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Conventional and Microwave Synthesis, Characterization and Study of Microbiological Activity of Complexes of Ni (II) [2-((Z)-(4-hydroxy-3-methoxy-5-((E)-thiazol-5-yl diazenyl) benzenylidene)amino)phenol] (MThBAP) in Extractive Spectrophotometric Determination of Nickel (II)

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Abstract: *A Schiff base and its complexes with Ni (II) have been synthesized by conventional methods & microwave synthesis which were characterized by elemental analysis, molar conductance, magnetic susceptibility, electronic spectra, IR and ESR spectroscopy. The metal complex is colored, solid and non - hygroscopic. The ligand behaves as {uni negatively} tridentate ligand coordinated to metal ions via azomethine (N), N=N and phenolic, anionic(O). Based on magnetic susceptibility values and electronic spectral analysis, the geometry of the complex was proposed to be octahedral. The molar conductivity data of the complex suggests their non-electrolytic nature. The ligand and metal complexes have been analyzed for their microbiological activity.*

Keywords: Coordination, Metal Complexes, Microbiological activity, Octahedral, Schiff base (MThBAP)

I. INTRODUCTION

Schiff base and its metal complex occupy an important place in the field of coordination chemistry (Shirodkar et al., 2001) Nitrogen and other atoms such as oxygen (Djebbar et al., 1997), (Bhattacharya et al.,1998; Liu et al., 1996; Wu et al., bases. Chelating ligands containing N, S, and O donor atoms show biological and catalytic activities (Amir et al., 2002; Hamada, 1997; Vaghasiy et al., 2004). Many metal complexes are synthesized and are studied. In the detection of metal cations at the micro level Schiff bases are used (Jungreis & Thabet, 1969). Microwave-assisted synthesis is a branch of green chemistry. It continues to develop at a very fast rate in organic, organometallic, and coordination chemistry. Microwave irradiated reactions are done under solvent-free or less solvent conditions. Reduced pollution, low cost and better yield, shorter reaction time, and simplicity in processing and handling are the advantages of Microwave-assisted synthesis. There are a few reports on the synthesis of metal complexes by microwave methods (Mahajan et al., 2009; Mishra et al., 2012; Mohanan et al., 2008; Sharma et al., 2010) ligand In the present paper, metal complex of Ni (II), with Schiff base 2-(((Z)-4-hydroxy-3-methoxy-5-((E)-thiazol-5 yldiazenyl) benzylidene) amino) benzoic acid have been synthesized by conventional as well microwave method and both process are compared. 2001) are present as donor atoms in metal complexes of Schiff's. Schiff base and its metal complexes are important in the field of coordination chemistry. Nitrogen and other atoms such as oxygen are used as bases for chelating ligands containing N, S, and O donor atoms, which exhibit biological and catalytic activities. Many metal complexes have been synthesized and studied, and a schiff base is often used in the detection of metal cations at the micro level. Microwave-assisted synthesis is a branch of green chemistry that has many advantages, including reduced pollution, low cost, better yield, shorter reaction time, and simplicity in processing and handling. There are a few reports on the synthesis of metal complexes by microwave methods, and in this paper, the metal complex of Ni (II) with Schiff base 2-(((Z)-4-hydroxy-3-methoxy-5-((E)-thiazol-5-yl-diazenyl)benzylidene)amino)benzoic acid was synthesized using both conventional and microwave methods. Both processes were compared, and the results were analyzed.

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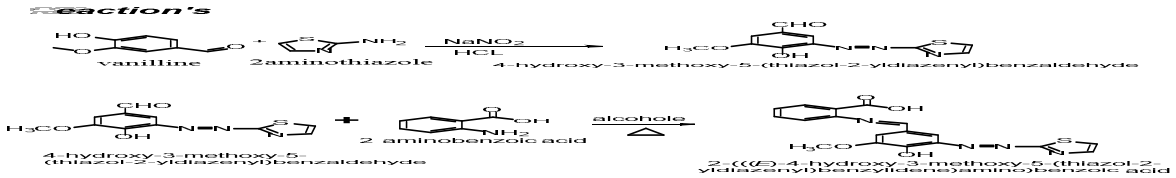
II. EXPERIMENTAL SECTION

ELICO - SL 159 spectrophotometer with optically matched quartz or glass cells of 1cm path length were used for absorbance measurement. An ELICO – LI 127 pH meter was employed for pH measurements.

General procedure synthesis of[2-((Z)-(4-hydroxy-3-methoxy-5-((E)-thiazol-5-yl diazenyl)benzenylidene)amino) phenol] :-

To synthesize [2-((Z)-(4-hydroxy-3-methoxy-5-((E)-thiazol-5-yl diazenyl)benzenylidene)amino) phenol], follow these steps: 1. Prepare a solution of 2-aminothiazol (0.005 moles) and slowly add sodium nitrite solution (0.005 moles) in increments of 2 ml at a time while keeping the temperature below 5°C for 30 minutes. 2. Stir the solution until red crystals form. 3. Gradually add the cold diazonium salt (0.005 moles) to a solution of vanilline in 25cm3 of 10% sodium hydroxide. 4. Stir the solution for an hour and then filter it using a Buchner funnel. 5. Clean the filtered object with a saturated solution of sodium chloride.

Fig.1: Synthesis of Azo Compound



Using 50 ml of ethyl alcohol as the solvent, 0.01 moles of the azo compound and 0.01 moles of 2-aminophenol are added to a flask with a circular bottom and a few shards of porcelain. It is recirculated for three hours while being connected to the water condenser. The combination is then put into a beaker and kept in the fridge for the remainder of the day. The final product underwent filtering and drying. Schiff's base compound 2-(((Z)-4-hydroxy-3-methoxy-5-((E)-thiazol-5-yldiazenyl)benzylidene)aminophenol (MThBAP) was created as brown crystals. The crystals have undergone regrowth.

Fig.2-Synthesis of Ligand MThBAP

4-hydroxy-3-methoxy-5-thiazol-2-yldiazenyl	2-aminophenol	[2-((Z)-(4-hydroxy-3-methoxy-5-((E)-thiazol-5-yldiazenyl)benzenylidene)amino)phenol]

Green synthesis of [2-((Z)-(4-hydroxy-3-methoxy-5-((E)-thiazol-5-yldiazenyl)benzenylidene)amino)phenol]

Microwave radiation was applied at 180 0 for 0.4 minutes to a beaker containing 0.005 moles of 2-aminophenol, 0.005 moles of azo compound, and a few drops of pure alcohol. In a short (2 min) reaction time, higher yields were attained. It is possible to create greenish-brown Schiff's base crystals.

Preparation of Nickel Complex by conventional method :

Preparation of Schiff's base ligand (MThBAP) by conventional method The complex of Nickel was made by using hydrated NiCl2 in an ethanolic solution of ligand in a molar ratio of 1:2 at pH 7.6 -8.4. For 4 – 5 hrs the mixture was refluxed in a water bathThe following is a description of the procedure used to prepare the Schiff's base ligand

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(MThBAP) using the conventional method. Nickel complex was made using hydrated NiCl2 in an ethanolic solution of ligand with a molar ratio of 1:2 at pH 7.6-8.4. The mixture was refluxed in a water bath for 4-5 hours and a dark brown colored solid was obtained after cooling at room temperature. The resulting complex with the formula [Ni L2] was washed with water and ethanol, recrystallized (Vogel, 1989), and dried, with a yield of 60-70%. The dark brown-colored solid was separated after cooling at room temperature. The resulting complex corresponding to the formula [Ni L2] was washed with water and ethanol was then recrystallized (Vogel, 1989) and dried(yield 60 - 70%).

Preparation of Nickel Complex by microwave method :

The process involved grinding hydrated NiCl2 and a ligand in a 1:2 ratio. The resulting mixture was taken in a 50 ml borosil beaker and mixed with 3-4 ml of dry ethanol as a solvent. The mixture was then irradiated in a microwave oven at 700 Watts for 2 minutes, resulting in a short reaction time of 1-2 minutes and higher yields. The product obtained was recrystallized with ethanol and ether and finally dried under reduced pressure over anhydrous CaCl2 in a desiccator. The progress of the reaction and purity of the product was monitored by TLC using silica gel G, resulting in a yield of 91-92%.

Biological Studies

The Cup Plate Method is a technique used to determine the antibacterial and antifungal activity of ligands and their Ni(II) complexes. Firstly, a stock solution of 2000 ppm of the ligand and Ni(II) complex is prepared by dissolving 20 mg in 10 ml solvent carbon tetrachloride on an active ingredient basis. This solution is kept at room temperature until it is needed.

Next, sterile Sabouraud's agar plates are used for the fungus Candida albican, and sterile Mueller Hinton agar plates are used for bacterial test cultures. These plates are seeded with 1ml of 24-hour-old, 0.1 O.D. cultures. For the surface spread on Sabouraud's agar plates, 0.2 ml of 48-hour-old Aspergillus culture is used.

Then, wells are punched in the above media and 50 µl of the compounds are added. Depending on their culture, the plates are incubated for 48 hours at 37°C or room temperature. The area around the well is inhibited and measured in millimeters. The plates are further incubated at 37°C or room temperature for 48 hours, depending on the culture. Finally, the zone of inhibition around the wells is measured in millimeters.

III. RESULTS AND DISCUSSION: MTH BAP

The metal complex [Ni L2] is a solid with a dark-brown color. It is non-hygroscopic and stable at room temperature. The metal-ligand ratio for these complexes is 1:2.

Table I contains the analytical and physical data for both the ligand and their respective metal complexes.

Table-I The Analytical and Physical data of ligand & Ni Complex

Compound (Colour)	Molecular Weight	Reaction period & %yield Conventional methods	Reaction period % Yield Micro synthesis	M.P.	% Elemental Analysis Found (Calculated)				
					C	H	O	S	N
Ligand $C_{17}H_{14}N_4O_3S$ (greenish brown)	354.38	4 hours 79%	0.4 Minutes 92%	240	57.00 (57.62)	3.90 (3.98)	13.50 (13.54)	9.08 (9.05)	15.78 (15.81)
Ni[C17 H13 N4 O3 S]2 Ni L_2	763.453	4.5 hours 73%	0.3 minutes 91% yield	286	53.01 (53.44)	3.39 (3.406)	12.50 (12.57)	8.33 (8.38)	14.63 (14.68)

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A . Infra-red Spectral Analysis

The infrared (IR) spectrum can be used to identify the frequency of functional groups of a ligand and complex through characteristic stretching. This study utilized the Shimadzu FTIR-IR AFFINITY-1SWL Spectrometer (4000-400 cm-1) with KBr pellets to record the IR spectrum. Table II provides a summary of the most significant infrared spectra bands of the ligand and investigated metal complexes, with Fig. 2 and Fig. 3 showing the corresponding IR figures.

The C=N stretching of the azomethine group appears as a broad band at 1613 cm-1 in the IR spectrum due to the ligand (Lever, 1973; Noorjahan et al., 2014). In Ni (II) complexes, the band shifted to lower regions at 1611 cm-1, indicating the coordination of azomethine nitrogen to the metal atom in the complexes (Anitha et al., 2013; Priya D., & Kumar, H., 2010). This shift occurred due to the donation of electron density from nitrogen to the metal, which takes place between the azomethine nitrogen and the metal atom.

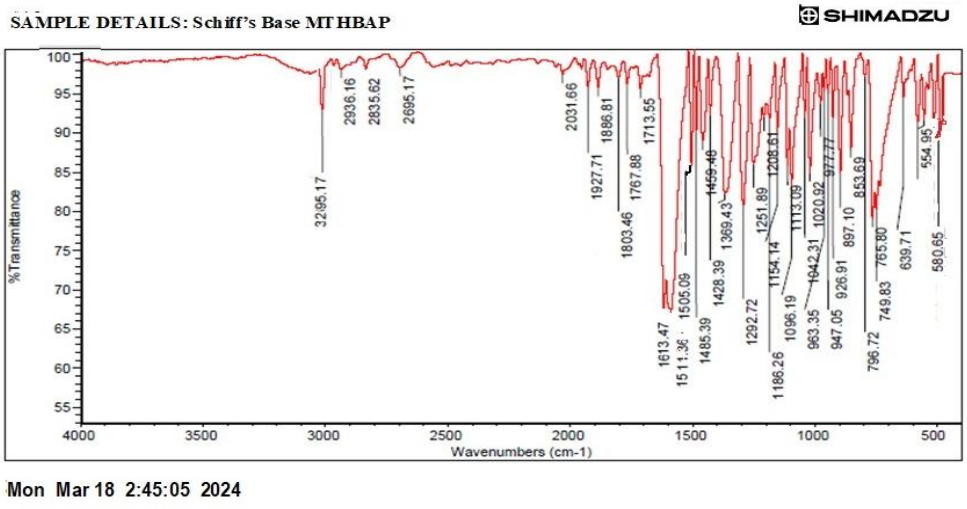
Based on the IR spectra analysis, it has been observed that a weak band around 3295 cm-1, which is caused by an intra-molecular hydrogen-bonded -OH group (Bellamy,1954), (Anitha et al., 2013; Hasan et al., 2016; Silverstein & Webster, 1998) is present in the ligand but is absent in the nuclear Ni complex. This indicates that the phenolic proton has dissociated upon complexation, and the phenolic anionic oxygen is now involved in coordination. This is further supported by the fact that the strong band at 1292 cm-1 due to C – O (phenolic) in the ligand has shifted to 1311 cm-1 in the spectra of complexes (Anitha et al., 2013).

Therefore, it can be concluded that the ligand behaves as a uni negatively tridentate ligand coordinated to the metal ion via azomethine (N) N=N and phenolic, anionic (O).

Table II infrared spectra bands of the ligand and investigated metal complexes

compound	υ(-OH)	υ(C-N)	υ(-CO)	υ(M-N)	υ(M-0)
ligand	3295.67 phenolic	1613.05	1292.72	----	-----
Ni- Complex	----------	1611.36	1311.42	479.38	455.78

Figure 3: IR Spectra of ligand



B. Molar Conductance

At room temperature, the molar conductance of a mononuclear nickel complex in DMF solvent (1x10-3 M) using an ELICO Conductivity meter with cell constant 1.0 cm-1 is 26 Ohm-1cm2mol-1. The obtained values were compared with known molar conductivities from Sulekh and Umendra (2005), indicating that the complexes are non-electrolytic in nature, as reported by Priya and Kumar (2010).

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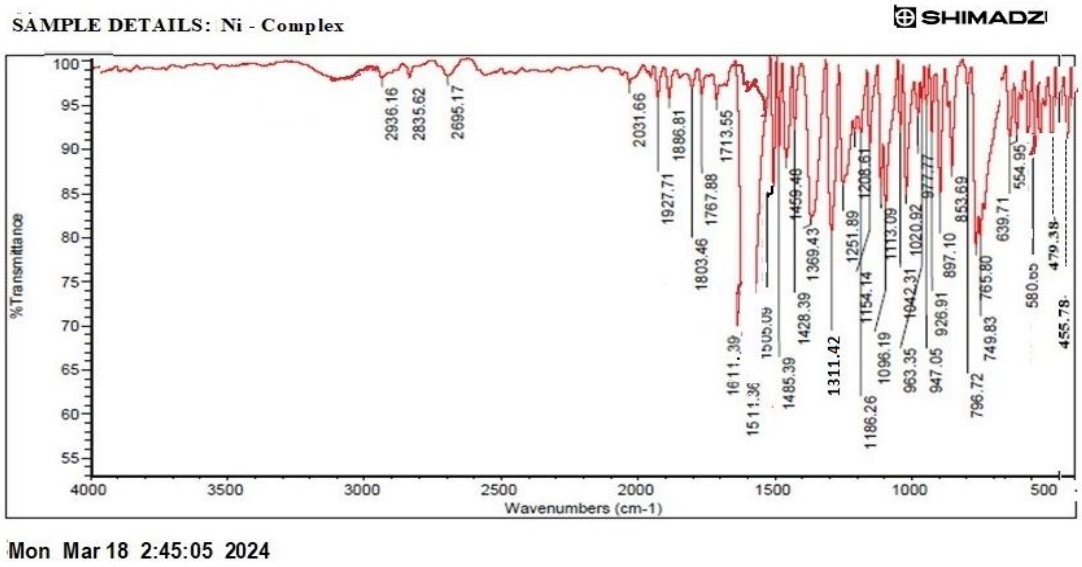
Figure 3: IR Spectra of complex

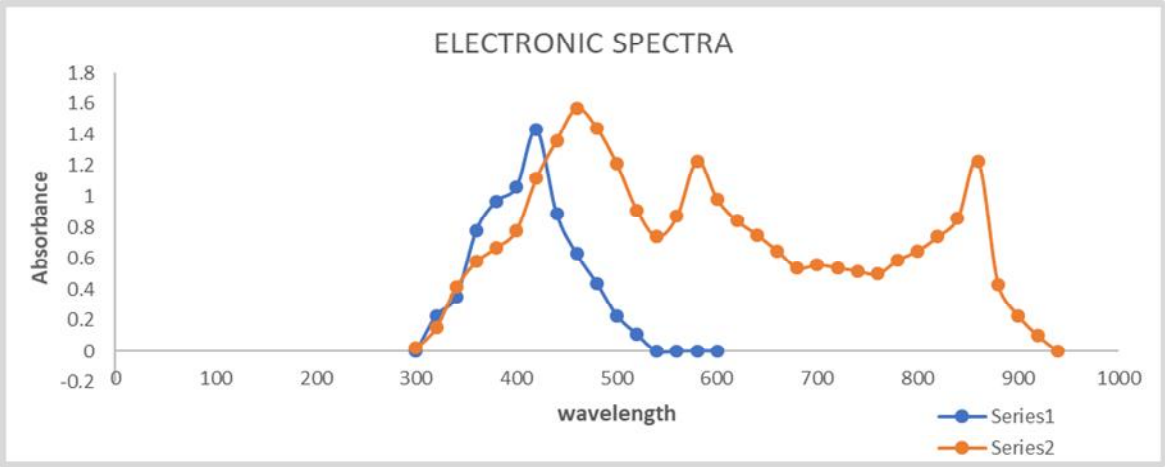


C. Electronic spectra and magnetic susceptibility measurements of complexes

The electronic transition takes place in the UV-Visible electromagnetic region. ELICO SL - 159 UV-Visible Spectrophotometer is used to record electronic spectra. The transition of metal ions occurs in a variety of structural environments. Hence the electronic structures are extremely varied and have been identified by UV-Visible spectroscopy. Data is shown in Table III and electronic spectra are shown in Fig. 4.

The electronic absorption spectra of Ni (II) complex exhibit the three band around **21739.13**cm^{-1} (460 nm), **17241.37** cm^{-1} (580 nm), **11627.9**cm^{-1} (860 nm), at room temperature which could be attributed to d-d transition and may be assigned to $3A_{2g} \rightarrow 3T_{1g\,(p)}$, $3A_{2g} \rightarrow 3T_{1g\,(f)}$, $3A_{2g} \rightarrow 3T_{1g\,(f)}$ transition (Adhikary et al., 2015; Al-hamdani & Shayma , 2011; Bardakçı et al., 2015; ,Amani, S., Alturiqiet.et al., 2018;, Salisu Abubakar et.all,2024, S Mini and *V Sadasivan 2016,) suggesting octahedral geometry .The magnetic moment value 3.12 B.M is consistent with the octahedral Ni (II)complex (Amani, S., Alturiqiet.et al., 2018 Priya D. & Kumar, H., 2010).

Fig-4 Electronic Spectra



The proposed geometry of [Ni L2] complexes, which is octahedral, was supported by the calculated values of several ligand field parameters, such as Racahinter electronic repulsion parameter (B'), nephelauxetic parameter (β), ligand field splitting energy (10 Dq), and ligand field stabilization energy (LFSE). These values were calculated using the

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Underhill and Billing equation, as described in previous studies (Amani, S., Alturiqiet et al., 2018; Salisu Abubakar et al., 2024; Satyanarayana D.N, 2011).

The calculated B' values for the [Ni L2] complexes were found to be lower than the free ion values due to the orbital overlap and delocalization of d-orbitals. The β values, which were calculated using the equation B'(complex)/B(free ion), play a significant role in determining the covalency of the metal-ligand bond. These values were found to be less than unity, suggesting a considerable amount of covalency for the metal-ligand bonds. The value of β was calculated to be 0.286, indicating the covalent character in the metal-ligand bond (Amani, S., Alturiqiet et al., 2018). The ligand parameters values are shown in table IV

Table III. Electronic Spectra and Magnetic Moment of the Ni (II) Complex

compound	Band, λ max	Band, λ max (nm)	Band, λ max (cm^{-1})	Assignments	μeff (B.M.)
Ni L_2	υ_1	860	11627.9	$3A_{2g} \rightarrow 3T_{1g\,(p)}$	3.18
	υ_2	580	17241.37	$3A_{2g} \rightarrow 3T_{1g\,(f)}$	
	υ_3	460	21739.13	$3A_{2g} \rightarrow 3T_{1g\,(f)}$	

Table IV. The ligand parameters of the Ni (II) Complex

Compound	Band, λ max	Band, λ max (cm^{-1})	Dq (cm^{-1})	B(cm^{-1})	υ_2/υ_1	β
NiL 2	υ_1	11627.9	1162.7	273.12	1.47	0.262
	υ_2	17241.37				
	υ_3	21739.13				

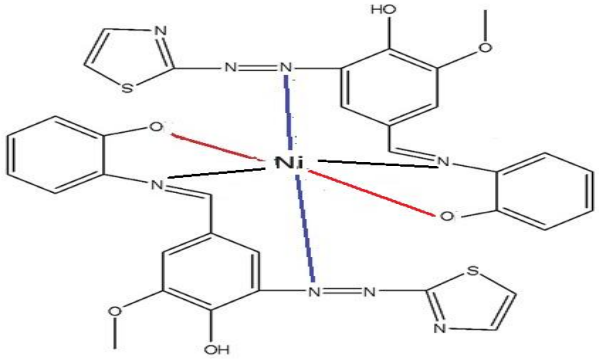
D. Determination of composition of the extracted species :

Composition of the complex was found to be 1:2 (Ni: L) Which was verified by using the Mole Ratio Method and Job's Continuous Variation Method (Makhijani et al.,2018)

E. Proposed Structure of the complex:

Analytical, magnetic, spectroscopic data and microbiology data suggests that the chelating nature of ligand forms stable complexes with Ni (II). Based on experimental evidence the probable structures of the complexes can be shown as Fig.5.

Fig-5 Proposed Structure



Proposed Structure of Ni - MTHBAP Complex

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Biological Activity:

A recent study tested the antibacterial and antifungal activity of a Schiff's base ligand and its nuclear Ni complex against various types of microorganisms including gram-positive bacteria like Staphylococcus aureus and Corynebacterium diphtheria, gram-negative bacteria like Escherichia coli and Klebsiella pneumonia, and fungi like Candida albicans and Aspergillus species.

The study found that the metal chelates of the Ni (II) complex had significantly higher antibacterial and antifungal activity compared to free ligands. This increase in activity was due to the chelation process, which reduces the polarity of the metal ion and enhances the lipophilicity of the complex. This makes it easier for the complexes to penetrate lipid membranes, block the metal sites on enzymes of microorganisms, and disturb the respiration process of the cell.

The variation in the activity of different complexes against different organisms was due to differences in the permeability of the microbe's cells or ribosomes. The microbiological activity was measured using a zone of inhibition, which was measured in millimeters.

These findings provide valuable insight into the potential use of Ni (II) chelates for the development of highly effective antibacterial and antifungal agents.

IV. CONCLUSION

Based on the investigations conducted, it can be inferred that the ligand [MThBAP] acts as a negatively charged, tridentate ligand that is coordinated to the metal ion through the azomethine (N), N=N and phenolic, anionic (O) bonds. The analytical data of the complexes match well with their molecular formula. A comparison between the IR value of the free ligand and the Ni metal complex showed that the nitrogen atom of azomethine and N=N, and the oxygen atom of phenolic of the tridentate Schiff bases are involved in bond formation with Ni(II) ions. This was confirmed by two new peaks in the IR of the complex (M-N and M-O). The job method and mole ratio method confirmed the metal-to-ligand ratio of 1:2, further supporting the complex. The electronic spectral and magnetic susceptibility measurements indicate that the mononuclear Ni (II) complex with MThBAP is octahedral. The ligand parameters of the complex provide information about the covalent nature of the complex. Based on these findings, the structures have been proposed for the complex, which is in good agreement with theoretical considerations (as shown in Fig.5). The metal complex exhibits enhanced microbiological activities when compared to the ligand.

Table V- Antibacterial and Antifungal activity

Complexes	Cultures					
	S.a	S.a	E.c	K.p	C.a	Asp
Schiff's base	21	8	9	7	3	-
NiL2	33	11	10	11	4	3

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