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Antioxidant Evaluation of Co(II) and Zn(II) Complexes with Schiff base derived from 2-hydroxy-1-naphthaldehyde and p-methoxyaniline

Ibrahim, A.K.,[1]*, Na'aliya, J.[2] and Haruna, A [3]
[12]*Department of pure and Industrial Chemistry, Bayero University, Kano. Nigeria*
[2]*Department of Chemistry, Federal University Gusau. Nigeria*
Corresponding Author: Ibrahim, A.K., E-mail: al.ameen91@yahoo.com

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ABSTRACT

The Metal Complexes of Co(II) and Zn(II) with Schiff base derived from 2-hydroxy-1-naphthaldehyde and p-methoxyaniline were synthesized and characterized using elemental analysis, conductivity measurement, magnetic susceptibility, infrared spectral analysis and solubility test. The Schiff base and its corresponding metal complexes were evaluated for antioxidant activity. The molar conductance values range (6.62-7.80Ω-1cm2mol-1) indicated the non electrolytic nature of the complexes. The magnetic susceptibility values revealed that Co(II) complex is paramagnetic while Zn(II) complex is diamagnetic.. The infrared spectra analysis suggested that the Schiff base behave as a bidentate ligand. The elemental analyses results revealed slight differences between observed and calculated percentages of C, H, and N in the Schiff base and metal complexes, the metal-ligand ratio was found to be 1:2 in all the complexes. The antioxidant activity of Schiff base and its complexes was measured on the basis of the radical scavenging effect of 1,1-diphenyl-2-picryl-hydrazyl (DPPH)-free radical activity. The results revealed that the Schiff base and its metal complexes exhibited excellent antioxidant properties.

1. Introduction

The chemistry of the carbon-nitrogen double bond plays a vital role in the progresses of chemical science. Schiff bases also called as imines, are characterized by the presence of the azomethine functional group (-C=N-), and are usually formed by condensation of carbonyl compound (aldehyde or Ketone) with a primary amine (Iniama and Isaac, 2013). The first reports of this kind of reaction have been published by Hugo Schiff in the 1860s. Thereafter Schiff bases have been intensively used as synthetic intermediates and as Ligands for coordinating transition and inner transition metal ions, and recently also for coordinating anions (Anita et al., 2010). The Schiff base Ligands may act as bidentate, tridentate, tetradentate, hexadentate Ligands, *etc*., which can be designed to yield mononuclear or binuclear complexes or one-dimensional, two-dimensional and three-dimensional metal-organic frameworks (Anita *et al.,* 2010).

Schiff base Ligands may contain a variety of substituent with different electron-donating or electron-withdrawing groups, and therefore may have different interesting chemical properties. Schiff bases have been widely used as ligand because of high stability of their coordination compounds, good solubility in common solvents such as ethanol, methanol, chloroform and Dimethylformamide e.t.c (Bharat *et al.,* 2015), relevant biological application, high flexibility and medicinal efficacy (Jai, *et al.,* 2016). Schiff bases obtained from aromatic aldehyde and aromatic amines have shown a number of applications in many fields such as pharmaceutical, life sciences and chemical science including inorganic and analytical chemistry (Muzammil *et al.,* 2015). These important compounds have been reported to possess diverse biological activities such as antifungal, analgesic, anti-inflammatory, antibacterial, antioxidant, antitubercular and antitumor, (Neelofar *et al.,* 2017).

Metal complexes have been receiving considerable attention for many years, due to their interesting characteristics in the field of material science and biological system (Rajendra, *et al.,* 2012). Metal complexes have important application in medicinal chemistry. Medical science demands such types of compounds which are more potent, biologically active, easily absorbable and nontoxic, and show fast action for treatment of diseases (Ibrahim *et al.,* 2017).
Extensive studies revealed that chelation makes the complex more stable and biologically more active in the presence of bio-metal. Metal ions fixed the complexes at the specific active site of the proteins and enzymes of the host and show their potentiality (Chaudhary, 2013).

2. Literature review

The Gomathi *et al*., (2013) reported the synthesis of new coordination complexes of Mn(II) and Zn(II) from Schiff base derived from 2-hydroxy-1-naphthaidehyde and p-toludine. The nature of bond and the structural features of the Schiff base and complexes have been deduces from elemental analysis, molar conductance measurement, magnetic susceptibility, IR, ^{1}H NMR, UV- visible and cyclic voltammetry studies.

Complex of cerium(III) with (E)-N-benzylidene-4-methoxyaniline is synthesized through a one-pot three-component reaction from aromatic aldehyde, aromatic amine and the $CeCl_3 \cdot 7H_2O$, as an efficient catalyst. This Ce(III) complex is characterized by IR, ^{1}H, and ^{13}C NMR-spectroscopy and mass-spectral data. Molecular structure, Mullikan charges, thermodynamic parameters; vibrational frequencies and intensities were calculated by Density Functional theory methods using the SDD basis set. The comparison between the calculated and experimental data in order to attain the best quality and to predict the structure, the best performance in the vibration spectra perfected of the title compound, and it was found that the harmonic vibration computed are in a good agreement with the observed in IR spectrum (Lakehala *et al.,* 2016).

Siddappa and Nabiya (2014) Prepared Schiff base of bis-hydrazones by the condensation of isatin mono-hydrazones with 2-hydroxyquinoline-3-carbaldehyde, which formed a series of complexes with Co(II), Ni(II), Cu(II), Zn(II), Cd(II) and Hg(II). The ligand and its metal complexes were characterized on the basis of elemental analysis, conductance data, magnetic susceptibility measurements, IR, UV-Vis, ^{1}H NMR, ESR and XRD studies. By these spectral studies it is found that Co(II), Ni(II) and Cu(II) complexes have been assigned octahedral geometry whereas the Zn(II), Cd(II) and Hg(II) complexes have been assigned tetrahedral geometry. The Schiff base and its metal complexes have been screened for their *invitro* antibacterial, antifungal and antioxidant activity.

In another development a chromone Schiff base complexes of Zn(II), Cu(II) , Ni(II) and Co(II) were successfully prepared in nano domain with crystalline or amorphous structures. The spectroscopic data revealed that the Schiff base behaves as a monoanionic tridentate ligand. The metal complexes exhibited octahedral geometry. Transmission electron microscope (TEM) analyses showed that Cu(II) complex have aggregated nanospheres morphology. The obtained nano-complexes were tested as antioxidant and antitumor agents. The Schiff base and its Cu(II) complex were found to be more potent antioxidant (IC_{50}(ligand) = 0.93mM; IC_{50}(Cu(II) complex) = 1.1mM than standard ascorbic acid (IC_{50} = 2.1mM) as evaluated by DPPH method. Also the Schiff base and its complexes were tested for the *in vitro* cytotoxicity against Ehrlich Ascites Carcinoma cell line (EAC), the Cu(II) nano-complex effectively inhibited EAC growth with IC_{50} value of 47mM in comparison with its parent compound and other prepared complexes. (Saif *et al*., 2016)

In another report the Co(II), Ni(II), Cu(II), and Zn(II) complexes of (4*E*)-4-[(2-{(*E*)-[1-(2,4 dihydroxyphenyl)-ethylidene]-amino}-ethyl)-imino]-pentan-2-one have been synthesized and characterized by elemental analysis, molar conductance, FT- IR spectral studies, and XRD. FT-IR confirmed the ligand coordinates to metal ion via the oxygen and nitrogen atoms of the phenolic group and azomethine group, respectively. Tetrahedral geometry is proposed for Co(II) complex and square-planar geometry for Ni(II) and Cu(II) complexes. The antibacterial studies of the compounds were determined and its show that the metal complexes are more active than the free Ligands. The antioxidant activity was examined using DPPH free radical scavenging method, and the result shows Cu(II); IC_{50} = 2.31 ± 1.54 μM and Co(II); IC_{50} = 1.83 ± 1.08μM respectively. (Ikechukwu P. Ejidike and Peter A. Ajibade, 2015).

Abhijit *et al.,* (2014) reported the evaluation of the biological activities of metal complexes of Indomethacin with cobalt, copper, manganese and zinc. In radiant tail flick method, complexes of Indomethacin with cobalt and copper at a dose of 20mg/kg showed significant central analgesic activity having 66.09% and 75.45% elongation of time after 30 minutes and complexes of Indomethacin with copper at a dose of 20mg/kg showed significant central analgesic activity having 62.47% elongation of time after 60 minutes compared to the standard morphine. In this study, Indomethacin and its complexes with

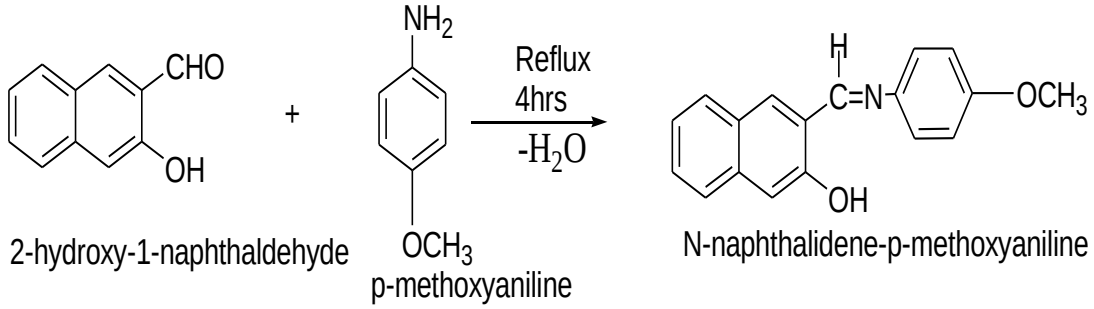
cobalt, copper and manganese showed mild antimicrobial activity and the Indomethacin manganese complex also displayed highest cytotoxicity with a lowest LC_{50} of 1.222 ± 0.2µg/ml and Indomethacin-cobalt, Indomethacin-copper and Indomethacin-zinc had significant LC_{50} of 1.549 ± 0.39µg/ml, 1.662 ± 0.17µg/ml and 1.903 ± 0.64µg/ml, respectively where standard vincristine sulphate had LC_{50} of 0.824 ± 0.04µg/ml. The complex of Indomethacin with cobalt, copper, manganese and zinc revealed % of inhibition 38.46 ± 1.03, 64.31 ± 0.21, 46.71 ± 0.46 and 30.79 ± 0.30, respectively and also had significant IC_{50} of 17.51 ± 0.62µg/ml, 12.31 ± 0.58µg/ml, 15.71 ± 0.16µg/ml and 19.84 ± 0.08µg/ml correspondingly. The study indicates that the complexes of Indomethacin had analgesic, antimicrobial, Cytotoxic and antioxidant activities which could be subjected for further therapeutic evaluation.

3. Methodology

All chemicals were purchased from Sigma Aldrich and used without further purification. All glass wares used were washed with detergent after soaking in conc. HNO_3, rinsed with distilled water and dried in an oven. Weighing was conducted using electrical Melter balance model AB54. Infrared spectral analysis was determined using Fourier transform infrared spectrophotometer (FTIR-8400S) range 4000-400cm^{-1}. Electrical conductance was measured using Jenway conductivity meter model 4010 range 20-200µs. Melting points and decomposition temperature were determined using microprocessor melting point apparatus (WRS-IB). Magnetic susceptibility was determined using magnetic susceptibility balance MKI Sherwood scientific ltd. Elemental analyses were determined using Series II CHNS/O 2400 Perkin Elmer. All chemicals used in this work were analytical grade and were used without further purification.

Preparation of Schiff base

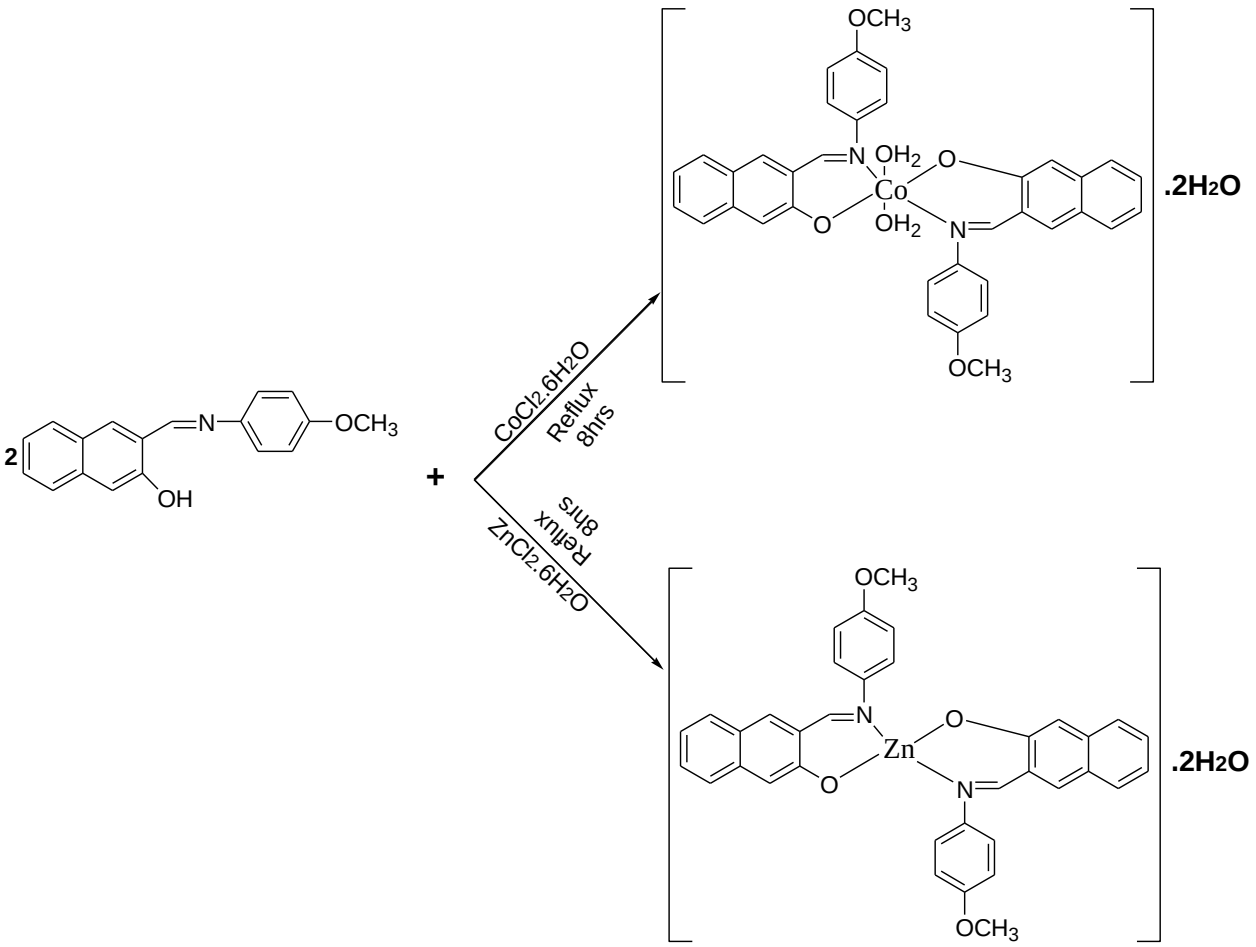
The Schiff base was prepared by mixing ethanolic solution of 2-hyroxy-1-naphthadehyde (4mmol) 25ml with that of ethanolic solution of p-methoxyaniline (4mmol) 25ml. The resulting solution was refluxed for 4hours and then cools to room temperature; on cooling the precipitate formed. Then the solid was filtered, washed and recrystallise with ethanol and diethyl-ether and then dried in desiccators over anhydrous $CaCl_2$ to obtain the required Schiff base (Gomathi, *et al.,* 2013)



Scheme1: Preparation of Schiff Base

Preparation metal complexes

The complexes were prepared according to literature reported by Gomathi, *et al.,* 2013, by mixing hot ethanolic solution (4mmol) 25ml of Schiff base ligand with hot ethanolic solution of (2mmol) 25ml of metal salts. The resulting mixture was reflux for 8hrs, the complex obtained in each case was cool to room temperature, filtered and washed with ethanol and diethyl-ether several times to remove any excess ligand. Finally the complex was dried each over anhydrous $CaCl_2$ in desiccators. The metal salts include Co(II) and Zn(II) salts respectively.



Scheme2: Preparation of Complexes

Determination of Melting point of Schiff base and Decomposition Temperature of the metal complexes

The melting point of Schiff base and the decomposition temperature of metal complexes were determined using microprocessor melting point apparatus (WRS-IB). The results obtained are shown in Table.1 (Ibrahim *et al.,* 2018)

Solubility Test

The solubility test of Schiff base and its metal complexes were carried out in water, ethanol, methanol, acetone, and chloroform, Dimethylsulfoxide, Dimethylformamide and Diethyl-ether in which 0.2g of each sample was tested in 10ml of each solvent. The results obtained are shown in table.2 (Yusuf *et al.,* 2018)

Determination of Water of Hydration in the Complexes

0.2g of prepared complex each was placed in a clean weighted Petri dish which was then placed in an oven at 110^{o}C for 72hrs, until a constant weight was obtained.

The weight loss if any, recorded as water of hydration from the constant weight of anhydrous complex; the percentage water of hydration was calculated for each complex using the expression below. (Ibrahim *et al.,* 2017)

$$\%\ \text{water of hydration} = \frac{\text{weight loss}}{\text{initial weight of sample}} \times 100\%$$

Molar conductance measurements

1mmol of each complex was dissolved in 10ml of Dimethylsulfoxide (DMSO) and the corresponding specific conductance value was recorded using Jenway conductivity meter model 4010. (Moamens, 2013)

From the specific conductance value recorded, the molar conductance of each metal complex was calculated using the expression below. The results obtained are shown in Table4.3.

$$\text{Molar conductance} = \frac{100 \times \text{specific conductance}}{\text{ionic concentration}}$$

Magnetic Susceptibility Measurement

The magnetic susceptibility of complexes was determined using magnetic susceptibility balance MKI Sherwood science ltd via the expression below. The results obtained are shown in Table4.5 (Javed, 2006).

$$\text{Xg} = \text{CL}\,\frac{(\text{R} - \text{Ro})}{10^9\text{M}}$$

Where Xg = Mass susceptibility, C = 1 (Constant), L = Sample length in the tube (whose range should be set between 1.5 to 3.5cm, R = Reading obtained from the sample packed in the tube, R_o = Reading obtained from preweight empty tube, M = mass of the sample in the tube (measured in gram).

Antioxidant experiments

The method used by Saif *et al*., (2016) was adopted with little modifications. DPPH (8 mg) was dissolved in methanol (300mL). Series dilutions were carried out with stock solutions (4 mmol) of free ligand and its metal complexes in methanol to obtain concentrations of 2.0-0.05 mmol. Diluted solutions (2mL each) were mixed with DPPH (2mL) and allowed to stand for 30 min, for any reaction to occur. The absorbance was recorded at 517nm using a JASCO model V-550 UV-Vis spectrophotometer when the odd electron becomes paired off in the presence of a free radical scavenger, the absorption reduces and the DPPH solution is decolorized as the color changes from deep violet to light yellow. The degree of reduction in absorbance measurement is indicative of the radical scavenging (antioxidant) activity. The experiment was performed twice and the average absorbance was noted for each concentration of the test compound that reduced 50% of the initial free radical concentration, which was calculated as µmol. Ascorbic acid was used as reference standards. Control sample was prepared containing the same volume without test and reference compounds. The radical scavenging activity of the tested samples, expressed as percentage inhibition of DPPH, was calculated according to the formula I(%) = [(Ao-At)/Ao]x100, where At is the absorbance value of tested sample and Ao is the absorbance value of blank sample, in particular time. The linear regression fitting between the % inhibition and log concentration was determined by probit analysis using IBM SPSS statistic 20.0 software. And the concentration corresponding to 50% inhibition was expressed as IC_{50} value. A lower IC_{50} value indicates greater antioxidants activity.

4. Results Discussion

Table1: Physical and Analytical Data of Schiff base and its metal Complexes

Compound	Colour	Mol.Formula	Mol.wt ($gmol^{-1}$)	Melt.pt/Dec. Temp.(^oC)	%Yield	Elemental analysis Calculate (Found)		
						%C	%H	%N
Schiff base	Yellow	$C_{18}H_{15}NO_2$	277	112	68.59	77.98 (74.56)	5.42 (3.93)	5.05 (6.24)
$[CoL_2(H_2O)_2].2H_2O$	Brown	$C_{36}H_{36}CoN_2O_8$	682.4	201	72.90	63.26 (62.83)	5.27 (3.64)	4.10 (4.70)
$[ZnL_2].2H_2O$	Yellow	$C_{36}H_{32}ZnN_2O_6$	653.4	215	67.33	66.12 (66.62)	4.90 (5.05)	4.29 (4.20)

Key: L = Ligand

Table.2: Solubility of the Schiff base and the Complexes in some common Solvents

Compound	Water	Methanol	Ethanol	Chloroform	Acetone	DMF	DMSO
Schiff base	IS	S	SS	S	S	S	S
$[CoL_2(H_2O)_2].2H_2O$	IS	S	SS	S	S	S	S
$[ZnL_2].2H_2O$	IS	S	SS	S	S	S	S

L = Ligand, DMSO = Dimethylsulfoxide, DMF = Dimethylformamide, IS = Insoluble, SS = Slightly soluble, S = Soluble

Table 3: IR Spectra of the Schiff base and metal Complexes

Compound	V(C=N) cm^{-1}	V(M-O) cm^{-1}	V(M-N) cm^{-1}	V(H_2O) cm^{-1}
Schiff base	1614	-	-	-
$[CoL_2(H_2O)_2].2H_2O$	1607	604	514	3515
$[ZnL_2].2H_2O$	1607	644	504	3484

Key: L= Ligand

Table 4: Conductivity Measurement of Complexes in DMSO

Complex	Concentration Moldm^{-3}	Specific Conductance Ohm^{-1} cm^{-1}	Molar Conductance Ohm^{-1} cm^2 mol^{-1}
$[CoL_2(H_2O)_2].2H_2O$	1.0×10^{-3}	6.62×10^{-6}	6.62
$[ZnL_2].2H_2O$	1.0×10^{-3}	7.80×10^{-6}	7.80

Key: L= Ligand, DMSO= Dimethylsulfoxide

Table 5: Magnetic Susceptibility of the Complexes

Compound	Xg(gmol^{-1})	Xm(gmol^{-1})	$\mu_{eff\ (BM)}$	Property
$[CoL_2(H_2O)_2].2H_2O$	1.52×10^{-5}	1.04×10^{-2}	4.98	Paramagnetic
$[ZnL_2].2H_2O$	-2.07×10^{-7}	-1.35×10^{-4}	-Ve	Diamagnetic

Key: L = Ligand

Table 6: In vitro DPPH radical scavenging activity of Schiff base and its metal Complexes

Compound	DPPH Scavenging activity IC_{50} (μmol)
Schiff base	0.496
$[CoL_2(H_2O)_2].2H_2O$	0.016
$[ZnL_2].2H_2O$	6.858
Ascorbic acid (Standard)	0.350

Key: L = Ligand, DPPH = 1, 1-diphenyl-2-picrylhydrazyl

5. Discussion

The reaction between 2-hydroxy-1-naphthaldehyde and p-methoxyaniline yielded Schiff base ligand (N-naphthalidene-p-methoxyaniline) which is yellow solid with the percentage yield of 68.59% and melting point temperature of 112°C. (Table.1) this is in agreement with the colour and closer in melting point reported by Gomathi *et al.,* (2013).

The reaction between Schiff base and hydrated Co(II) and Zn(II) salt formed complexes with different texture and colours (Table.1) the colours may be due to charge transfer or nature of the ligand. The decomposition temperature of the prepared Complexes fall in the range of 210°C-215°C respectively (Table.1), these high temperature indicated the good stability of the Complexes due to coordination between the Schiff base and the metal ion and bulky nature of the Complexes, this is closer to the results reported by Ibrahim *et al.,* (2017). Also the elemental analysis data revealed slight differences between the calculated and observed percentage values of CHN respectively (Table.1). These values are in good agreement with the proposed stoichiometry of the prepared complexes.

The Schiff base and its metal complexes are soluble in some common organic solvents such as methanol, acetone, chloroform, Dimethylsulfoxide (DMSO) and Dimethylformamide (DMF), and slightly soluble in ethanol and insoluble in water and Diethyl-ether. (Table.2).

The values obtained in the spectrum of the Schiff base showed a band at 1614cm^{-1} which is attributed to V (>C=N-) stretching vibration and this band shifted to lower region (1607cm^{-1}) in the spectra of the Co(II) and Zn(II) complexes respectively. (Table.3) indicating the chelation of the Schiff base to the metal through nitrogen atom of azomethine group and phenolic oxygen. The new bands appeared in the spectra of the complexes in the ranges (604-644cm^{-1}) and (504-514cm^{-1}), these are attributed to V(M-O) and V(M-N) stretching vibration respectively, (Table.3) also indicating the coordination of the Schiff base ligand to the metal(II) ion. Another bands appeared in the spectra of the complexes in the region range (3484-3515cm^{-1}) which may be attributed to V(H_2O) stretching vibration. These results are in good agreement with the previous literatures

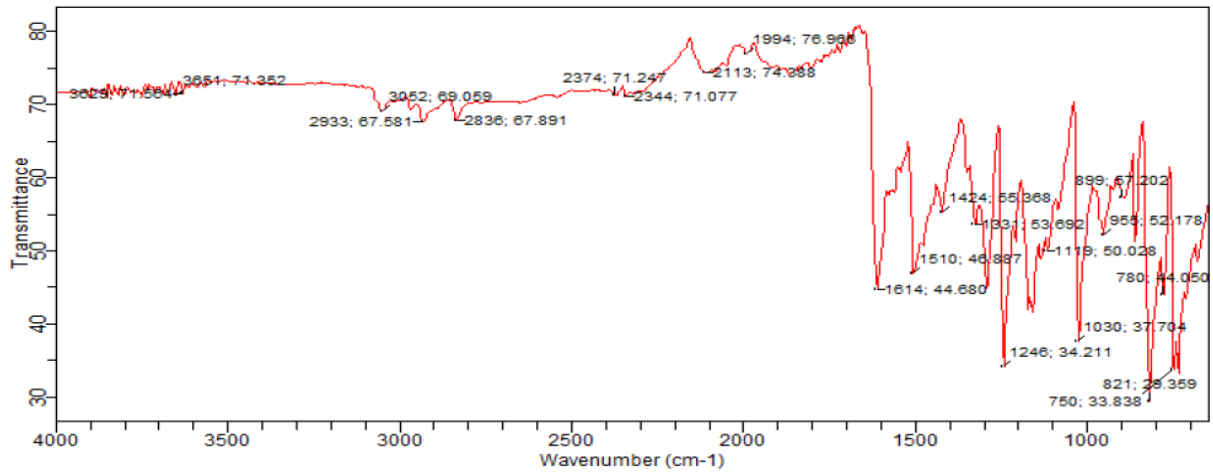


Figure1: IR Spectrum of Schiff base

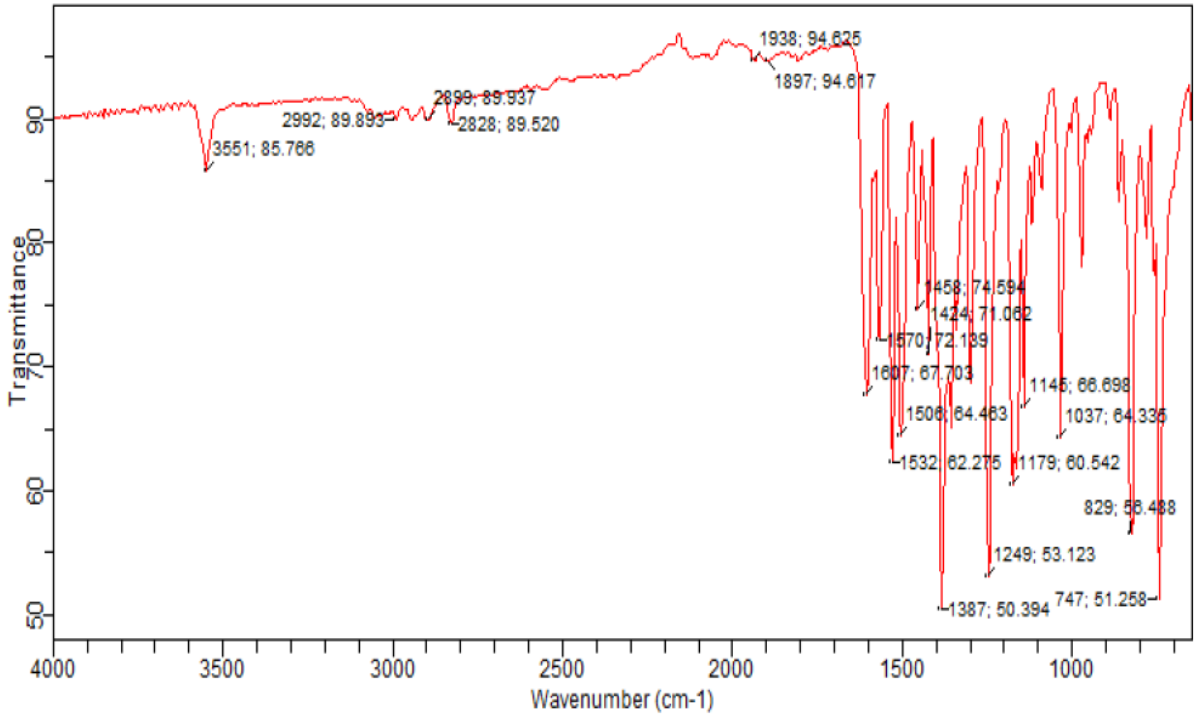


Figure 2: IR Spectrum of Co(II) Complex

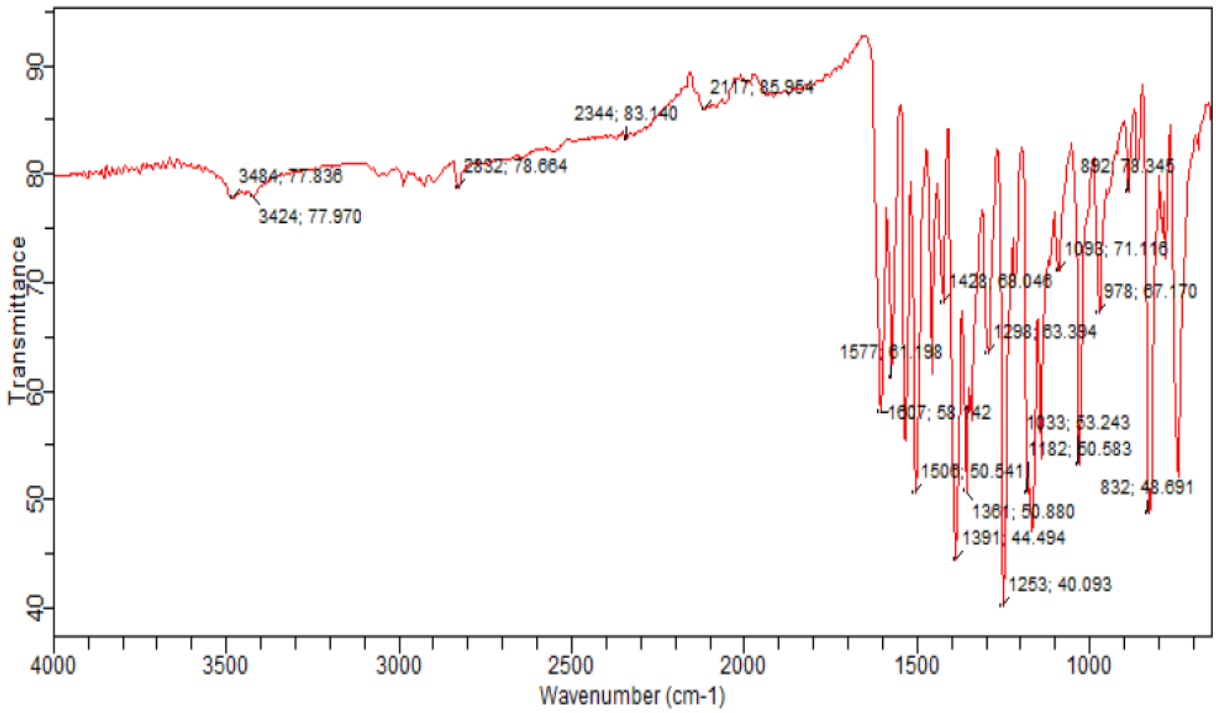


Figure 3: IR Spectrum of Zn(II) Complex

The molar conductance values range (6.62-7.80$\Omega^{-1}cm^2mol^{-1}$) indicated both Co(II) and Zn(II) Complexes are non electrolytes. (Table.4) and these values are very close to the values reported by Uddin *et al*., (2014).

The values obtained from magnetic susceptibility measurement of the prepared complexes at room temperature i.e.; 4.98BM indicated Co(II) complex as paramagnetic while –Ve indicated Zn(II) as diamagnetic. (Table.5)

The result obtained from DPPH scavenging activity of Schiff base and its metal complexes shows IC_{50} = 0.496 for Schiff base, IC_{50} = 0.016 for Co(II) complex and IC_{50} = 6.858 for Zn(II) complex (Table.6), these values indicated excellent antioxidant properties of the prepared compounds.

6. Conclusion

The Schiff base derived from 2-hydroxy-1-naphthaldehyde and p-methoxyaniline and its corresponding Co(II) and Zn(II) complexes were prepared and characterized successfully. The molar conductance values obtained indicated non electrolytic nature of the Complexes. The Co(II) complex is paramagnetic while Zn(II) complex is diamagnetic. The IR and elemental analysis data revealed 1:2 metal-ligand ratios in all the complexes. Also all the prepared compounds indicate promising antioxidant activity.

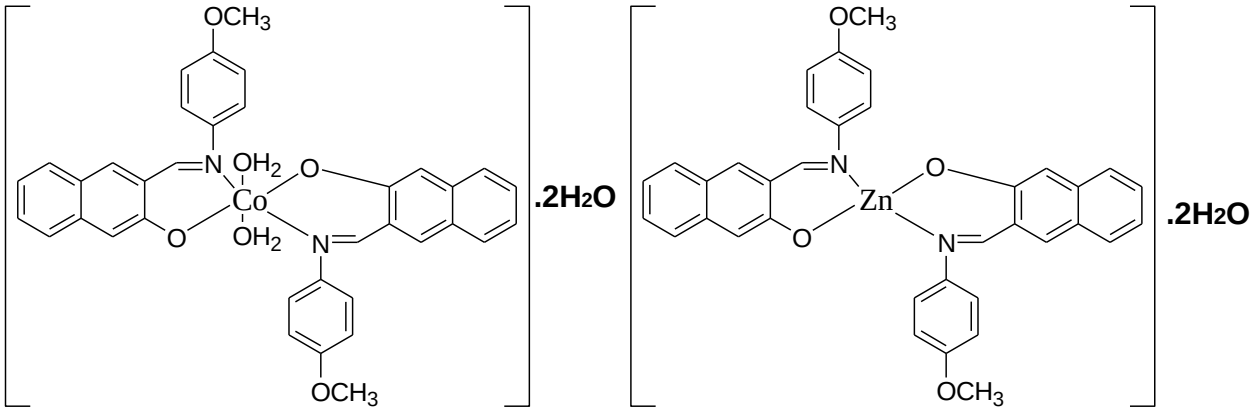


Figure 4: Proposed structures of prepared Complexes

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