



Spectral and Thermal Characterization of Mn(II), Ni(II) and Zn(II) Complexes Containing Schiff Base Ligands Towards Potential Biological Application

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Authors' contributions

This work was carried out in collaboration between all authors. Author MKEZ designed the study. Authors MSAD, FA and MSH performed the experiment and the statistical analysis, wrote the protocol, wrote the first draft of the manuscript and managed the analyses of the study. Authors MAM and MMH managed some literature searches. All authors read and approved the final manuscript.

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ABSTRACT

Some metal complexes of Zn(II), Mn(II) and Ni(II) ions with tetradentate N, O coordinating Schiff base ligand [2-bis(2-hydroxybenzylidene)hydrazinecarboxamide], (C₁₅H₁₃N₃O₃) were synthesized. The Schiff base ligand was derived from the condensation of semicarbazide hydrochloride and salicylaldehyde. The overall reactions were monitored by TLC and UV-Visible spectral analysis. All the ligands and metal complexes were isolated from the reaction in the solid form and characterized by IR, UV-Visible, Thermal analysis, Magnetic Susceptibility, Conductivity and some physical measurements. Spectroscopic evidence indicated that the Schiff base was behaved as N, O coordinating chelating agent. Magnetic susceptibility data coupled with electronic spectra suggested that [ZnC₁₅H₁₁N₃O₃] and [MnC₁₅H₁₁N₃O₃].2H₂O complexes are tetrahedral whereas [NiC₁₅H₁₁N₃O₃].H₂O complex is square planar. The conductivity value indicated that all the complexes were non-electrolyte. Thermal analysis (TGA and DTG) data showed the possible

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degradation pathway of complexes ($C_{12}H_8O_2$, at 200-490 °C and $-NH_3O^-$, at 400-650 °C). The Schiff bases and their metal complexes have been shown moderate to strong antimicrobial activity against some pathogenic bacteria.

Keywords: Schiff base; metal complexes; semicarbazide; thermal analysis; antimicrobial activity.

1. INTRODUCTION

Schiff base and their metal complexes have been extensively studied over the recent years due to their wide variety of biological activities, industrial and pharmaceutical applications [1-5]. Schiff base complexes of transition metal ions have great importance in coordination chemistry. The convenient route of synthesis and thermal stability of Schiff base complexes have contributed significantly for their possible applications. Multidentate ligands are extensively used for the preparation of metal complexes with interesting properties [6-9]. Among these ligands, Schiff bases containing nitrogen and phenolic oxygen donor atoms are of considerable interest due to their potential application in catalysis, medicine and material science [10-13]. Transition metal complexes of these ligands exhibit varying configurations, structural liability and sensitivity to molecular environments. The central metal ions in these complexes act as active sites for pharmacological agent. This feature is employed for modeling active sites in biological systems. Therefore, in view of our interest in synthesis of new Schiff base complexes, which might find application as pharmacological and as luminescence probes, we have synthesized and characterized new transition metal complexes of Schiff base formed by the condensation of semicarbazide hydrochloride and salicylaldehyde.

2. EXPERIMENTAL

2.1 Materials and Methods

All chemicals and solvents used were commercial products and were used as supplied. All metal(II) salts were used as chloride or sulphate. The solvents such as ethanol, methanol, chloroform, diethyl ether, petroleum ether, DMSO (dimethyl sulfoxide) and acetonitrile were purified by further distillation. The melting point or the decomposition temperature of all the prepared ligand and metal complexes were observed in an electrothermal melting point apparatus model No. AZ6512. Vibrational spectra (IR) were recorded with a NICOLET 310, FTIR spectrophotometer, Belgium, in the range

4000-225 cm^{-1} with a KBr disc as reference. UV-Visible spectra of the complexes in DMSO (0.5x 10⁻³M) were recorded in the region 200-800 nm on a Thermoelectron Nicolet evolution 300 UV-Visible spectrophotometer. The SHERWOOD SCIENTIFIC Magnetic Susceptibility Balance that following the Gouy Method were used to measure the magnetic moment of the solid complexes. The electrical conductance measurements were made at room temperature in freshly prepared aqueous solution (10⁻³ M) and in DMSO using a WPACM35 conductivity meter and a dip-cell with a platinum electrode. Some conductivity were also measured in PTI-18 Digital conductivity meter. The thermogravimetric analyses (TGA) were performed on Perkin Elmer Simultaneous Thermal Analyzer, STA-8000. The purity of the ligand and metal complexes were tested by thin layer chromatography (TLC).

2.2 Synthesis of Schiff bases

2.2.1 Synthesis of schiff base [SB]

Semicarbazide hydrochloride (1.12g, 10 mmol) dissolved in absolute ethanol / (30 mL) and distilled water (10 mL). Then salicylaldehyde (2.09 mL, 20 mmol) was added slowly to a constant stirring solution of semicarbazide hydrochloride (1.12 g, 10 mmol) with 2 ml acetic acid and the mixture was refluxed for 4-5h. On cooling, a solid product was formed which was filtered, washed with ethanol, diethyl ether and dried in vacuum over anhydrous $CaCl_2$. The synthetic pathway of ligand formation is shown in Scheme-1 and was monitored by TLC using petroleum ether, ethyl acetate, toluene and methanol solvents. The product was found to be soluble in methanol, chloroform, DMF and DMSO and insoluble in Ethanol, acetone, diethyl ether, Petroleum ether and Isopropanol.

2.3 Preparation Procedure of Schiff Base [SB] Metal Complexes

During complexation reaction a warm methanolic solution 30 mL (0.283 gm, 1mmol) of prepared Schiff base ligand ($C_{15}H_{13}N_3O_3$) was taken in a two-necked round bottom flask and kept on magnetic stirring. A warm 10 mL methanolic

Horiba conductivity meter B173 in which cell constant is fixed. So, the observed conductivity is the specific conductance. The experimental results showed that all the complexes are non-electrolyte in nature.

3.3 Characterizations by Magnetic Susceptibility

3.3.1 Measurement of magnetic susceptibility

The measurements of magnetic susceptibilities were made at about constant temperature; Curie-law was used and was calculated from the following equation.

$$\mu_{\text{eff}} = 2.83 \sqrt{\chi_{\text{m}}^{\text{corr}} \cdot T} \text{ B.M.} \quad (2)$$

Thus μ_{eff} obtained is known as effective magnetic moment. The observed values of effective magnetic moment (μ_{eff}) of the complexes at room temperature are given in Table 1. From the above data it is showed that the Zn(II) and Ni(II) complexes are diamagnetic and Mn(II) complex is paramagnetic in nature.

3.4 IR Spectra of Schiff Base Ligand-(SB) and its Metal Complexes

a. IR spectra of Schiff Base ligand [C₁₅H₁₃N₃O₃]:

An absorption band of Schiff base ligand showed at 1627 cm⁻¹, which is assigned as azomethine, $\nu(\text{HC}=\text{N})$ linkage [14]. The band indicated the formation of the Schiff base complex. An absorption band at 3494 cm⁻¹ observed due to $\nu(\text{-OH})$ stretching present [15]. The band at 3153 cm⁻¹ was assigned as $\nu(\text{-NH-})$ and was supported by the presence of $\delta(\text{NH}_2)$ deformation bands around 1600–1500 cm⁻¹. The strong band at 1697 cm⁻¹ assigned for $\nu(\text{C}=\text{O})$ attributed that the bond for amide group in the Schiff base

ligand [16-17]. The IR characteristic bands were given in Table 2.

b. IR spectra of Zn(II) complex [ZnC₁₅H₁₁N₃O₃]

In Zn(II) complex the absorption frequency of ligand shifted to the lower absorption frequency at 1619 cm⁻¹ suggested that CH=N group was coordinated to metal atom [14,18-22]. This may be explained based on a drift of the lone pair density of the azomethine nitrogen towards the metal atom [23,24], indicating that coordination occurred through the nitrogen of the (C=N) groups. The –OH band was also shifted to the lower absorption frequency at 3437 cm⁻¹ is due to the coordination of –OH groups as a deprotonated phenolic oxygen with the metal in the complex and $\nu(\text{-NH-})$ shifted to the region 3339 cm⁻¹ in the complex (Fig. 2) may be assigned to N–H stretching vibrations due to complex formation. Further conclusive evidence of the coordination of Schiff base with metals was the IR absorption spectra of the complex at 524 cm⁻¹ assigned as $\nu(\text{M-O})$ bond and at 307 cm⁻¹ assigned as $\nu(\text{M-N})$ bond in which –OH and HC=N group coordinate with metal atom [25,26].

c. IR spectra Ni(II) complex [NiC₁₅H₁₁N₃O₃].H₂O

In Ni(II) complex the absorption band of the ligand was shifted to the lower absorption frequency at 1615 cm⁻¹ suggested that CH=N group was also coordinated to the metal atom. This may be explained based on a drift of the lone pair density of the azomethine nitrogen towards the metal atom, which indicates that the coordination occurred through the nitrogen of the (C=N) groups. The –OH absorption band was also shifted to the lower absorption frequency at 3434 cm⁻¹ due to the coordination of –OH groups as deprotonated phenolic oxygen with the metal in the complex and $\nu(\text{-NH-})$ shifted to the region 3335 cm⁻¹ assigned to N–H

Table 1. Physical and analytical data of the Schiff base and metal complexes

Compounds/Mol. formula	Molecular weight	Color	Yield (%)	Decomposition temperature (°C)	μ_{eff} in B.M.
[SB] [C ₁₅ H ₁₃ N ₃ O ₃]	283	White	78	210	-
[Zn(SB)] [ZnC ₁₅ H ₁₁ N ₃ O ₃]	346.38	Yellowish white	75	245	0.43
[Mn(SB)] [MnC ₁₅ H ₁₁ N ₃ O ₃].2H ₂ O	371.94	Pale Yellow	71	>300	5.62
[Ni(SB)] [NiC ₁₅ H ₁₁ N ₃ O ₃].H ₂ O	357.69	Green	65	>300	0.55

stretching vibrations due to complex formation. Further conclusive evidence of the coordination of Schiff base with metals was the absorption of the complex at 529 cm^{-1} assigned as $\nu(\text{M}-\text{O})$ bond and at 376 cm^{-1} assigned as $\nu(\text{M}-\text{N})$ bond.

d. IR spectra of Mn(II) complex $[\text{MnC}_{15}\text{H}_{11}\text{N}_3\text{O}_3]\cdot 2\text{H}_2\text{O}$

In Mn(II) complex the absorption frequency of ligand was shifted to the lower frequency at 1611 cm^{-1} suggested that $\text{CH}=\text{N}$ group was also coordinated to metal atom [19,27-28]. This may be explained based on a drift of the lone pair density of the azomethine nitrogen towards the metal atom, indicating that coordination occurred through the nitrogen of the $(\text{C}=\text{N})$ groups. The $-\text{OH}$ band was also shifted to the lower absorption frequency at 3439 cm^{-1} is due to the coordination of $-\text{OH}$ groups as a deprotonated phenolic oxygen with the metal in the complex and $\nu(-\text{NH}-)$ shifted to the region 3337 cm^{-1} in the complex may be assigned to $\text{N}-\text{H}$ stretching vibrations due to complex formation. Further conclusive evidence of the coordination of Schiff base with metals was the absorption of complex at 524 cm^{-1} assigned as $\nu(\text{M}-\text{O})$ bond and at 314 cm^{-1} assigned as $\nu(\text{M}-\text{N})$.

3.5 Characterization by UV-visible Spectra

a. Electronic spectra of the ligand and complexes

The electronic spectrum of ligand exhibits high intense absorption peaks at 270 nm and 310 nm which have been assigned to $\pi\rightarrow\pi^*$ and $n\rightarrow\pi^*$ transition respectively. The UV-Vis spectra of the complex showed the presence of three strong absorption bands in the UV-Vis region at 265, 295 and 365 nm. The UV spectra of the ligand at 270 nm and at 305 nm are shifted in the Zn(II) complex. This confirmed the coordination of ligand to metal center [28]. The peak at 265 nm is attributed to $\pi\rightarrow\pi^*$ transition and the peak at 295 nm is assigned to $n\rightarrow\pi^*$ transition due to lone pair of electrons of an azomethine nitrogen and an antibonding p orbital. The presence of an absorption band at 365 nm in the case of the complex assigned as a ligand-to-metal charge transfer (LMCT). Since the zinc ion has d^{10} configuration and the observed absorption spectra of Zn(II) complex could be assigned the absence of any d-d electronic transitions the Zn(II) complex is diamagnetic in nature. However, taking into account the spectrum and

the magnetic moment value, the configuration of the Zinc(II) ion, a tetrahedral geometry [29,30] could be assumed for its complex. The UV-Vis spectra of Mn(II) complex at 265 nm and 295 nm are assigned as $\pi\rightarrow\pi^*$ and $n\rightarrow\pi^*$ transition respectively. The UV-Vis spectra of Mn (II) complex displayed absorption at the 320 nm, 390 nm assigned to the ligand field and charge transfer transitions. The metal complexes also exhibited d-d transition in the visible region of their spectra. The band at 440 nm is attributed to (d-d) transition of type ${}^6\text{A}_1\rightarrow{}^4\text{T}_2$ [31]. The magnetic moment of Mn(II) Complex is 5.62 B.M. The assigned spectrum transition and magnetic moment value indicate the tetrahedral structure of manganese (II) complex. The UV-Vis spectra of Ni(II) complex displayed absorption at 295 nm and 320 nm are assigned to the ligand to metal charge transfer transition (Fig. 1) [32]. The band at 340 nm and 400 nm is attributed to (d-d) transition of type ${}^1\text{A}_{1g}\rightarrow{}^1\text{A}_{2g}$ and ${}^1\text{A}_{1g}\rightarrow{}^1\text{B}_{1g}$ [33, 34]. The magnetic moment of Ni(II) complex is 0.55 B.M. The assigned spectrum transition and magnetic moment value indicate the square planar structure of Ni(II) complex. All the obtained electronic spectral data of ligand and complexes are shown in Table-3.

3.6 Thermo Gravimetric Analysis

Thermogravimetric analysis (weight changes) was performed for complexes in the temperature range from room temperature up to 800°C using Nitrogen as an inert gas. The heating rate was suitably controlled at $30^\circ\text{C min}^{-1}$. The data obtained strongly supported the proposed structure of the complexes. The TGA and DTG data of complexes in different temperature ranges are listed in Table-3.

a. For $[\text{ZnC}_{15}\text{H}_{11}\text{N}_3\text{O}_3]$ Complex

The decomposition of Zn(II) complex occurred in three main steps. The data from the TGA and DTG curve clearly showed that the metal complex was stable up to 200°C , indicating the absence of hydrated and coordinated water [35]. Then Where, the 1st step involves the major fragmentation of part $\text{C}_{12}\text{H}_8\text{O}_2$ (calculated 53.25%, experimental 52.12% weight) of the complex at temperature range $200-490^\circ\text{C}$. In 2nd step occurred around $490-640^\circ\text{C}$ (calculated 21.09%, experimental 20.32% weight) due to decomposition of $-\text{NH}_3\text{O}-$ and in 3rd step the complex is completely decomposed and removed as Zn/ZnO at above 650°C [14,35]. The TGA and DTG curve is shown in Fig.-2.

b. For $[\text{MnC}_{15}\text{H}_{11}\text{N}_3\text{O}_3]\cdot 2\text{H}_2\text{O}$ Complex

The TGA and DTG curve showed that the Mn(II) complex was decomposed in four steps. At 1st steps in the temperature range 150-220 °C (calculated 8.91%, experimental 20.32% weight) which can be attributed to the elimination of two hydrated water molecules [36]. The 2nd step involves the removal of $\text{C}_{12}\text{H}_8\text{O}_2$ (calculated 56.75%, experimental 56.28% weight) at temperature range 270-460°C. The part of complex -NH₃O- was decomposed between temperature range 460-650 °C (calculated 22.24%, experimental 23.14% weight) at 3rd Step

of decomposition. In 4th step, the complex is completely decomposed and removed as Mn/MnO at above 700°C [37,38]. The TGA and DTG curve is shown in Fig. 3.

c. For $[\text{NiC}_{15}\text{H}_{11}\text{N}_3\text{O}_3]\cdot \text{H}_2\text{O}$ Complex

The Ni(II) Complex was decomposed in four steps. The TGA and DTG curve showed that complex was decomposed at 1st steps in the temperature range 70-150°C (calculated 4.98 %, experimental 5.05% weight) which can be attributed to the elimination of one hydrated water molecules. The 2nd step involves the

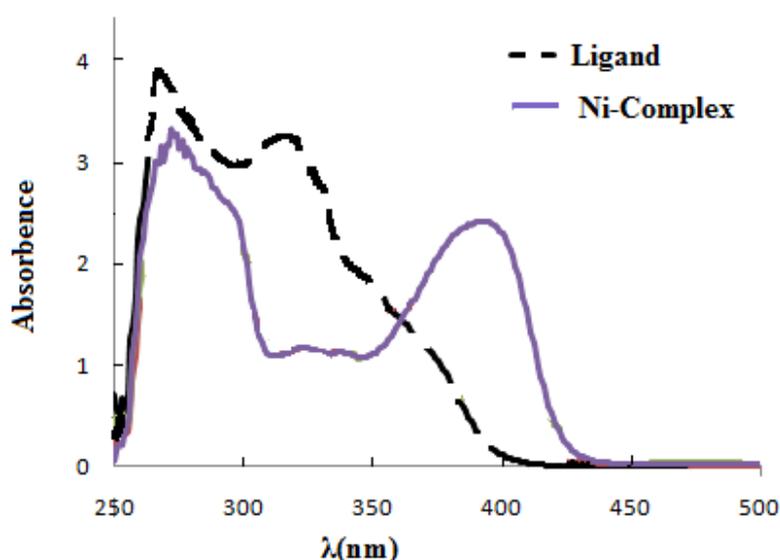


Fig. 1. Electronic spectra of Ligand $[\text{C}_{15}\text{H}_{13}\text{N}_3\text{O}_3]$ & Complex $[\text{NiC}_{15}\text{H}_{11}\text{N}_3\text{O}_3]\cdot \text{H}_2\text{O}$

Table-2. The electronic spectral data Schiff base and its metal complexes

Ligand/Complex	$\lambda(\text{nm})$	Wave number cm^{-1}	Assignment
[SB]	270	37037	$\pi \rightarrow \pi^*$
$[\text{C}_{15}\text{H}_{13}\text{N}_3\text{O}_3]$	305	32258	$n \rightarrow \pi^*$
[Zn(SB)]	265	37735	$\pi \rightarrow \pi^*$
$[\text{ZnC}_{15}\text{H}_{11}\text{N}_3\text{O}_3]$	295	33898	$n \rightarrow \pi^*$
	365	27397	Charge transfer (C.T)
[Mn(SB)] $[\text{MnC}_{15}\text{H}_{11}\text{N}_3\text{O}_3]\cdot 2\text{H}_2\text{O}$	265	37735	$\pi \rightarrow \pi^*$
	295	33898	$n \rightarrow \pi^*$
	320	31250	Charge transfer(C.T)
	390	25641	Charge transfer(C.T)
	440	22727	${}^6\text{A}_1 \rightarrow {}^4\text{T}_2$
[Ni(SB)] $[\text{NiC}_{15}\text{H}_{11}\text{N}_3\text{O}_3]\cdot \text{H}_2\text{O}$	275	36363	$n \rightarrow \pi^*$
	295	33898	Charge transfer(C.T)
	320	31250	Charge transfer(C.T)
	340	29411	Charge transfer(C.T)
	400	25000	d-d

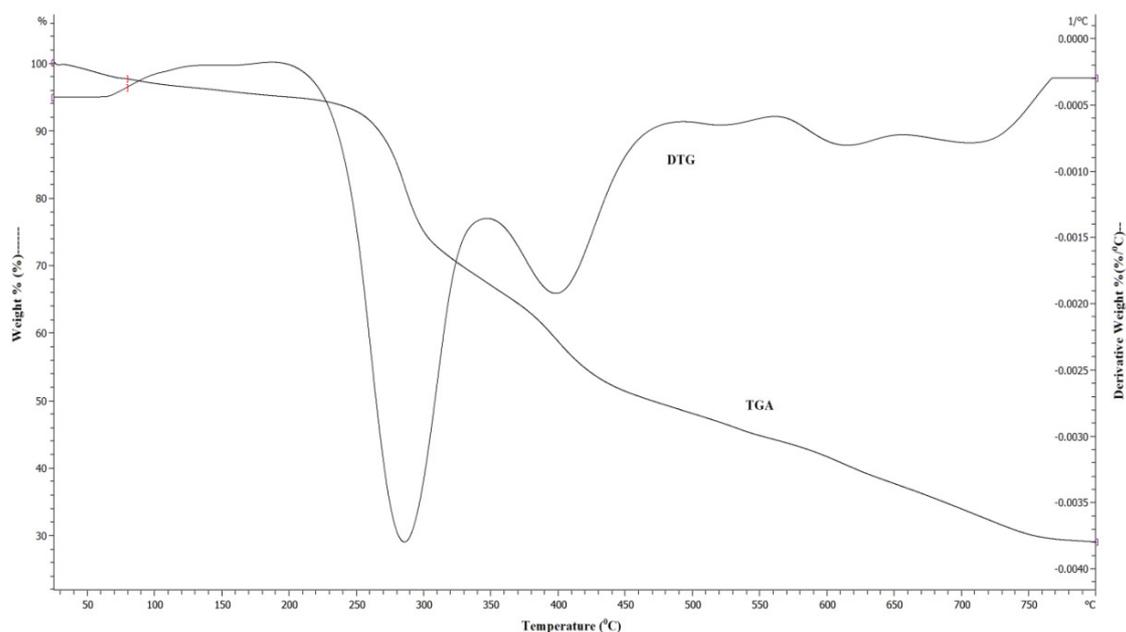


Fig. 2. TGA Band DTG curve of Complex $[\text{ZnC}_{15}\text{H}_{11}\text{N}_3\text{O}_3]$

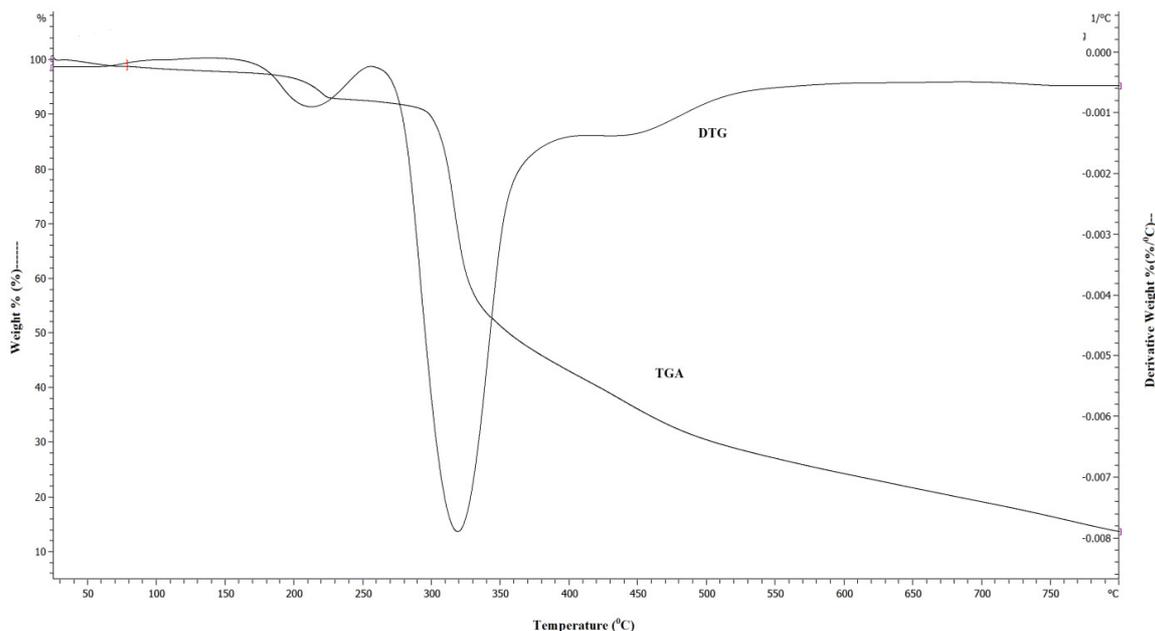


Fig. 3. TGA Band DTG curve of Complex $[\text{MnC}_{15}\text{H}_{11}\text{N}_3\text{O}_3] \cdot 2\text{H}_2\text{O}$

removal of $\text{C}_{12}\text{H}_8\text{O}_2$ (calculated 53.75%, experimental 54.17% weight) at temperature range 240-400 $^{\circ}\text{C}$. The part of complex - NH_3O - was decomposed between temperature range 460-650 $^{\circ}\text{C}$ (calculated 22.55%, experimental 21.20% weight) at 3rd Step of decomposition. In 4th step, the complex is completely decomposed and removed as Ni/NiO at above 600 $^{\circ}\text{C}$. The TGA and DTG curve is shown in Fig. 4.

4. ANTIBACTERIAL ACTIVITY

The prime objective of performing the antibacterial screening is to determine the susceptibility of the pathogenic microorganism to test the compound which, in turn, is used to a selection of the compound as a therapeutic agent. The free Schiff base ligand and their metal complexes were screened for their antibacterial activity against strains the *Bacillus cereus*

ATCC25923, *Streptococcus agelactiae*, *Escherichia coli* ATCC 25922, *Shigella dysenteriae*. The compounds were tested at a concentration of 30 µg/0.01 mL in DMSO solution using the paper disc diffusion method

with Kanamycin as standard [39]. The susceptibility zones were measured in diameter (mm) and the result are listed in **Table 4**. The susceptibility zones were the clear zones around the discs killing the bacteria.

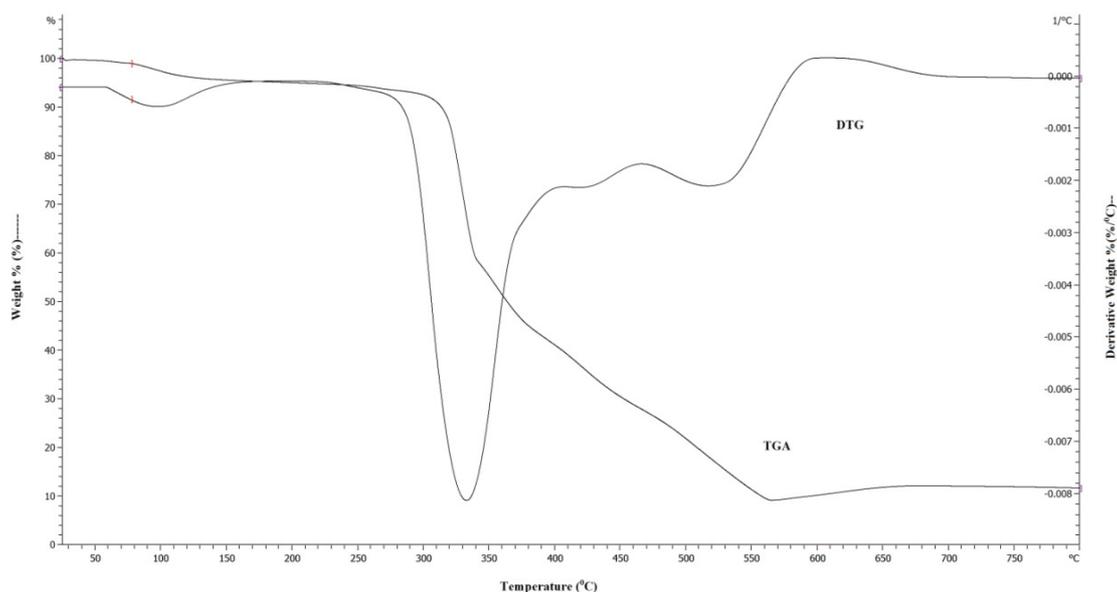


Fig. 4. TGA Band DTG curve of Complex $[\text{NiC}_{15}\text{H}_{11}\text{N}_3\text{O}_3]\cdot\text{H}_2\text{O}$

Table 3. Thermal data of Zn (II), Mn (II), and Ni(II) complexes

Complexes	Steps	Temperature Range/ °C	DTG peak/ °C	TG mass loss% calc./found	Assignments
$[\text{C}_{15}\text{H}_{11}\text{ZnN}_3\text{O}_3]$	1 st	200-490	285	53.25/53.12	$\text{C}_{12}\text{H}_8\text{O}_2$
	2 nd	490-640	600	21.09/20.32	$-\text{NH}_3\text{O}-$
	3 rd	>650		24.56/23.50	Zn/ZnO
$[\text{C}_{15}\text{H}_{11}\text{MnN}_3\text{O}_3]\cdot 2\text{H}_2\text{O}$	1 st	130-220	200	8.91/9.82	$2\text{H}_2\text{O}$
	2 nd	270-460	320	56.75/56.28	$\text{C}_{12}\text{H}_8\text{O}_2$
	3 rd	460-650		22.24/23.14	$-\text{NH}_3\text{O}-$
	4 th	>700		12.44/12.27	Mn/MnO
$[\text{C}_{15}\text{H}_{11}\text{NiN}_3\text{O}_3]\cdot \text{H}_2\text{O}$	1 st	70-150	100	4.98/5.05	H_2O
	2 nd	240-400	335	53.77/54.17	$\text{C}_{12}\text{H}_8\text{O}_2$
	3 rd	400-530	520	22.55/21.20	$-\text{NH}_3\text{O}-$
	4 th	>600		19.27/20.88	Ni/NiO

Table 4. Antibacterial activities of the compounds

Bacterials strains	Zone of inhibition, diameter in (mm)			
	$[\text{ZnC}_{15}\text{H}_{11}\text{N}_3\text{O}_3]$ (10 µg/disc)	$[\text{MnC}_{15}\text{H}_{11}\text{N}_3\text{O}_3]\cdot 2\text{H}_2\text{O}$ (10 µg/disc)	$[\text{NiC}_{15}\text{H}_{11}\text{N}_3\text{O}_3]\cdot \text{H}_2\text{O}$ (10 µg/disc)	Kanamycin (30 µg/disc)
Gram positive				
<i>Bacillus cereus</i>	11	13	08	26
<i>Staphylococcus agelactiae</i>	14	09	12	26
Gram negative				
<i>Escherichia coli</i>	13	10	12	26
<i>Shigella dysenteriae</i>	16	11	13	26

5. CONCLUSION

The foregoing observations suggest that $[ZnC_{15}H_{11}N_3O_3]$ and $[MnC_{15}H_{11}N_3O_3]$. $2H_2O$ complexes are tetrahedral whereas $[NiC_{15}H_{11}N_3O_3]$. H_2O complex is square planar. All the complexes were non-electrolyte. Thermal analysis (TGA and DTG) data showed the possible degradation pathway of complexes ($C_{12}H_8O_2$, at 200-490°C and $-NH_3O^-$, at 400-650°C). All these Schiff bases and their metal complexes have been screened for their antibacterial activities. The complexes showed good antibacterial activity compared to the ligands. This may be explained due to the enhanced lipophilic property of the central metal ion as a result of chelation with the ligand moieties.

COMPETING INTERESTS

Authors have declared that no competing interests exist.

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