

Synthesis, Characterization and Study of Antimicrobial Activity of Mn(II) and Ni(II) Complexes with Schiff Base Derived from 2-Hydroxy-benzaldehyde and 1-Naphthylamine

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Abstract: The Schiff base; 2-hydroxybenzalidene-1-naphthylamine and its Mn(II) and Ni(II) complexes were synthesized and characterized using, conductivity measurement, magnetic susceptibility, elemental analyses, melting point/decomposition temperature, electronic spectral analyses, infrared spectral analysis and solubility test. The Schiff base and its metal complexes were tested for antimicrobial activity. The molar conductance values range ($6.52 - 23.1 \Omega^{-1} \text{cm}^2 \text{mol}^{-1}$) revealed non-electrolytic nature of the complexes. The magnetic susceptibility values; 5.91 BM indicated Mn(II) complex as paramagnetic while $-Ve$ indicated Ni(II) complex as diamagnetic. The elemental analyses results revealed slight differences between calculated and observed percentages of values of C, H, and N, which is in line with the proposed structures of the synthesized compounds. The high decomposition temperature range ($201 - 223^\circ\text{C}$) indicated good stability of the complexes. The infrared spectra analysis results suggested that the Schiff base behave as bidentate ligand coordinates to metal ion via azomethine nitrogen and phenolic oxygen. The antimicrobial activity of the Schiff base and its metal complexes were carried out using agar well diffusion method against two bacteria strains; (*Salmonella typhi* and *streptococcus pneumoniae*) and two fungal isolates; (*Aspergillus fumigatus* and *Rhizopus species*). The results revealed that the Schiff base and its metal complexes exhibited moderate antimicrobial activity as compared with the standard drugs; (Gentamycin and Nystatin).

Keywords: Schiff Base, Complexes, 2-hydroxybenzaldehyde, 1-naphthylamine, Antimicrobial Activity

1. Introduction

The Schiff bases are the condensation products of primary amine and carbonyl compounds, and are an important class of ligand that coordinates with metal ions via azomethine nitrogen [4]. Schiff base represents one of the most employed classes of ligand in coordination chemistry due to convenient synthetic preparation and versatility. These aspects influence their ability to form stable complexes with a large number of transition metal ions [8]. They have been widely studied because of their industrial and biological applications [18]. The Schiff bases are known to be neoplasm inhibitors, antiviral, antibacterial, antioxidant, anticancer and plant growth regulator [3]. Schiff base metal complexes play a significant role in the development of chelation chemistry. The chelation

makes these compounds an effective and stereo-specific catalyst for oxidation, reduction and hydrolysis, and they also show biological activity and other transformation of organic and inorganic chemistry [11]. Metal complexes have been receiving considerable attention for many years, due to their interesting characteristics in the field of material science and biological system [1]. 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 [5].

2. Literature Review

The synthesis and characterization of Schiff base derived

from salicylaldehyde and ethylenediamine was reported by Naila, *et al.*, etc. [15]. This Schiff base has been identified by infrared spectra and melting point. Antibacterial activity of the prepared Schiff base was also studied against two Gram positive bacteria; *Staphylococcus aureus* and *Corynebacterium diphtheriae*, and two Gram negative bacteria; *Salmonella typhi* and *Escherichia coli* and the results indicated this Schiff base as inhibitor for bacterial growth.

Another novel Schiff bases were prepared by the reaction of 2-hydroxy-1-naphthaldehyde, 2-hydroxybenzaldehyde, 2-hydroxy-5-methoxybenzaldehyde and 2-hydroxy-5-nitrobenzaldehyde with phenazopyridine hydrochloride respectively, and their structures were elucidated by means of spectroscopic techniques. The electrochemical reduction of phenazopyridine hydrochloride and its Schiff bases were realized on a glassy carbon electrode in Dimethylsulfoxide (DMSO) using the cyclic voltammetric technique. The effects of functional groups on reduction potential of the Schiff bases were investigated. A general electrochemical reduction mechanism of the compounds was suggested [17].

Also Ibrahim *et al.*, [7] reported the synthesis of Cu(II) and Zn(II) complexes with Schiff base; N-salicylidene-4-chloroaniline, the Schiff base and its metal complexes were characterized using solubility test, infrared spectral analysis conductivity measurement, magnetic susceptibility, metal-ligand ratio determination and also screened for antimicrobial activity. The low molar conductance values range ($16 - 27 \text{ Ohm}^{-1} \text{ cm}^2 \text{ mol}^{-1}$) indicated all the complexes as non-electrolytes. The magnetic susceptibility revealed the Cu(II) complex as paramagnetic while Zn(II) complex as diamagnetic. The metal-ligand ratio was found to be 1:2 in all the complexes. The infrared spectral analysis suggested that the Schiff base behaves as a bidentate ligand. The Schiff base and its metal complexes were tested for antibacterial activity against *Staphylococcus aureus* and *Escherichia coli* and fungal activity against *Aspergillus flavus* and *Mucor species*. The results indicated moderate antibacterial and antifungal activity against the tested organism when compared with the standards (Ciprofloxacin and Ketoconazole), and this activity increases by increasing concentration. The metal complexes showed higher activity than free Ligand due to chelation.

The Fe(III) complexes with Schiff base derived from o-phenylenediamine, salicylaldehyde and isatin, 2-hydroxy-1-naphthaldehyde and acetyl acetone were reported by Nagajothi *et al.*, [14]. The complexes were characterized by elemental analysis, molar conductance, magnetic susceptibility, IR, UV-Vis spectral data and thermal analyses. The elemental analysis of the complexes confine with its stoichiometry. The complexes were found to be electrolytic in nature on the basis of molar conductance values. From the spectral data an octahedral geometry has been proposed for all the complexes. The possible geometries of metal complexes were evaluated using 3D molecular modeling picture. The metal complexes have been screened for their antibacterial and antifungal activity.

Four Pt(II) complexes with Schiff bases derived from salicylaldehyde and 2-furaldehyde with o and p phenylenediamine were reported by Akmal *et al.*, [2]. The synthesized Schiff bases and their metal complexes were characterized by elemental analysis, IR, UV-vis spectroscopy and thermal analyses. The data obtained show that Schiff bases were interacted with Pt(II) ions in the neutral form as a bidentate ligand via oxygen and nitrogen as the most probable coordination sites. The square planar geometrical structure with two coordinated water molecules was proposed for all complexes. The Schiff base and their metal complexes were screened for antimicrobial activity against bacterial species; *E. coli*, *B. subtilis*, *P. aereuginosa*, *S. aureus*, fungus; *A. niger*, *A. flavus*; and yeasts; *C. albican*, *S. cervisiae*. The results show that the Pt(II) complexes are more potent antimicrobial than the parent Schiff bases.

In addition Jai *et al.*, [9] reported two new Schiff bases derived from 1-hydroxy-2-naphthaldehyde, 2-hydroxybenzaldehyde with 2,2-(ethylenedioxy)bis(ethylamine) and their organosilicon and organotin complexes have been synthesized and characterized by spectroscopic techniques (UV-Vis, IR and NMR) in combination with elemental analyses. Spectroscopic studies suggest bonding between the central atom and Ligand in a penta and hexa coordinated fashion. Prepared Schiff bases and their corresponding complexes were evaluated for their antibacterial activity against Gram positive bacteria; *Staphylococcus epidermidis*, *Staphylococcus hominis* and Gram negative bacteria; *Pseudomonas aeruginosa* and *Klebsilla pneumoniea*, and this study revealed that the compounds exhibited antibacterial activity even higher than the standard drug; ciprofloxacin.

3. Material and Method

All the reagents were analytically grade and used without further purification. The 2-hydroxybenzaldehyde and 1-naphthylamine were used for the synthesis of the Schiff bases. The hydrated metal salt; $\text{MnCl}_2 \cdot \text{XH}_2\text{O}$ and $\text{NiCl}_2 \cdot \text{XH}_2\text{O}$ were used for the synthesis of metal complexes. The ethanol, methanol, acetone, chloroform, dimethylsulfoxide, dimethylformamide, diethyl-ether and conc. HNO_3 were used as a solvents, also anhydrous CaCl_2 was used as a drying agent. 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 the electrical melter balance model AB54.. Electrical conductance was measured using Jenway conductivity meter model 4010 range 20 - 200 μS . Infrared spectral analysis was determined using Fourier transform infrared spectrophotometer (FTIR-8400S) range 4000 - 400 cm^{-1} . The UV-Vis spectral analyses were determined using Perkin Elmer UV-Vis Spectrophotometer Lambda-35. The Melting points and decomposition temperature were determined using microprocessor melting point apparatus (WRS-IB). The Elemental analyses were determined using Series II CHNS/O Analyzer 2400 Perkin Elmer. The

Magnetic susceptibility was determined using magnetic susceptibility balance MKI Sherwood scientific ltd.

Synthesis of Schiff base

The Schiff base was synthesized by mixing 25ml (0.004 mol) ethanolic solution of 2-hydroxybenzaldehyde with that of 25ml (0.004 mol) ethanolic solution of 1-naphthylamine in the ratio 1:1. The resulting solution was refluxed for 4hrs and then cooled to room temperature; on cooling the precipitate formed which was then filtered, washed with ethanol and recrystallized with diethyl-ether and then dried in desiccators over anhydrous CaCl₂ to obtain the required Schiff base [6].

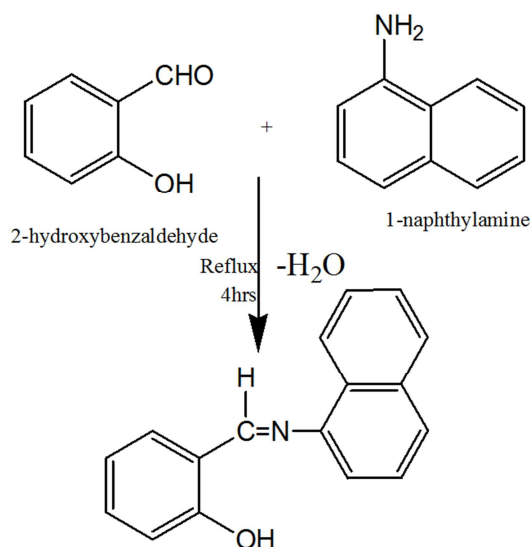


Figure 1. Synthesis of Schiff base.

Synthesis of metal complex

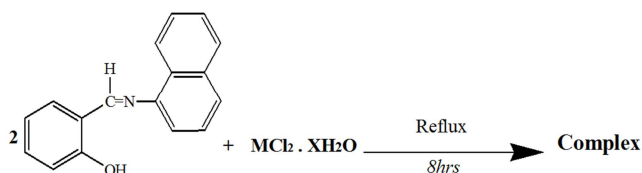


Figure 2. Synthesis of metal complexes.

The metal complexes were synthesized by mixing 25ml (0.004 mol) hot ethanolic solution of Schiff base with 25ml (0.004 mol) hot ethanolic solution of hydrated metal chloride salts in the ratio 1:2. The resulting mixture each was refluxed for 8hrs, the complex obtained in each case was cooled to room temperature, filtered, washed with ethanol and

recrystallized with diethyl-ether several times to remove any unreacted ligand. Finally each of the complexes was dried over anhydrous CaCl₂ in desiccators. The metal salts used are MCl₂ · xH₂O where M = Mn and Ni respectively [6].

Magnetic susceptibility measurements

The magnetic susceptibility of complexes was determined using magnetic susceptibility balance MKI Sherwood science ltd via the expression below According to modified procedure reported by Javed [10].

$$X_g = CL \frac{(R-R_0)}{10^3 M} \quad (1)$$

Where X_g = 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, R₀ = Reading obtained from preweight empty tube, M = mass of the sample in the tube (measured in gram).

Molar conductance measurements

The molar conductance of the synthesized complexes was determined by dissolving each of 1mmol of complex in 10 ml of dimethylsulfoxide (DMSO) and the corresponding specific conductance value was recorded using Jenway conductivity meter model 4010. According to modified procedure reported by Moamens [13].

From the specific conductance value recorded, the molar conductance of each metal complex was calculated using the expression below.

$$\text{Molar conductance} = \frac{1000 \times \text{specific conductance}}{\text{ionic concentration}} \quad (2)$$

Antimicrobial studies

The synthesized compounds were tested for antimicrobial activity against two bacterial strains; i.e. (*S. typhi* and *S. pneumoniae*) and two fungal isolates; i.e. (*A. fumigatus* and *R. species*) using agar well diffusion method.. The bacteria strains were spread on the nutrient agar, while the fungal isolates were spread on the potato dextrose agar using the sterile cotton swab. The recommended concentration of the test sample in DMSO was introduced in the respective wells. Other well supplemented with the standard antibiotics; i.e. (Gentamycin and Nystatin) respectively, to serve as positive controls. The plates were incubated at 37°C for 24h in the case of bacteria and 37°C for 48h in the case of fungi. The activity was determined by measuring the diameter of zones showing complete inhibition in (mm) [16].

4. Results

Table 1. Physical and Analytical Data of Schiff base and its metal Complexes.

Compound	Colour	Melt.pt/Dec. Temp. (°C)	%Yield	Elemental analysis Calculate (Found)		
				%C	%H	%N
Schiff base	Brown	106	70.47	84.59 (85.50)	5.26 (4.56)	5.67 (5.21)
[MnL ₂]	Brown	201	69.91	72.23 (72.60)	4.60 (4.31)	4.96 (5.65)
[NiL ₂]	Green	223	72.63	72.10 (72.88)	4.57 (4.55)	4.92 (5.31)

Key: L = Ligand, Mn = Manganese, Ni = Nicke

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

Compound	Water	Methanol	Ethanol	Chloroform	Acetone	DMF	DMSO
Schiff base	IS	SS	SS	S	S	S	S
[MnL ₂]	IS	SS	SS	S	S	S	S
[NiL ₂]	IS	SS	SS	S	S	S	S

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

Table 3. IR Spectral data of the Schiff base and metal Complexes.

Compound	V(O-H) cm ⁻¹	V(C=N) cm ⁻¹	V(M-O) cm ⁻¹	V(M-N) cm ⁻¹	V(H ₂ O) cm ⁻¹
Schiff base	3357	1611	-	-	-
[MnL ₂]	-	1603	681	589	-
[NiL ₂]	-	1607	607	548	-

Key: L= Ligand, Mn = Manganese, Ni = Nickel

Table 4. Conductivity measurement of the metal Complexes in DMSO.

Complex	Concentration Moldm ⁻³	Specific Conductance Ohm ⁻¹ cm ⁻¹	Molar Conductance Ohm ⁻¹ cm ² mol ⁻¹
[MnL ₂]	1.0×10 ⁻³	6.52×10 ⁻⁶	6.52
[NiL ₂]	1.0×10 ⁻³	23.1×10 ⁻⁶	23.1

Key: L= Ligand, Mn = Manganese, Ni = Nickel, DMSO= Dimethylsulfoxide

Table 5. Magnetic susceptibility of the metal Complexes.

Compound	Xg(gmol ⁻¹)	Xm(gmol ⁻¹)	μ _{eff} (BM)	Property
[MnL ₂]	2.59×10 ⁻⁵	1.46×10 ⁻²	5.91	Paramagnetic
[NiL ₂]	-4.13×10 ⁻⁷	-2.27×10 ⁻⁴	-Ve	Diamagnetic

Key: L = Ligand, Mn = Manganese, Ni = Nickel

Table 6. Electronic spectral data of Schiff base and its metal Complexes.

Compound	Solvent	π→π* (nm)	n→π* (nm)	LMCT
Schiff base	DMSO	220	352	-
[MnL ₂]		231	341	400
[NiL ₂]		228	381	402

Key: L = Ligand, Mn = Manganese, Ni = Nickel, DMSO = Dimethylsulfoxide

Table 7. Antimicrobial activity of Schiff base and its metal Complexes.

Compound	Zone of inhibition			
	Bacteria	Fungi		
	<i>S. typhi</i> (mm)	<i>S. pneumoniae</i> (mm)	<i>A. fumigatus</i> (mm)	<i>R. species</i> (mm)
Schiff Base	10	09	11	09
[MnL ₂]	11	12	14	13
[NiL ₂]	12	13	14	11
Gentamycin (Ctrl)	31	34	-	-
Nystatin (Control)	-	-	38	40

: A = Aspergillus, S = Salmonella S = Streptococcus, R = Rhizopus L = Ligand

5. Discussion

The condensation reaction between 2-hydroxybenzaldehyde and 1-naphthylamine (Figure 1) yielded Schiff base ligand which is brown solid with the percentage yield of 70.47% and melting point of 106°C (Table 1), this is in agreement with the colour and closer in melting point reported in the previous literatures.

The reaction between prepared Schiff base and Mn(II) and Ni(II) chloride salt (Figure 2) formed a complexes with

different physico-chemical properties; such as colour, decomposition temperature and elemental composition (Table 1), the colours may be due to charge transfer or nature of the ligand. The decomposition temperature of the complexes fall within the range of 201 – 223°C (Table 1), these high temperature revealed the good stability of the complexes, may be due to chelation between the Schiff base ligand and the metal ion or the large sizes of the complexes. Also the elemental analysis results revealed slight differences between the observed and calculated percentage of CHN respectively (Table 1). These results are in line with the

proposed formula of the compounds.

The prepared compounds are soluble in some organic solvents such as Chloroform, Dimethylformamide Acetone, Dimethylsulfoxide, slightly soluble in Methanol and Ethanol and insoluble in Diethyl-ether and water (Table 2).

The results obtained from the spectrum of the Schiff base shown a band at 3357cm^{-1} which may be assigned to $\nu(\text{-OH})$ stretching vibration, and this band disappeared in the spectra of the metal complexes revealing the coordination of the Schiff base with the metal ion through phenolic oxygen, another band appeared at 1611cm^{-1} in the spectrum of the

Schiff base which is assigned to $\nu(>\text{C}=\text{N}-)$ stretching vibration and this band shifted to lower wave number ($1603 - 1607\text{cm}^{-1}$) in the spectra of metal complexes (Table 3), showing the chelation of the Schiff base with the metal ion through nitrogen atom of azomethine group. The new bands appeared in the spectra of the metal complexes in the range ($607 - 681\text{cm}^{-1}$) and ($548 - 589\text{cm}^{-1}$), these bands are assigned to $\nu(\text{M-O})$ and $\nu(\text{M-N})$ stretching vibration respectively (Table 3), also showing the chelation of the Schiff base to the metal(II) ion. This is in line with the similar report in the previous findings.

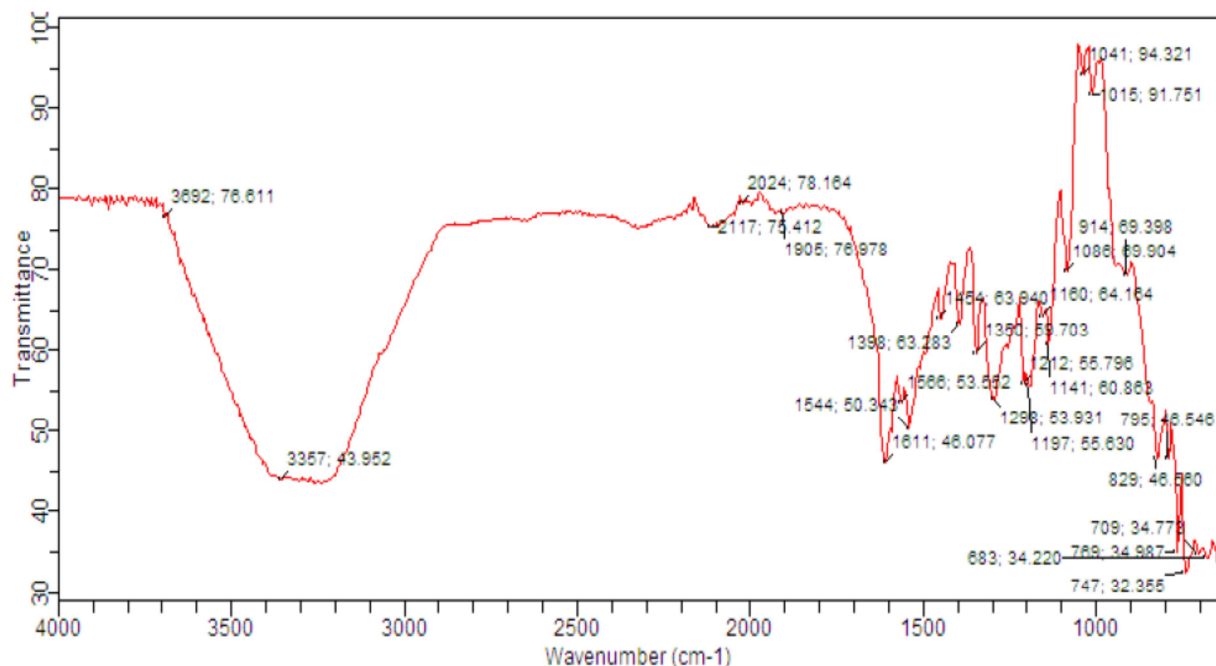


Figure 3. FTIR Spectrum of Schiff base.

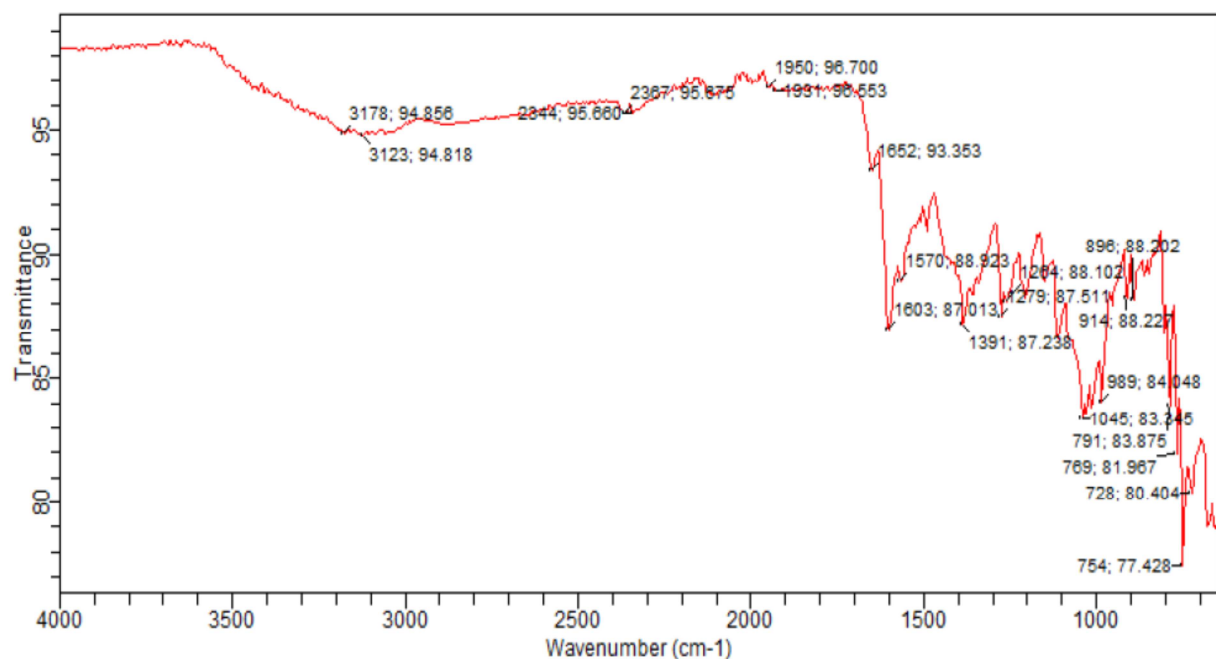


Figure 4. FTIR Spectrum of Mn(II) complex.

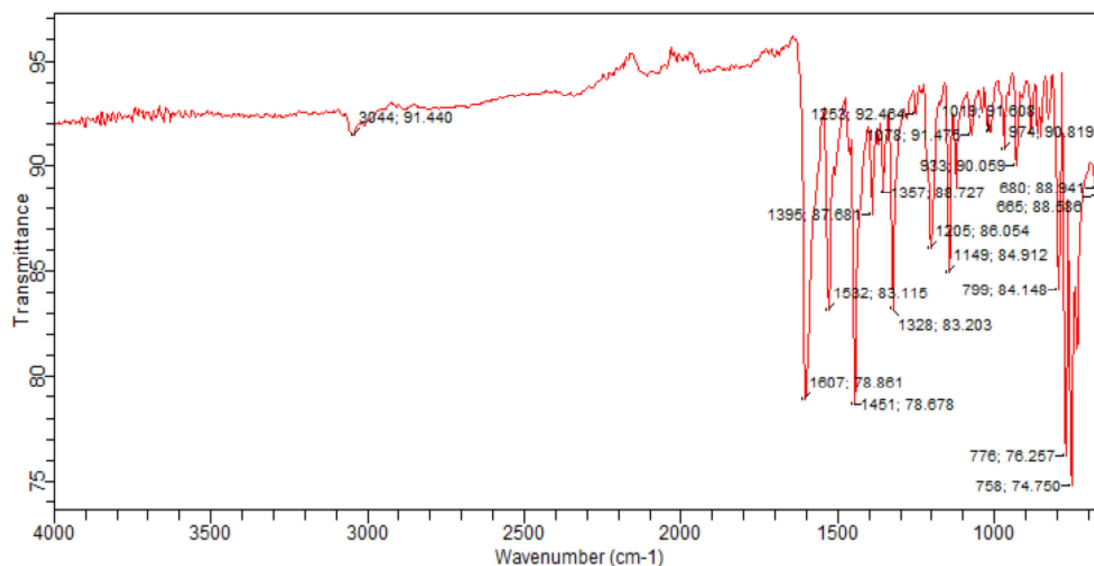


Figure 5. FTIR Spectrum of Ni(II) complex.

The molar conductance values range ($6.52 - 23.1 \Omega^{-1} \text{cm}^2 \text{mol}^{-1}$) revealed prepared complexes as non electrolytes. (Table 4) the values obtained are closer to values reported in the previous literature.

The magnetic moment obtained from magnetic susceptibility measurement of the complexes at room temperature revealed Mn(II) complex as paramagnetic with magnetic moment (5.91BM) as while Ni(II) complex as diamagnetic with (-Ve value) (Table 5).

The electronic spectra of the prepared compounds were carried out in DMSO using Perkin Elmer UV-Vis Spectrophotometer Lambda-35, and the results are shown in

(Table 6), The electronic Spectrum of the Schiff base revealed an absorption bands at 220nm and 352nm which may be attributed to $\pi \rightarrow \pi^*$ and $n \rightarrow \pi^*$ transitions of benzene and azomethine group respectively. These absorption bands shifted to lower and higher wave numbers in the spectra of the metal complexes in the ranges of (228 - 231nm) and (341 - 381nm) assigned to $\pi \rightarrow \pi^*$ and $n \rightarrow \pi^*$ indicating chelation of the metal ion to the Schiff base. Another absorption bands appeared in the spectra of the metal complexes in the range of (400 – 402nm) which may be assigned to ligand-metal charge transfer LMCT, also indicated the chelation of metal ion to Schiff base ligand.

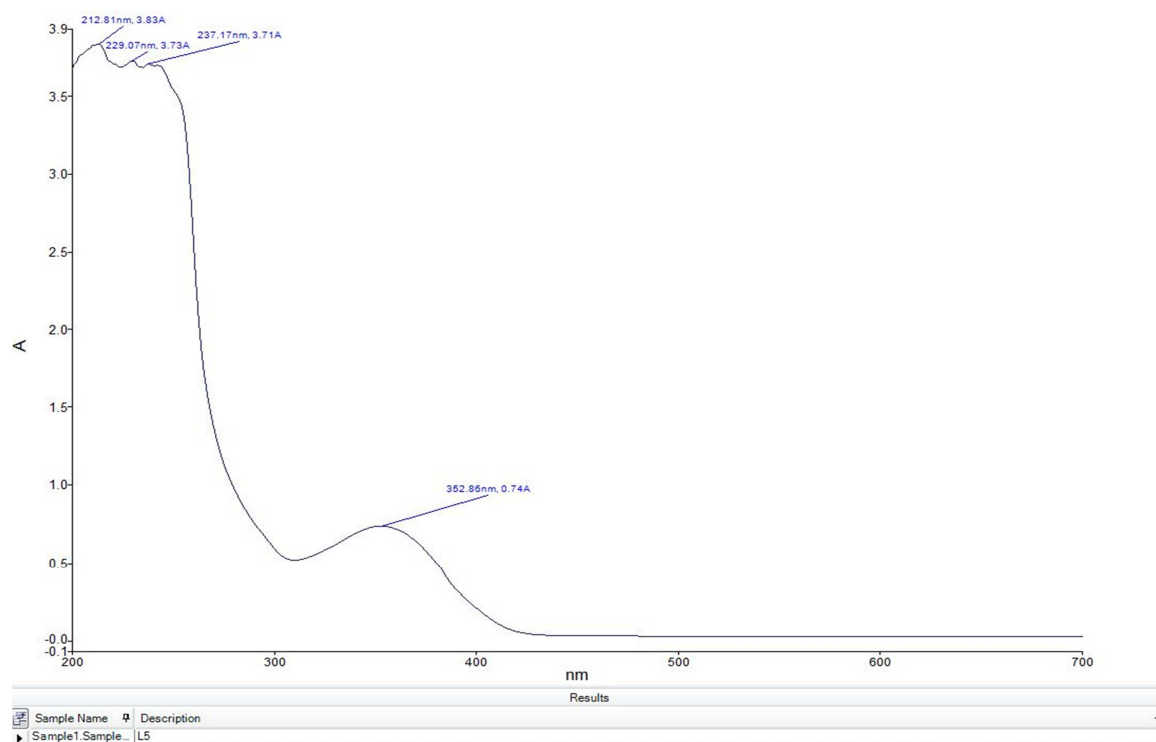


Figure 6. Electronic spectrum of Schiff base.

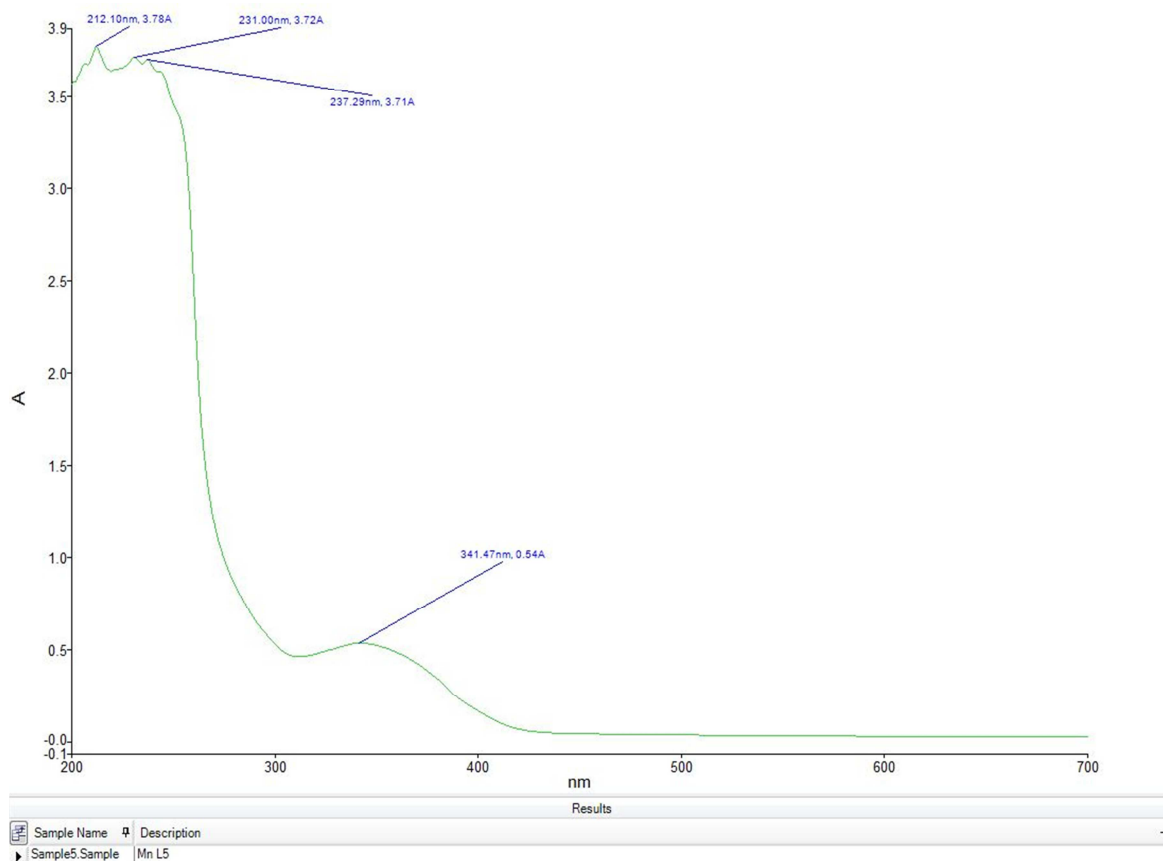


Figure 7. Electronic spectrum of Mn(II) complex.

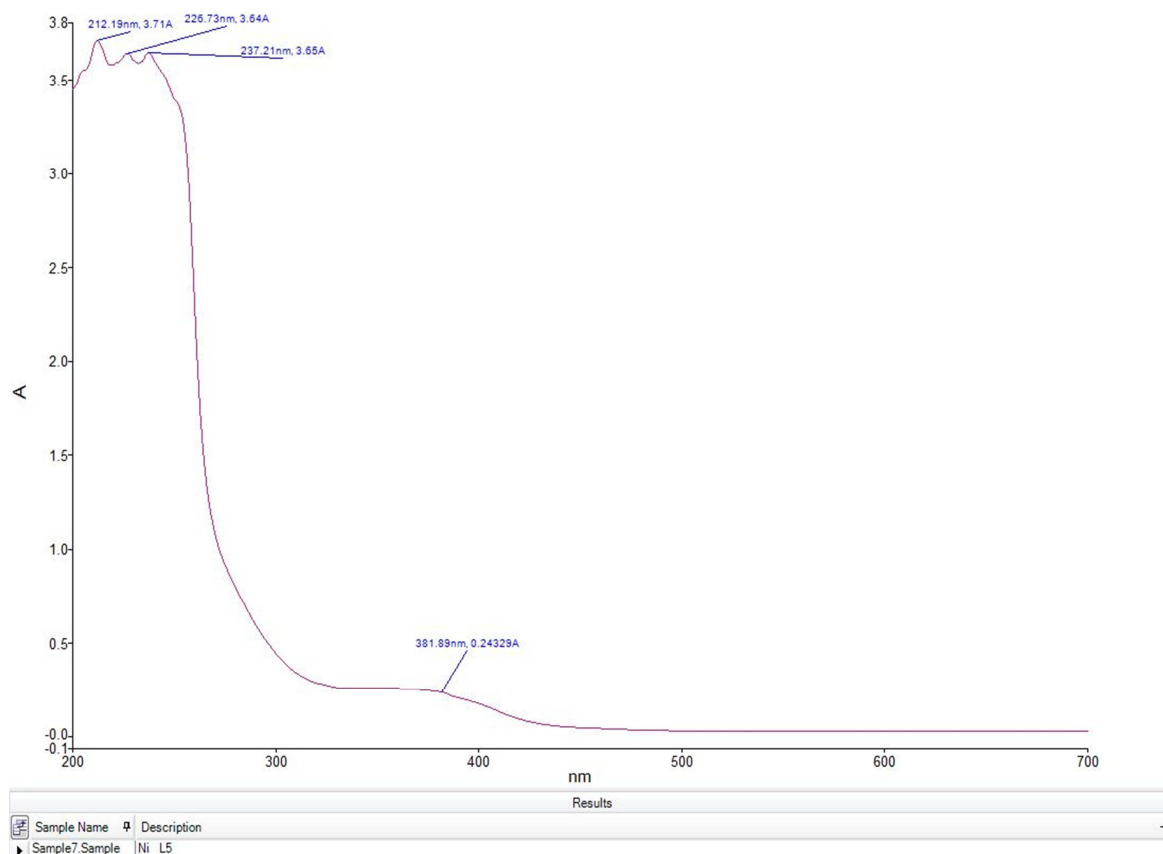


Figure 8. Electronic spectrum of Ni(II) complex.

The antimicrobial activity test of the prepared compounds was conducted using agar well diffusion method in dimethylsulfoxide, against two bacterial strains (*S. typhi* and *S. pneumoniae*) and two fungal isolates (*A. fumigatus* and *R. species*). The diameter of the inhibition zones were measured and recorded as shown in Table 7 and Figure 9. The results indicated moderate antimicrobial activity in comparison with the standard antibiotic (Gentamycin and Nystatin). The metal complexes showed higher activity than free ligand, which may be due to chelation processes, that may be explain using chelation theory and overtone concept of the semipermeable membrane. [12]

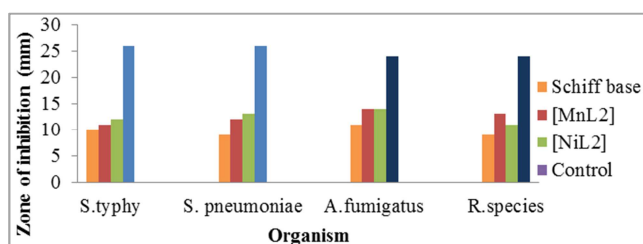
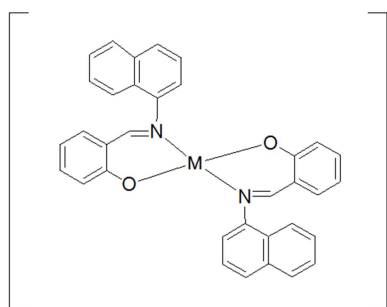


Figure 9. Antimicrobial Activity Schiff base and its metal complexes.

6. Conclusion

The Schiff base; 2-hydroxybenzalidene-1-naphthylamine and its Mn(II) and Ni(II) complexes were successfully synthesized and characterized using conductivity measurement, infrared spectral analysis, elemental analysis, magnetic susceptibility, electro-spectroscopic analysis, melting point/decomposition temperature and solubility test. The molar conductance values range ($6.52 - 23.1 \Omega^{-1} \text{cm}^2 \text{mol}^{-1}$) revealed non electrolytic nature of the complexes. The magnetic susceptibility results indicated Mn(II) complex as paramagnetic while Ni(II) complex as diamagnetic. The FTIR and elemental analysis data revealed 1:2, metal-ligand ratios in all the prepared complexes. The decomposition temperature values range ($201 - 223^\circ\text{C}$) indicated good stability of the complexes. These prepared compounds were tested for antimicrobial activity against two bacterial strains (*S. typhi* and *S. pneumoniae*) and two fungal isolates (*A. fumigatus* and *R. species*); and the results indicated that the compounds possessed moderate antimicrobial activity when compared with the standard antibiotics (Gentamycin and Nystatin).



Key: M = Mn(II), Ni(II)

Figure 10. Proposed structures of prepared Complexes of Schiff base.

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