 for antifungal activity shown by selected fungi isolates of *Aspergillus flarus, Mucor iudicus,* and *Asphergillus niger* pres.

Table 5. Antibacterial activity on the schiff base and its metal (II) complexes

Compound	**Concentration (μgcm^{-3})**	**Bacterial inhibition zones in mm**		
		Staphylococcus aureus	Eschirichia coli	Salmonella typhi
L	1000	07	09	08
	2000	06	08	10
	3000	09	07	09
$[CdL(H_2O)_3].H_2O$	1000	09	10	11
	2000	11	13	13
	3000	13	18	16
$[CuL(H_2O)_3].H_2O$	1000	11	11	10
	2000	12	15	15
	3000	16	18	14

Key: L = $C_{17}H_{14}N_2O_3$

Table 6. Antifungal activity on the schiff base and its metal (II) complexes

Compound	Concentration (μgcm^{-3})	Fungal inhibition zones (mm)		
		Aspergillus flarus	Mucor iudicus	Asphergillus niger
L	1000	06	03	04
	2000	08	05	06
	3000	09	07	07
$[CdL(H_2O)_3].H_2O$	1000	10	11	12
	2000	12	16	15
	3000	20	20	19
$[CuL(H_2O)_3].H_2O$	1000	11	09	11
	2000	15	12	14
	3000	18	17	17

Key: L = $C_{17}H_{14}N_2O_3$

4. Conclusion

The Schiff base and its metal (II) complexes were synthesized and characterized. The non-electrolytic nature of the complexes was revealed by conductivity measurement data. The elemental analysis data suggest 1:1 metal to ligand ratio. The infrared data indicated that the Schiff base ligand was coordinated to the central metal ion in a tridentate manner via the azomethine nitrogen and phenolic oxygen atoms after deprotonation. The solubility tests carried out showed that the ligand and both of its metal (II) complexes were soluble in dimethylformamide and dimethylsulphoxide, slightly soluble in acetone and ethanol and insoluble in water and n-hexane. The ligand and its metal (II) complexes were tested for antimicrobial and antifungal activity against some bacteria and fungi isolates. Both of the complexes were found to be more active against the bacteria *and* fungi than the Schiff base.

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Recommended Citation

Jabbi AM, Husaini M., Aliyu HN. Synthesis and characterization of schiff base of 3-[(2-Hydroxy-phenylimino)-methyl]-6-methoxy-quinolin-2- ol and its metal complexes and their evaluation for antibacterial and antifungal activity. *Alg. J. Eng. Tech*. 2020; 2: 029-036. http://dx.doi.org/10.5281/zenodo.3945251

5. Supplemented data

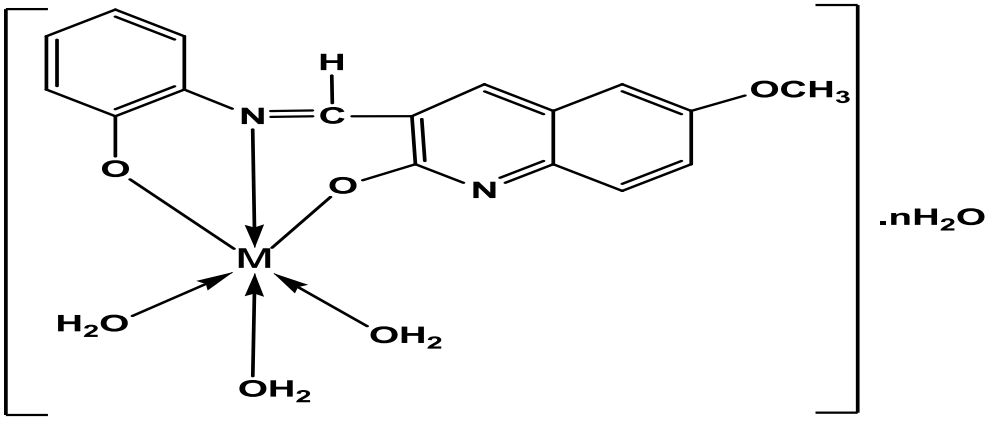

Fig 1. Proposed structure of Metal (II) Complex

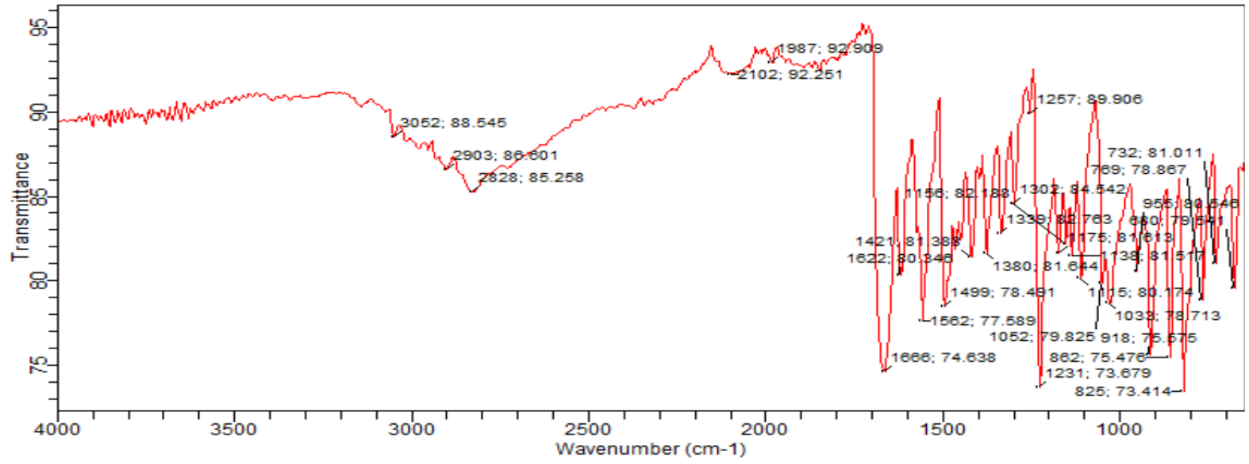

Fig 2. FTIR of the 2-Hydroxy-6-methoxy-3-quinolinecarboxaldehyde

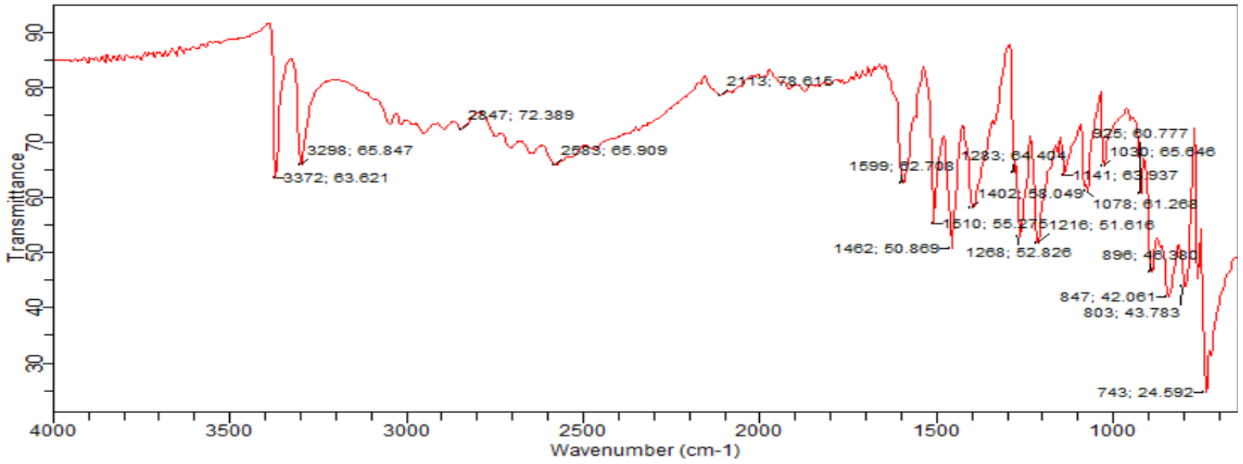

Fig 3. FTIR of 2-Aminophenol

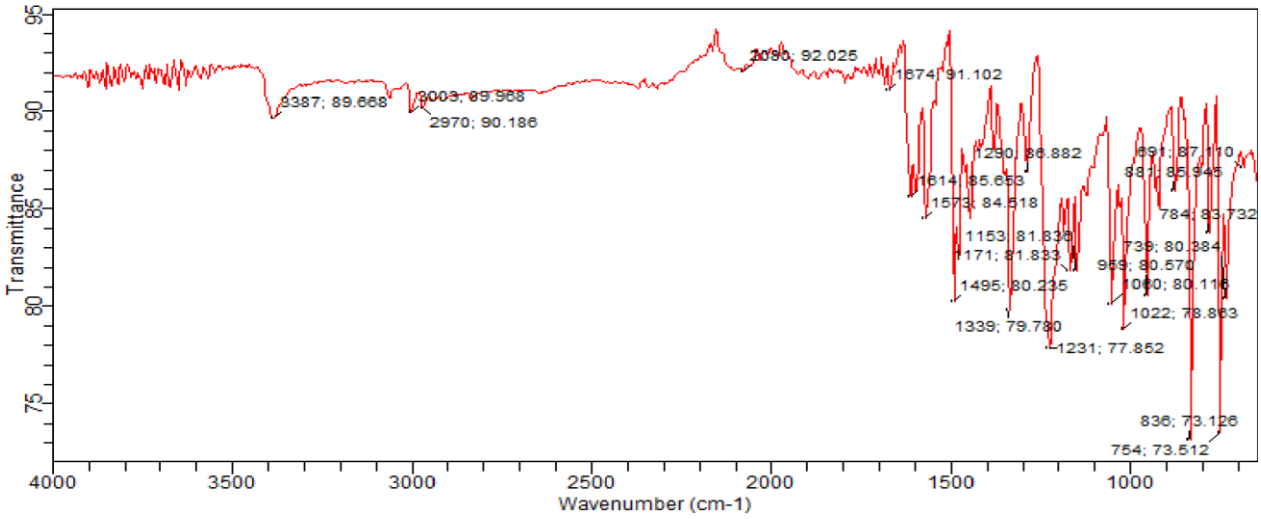

Fig 4. FTIR of Schiff base Ligand

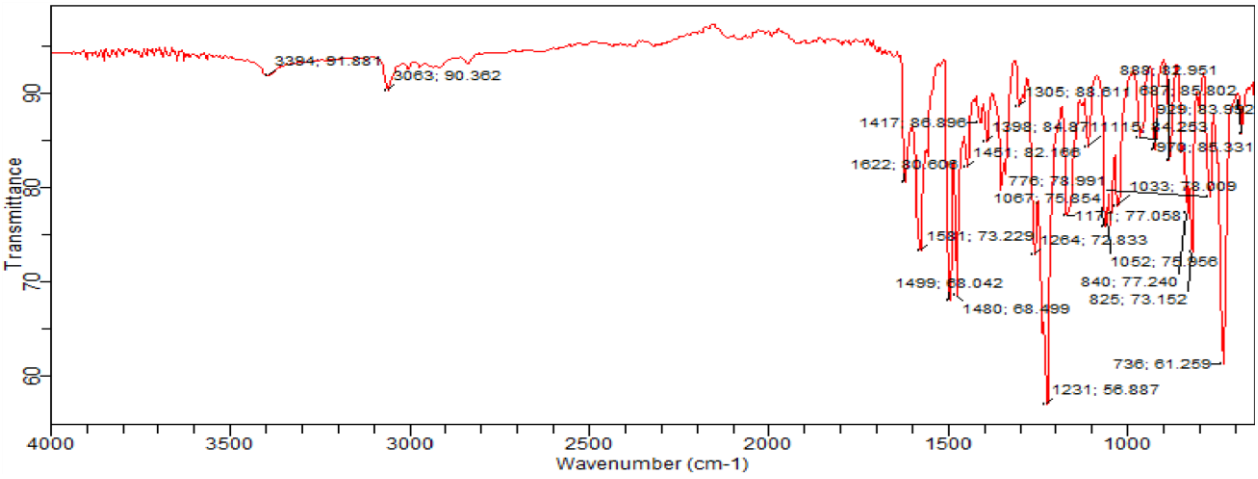

Fig 5. FTIR of Cd (II) complex

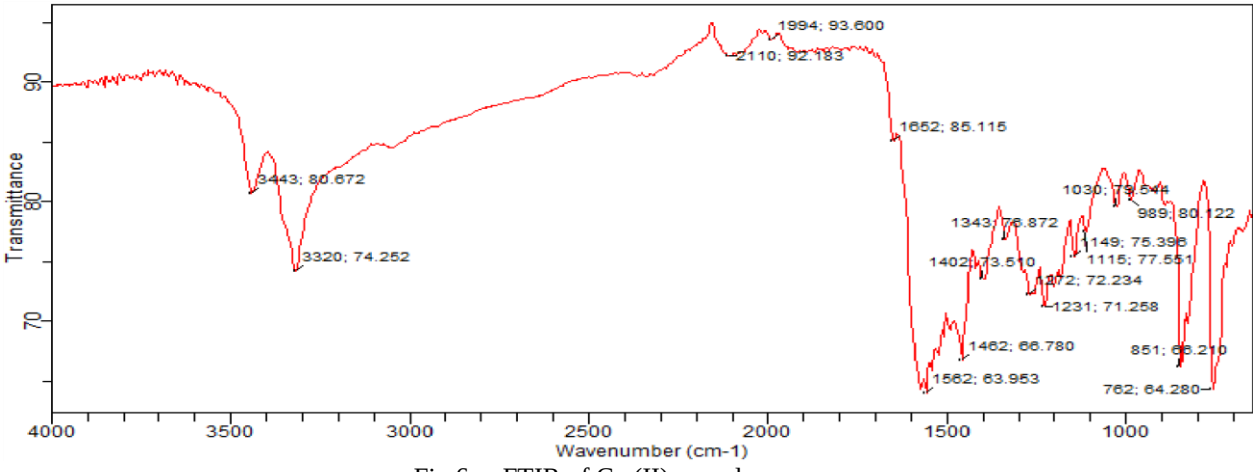

Fig 6. FTIR of Cu (II) complex