SYNTHESIS, CHARACTERISATION AND ANTIMICROBIAL STUDIES OF VANADIUM (IV) AND NICKEL (II) COMPLEXES WITH N–(4-NITROBENZYLIDENE)-1- NAPHTHYLAMINE

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ABSTRACT : The Schiff base ligand (4-nitrobenzylidene)-1-naphthylamine was prepared by the condensation between 4-nitrobenzaldehyde and 1-naphthylamine.The Ni and V complexes of the corresponding ligand were prepared and was characterized by different methods like CHN analysis, IR and UV spectra. The biological activity of the complexes were studied by conducting antibacterial and antifungal studies. The V and Ni complexes shows antibacterial and antifungal activities at different concentrations. From CHN analysis and IR spectral data, the structure of Ni complex is found to be tetrahedral and that of Vanadium complex is triagonal bipyramidal structure.

INDEX TERMS : Schiff base, 4-nitrobenzaldehyde, 1-naphthylamine, antibacterial, antifungal.

INTRODUCTION

Coordination chemistry is the study of compounds that have a central metal surrounded by molecules or ions, known as ligands. The ligands are attached to the central metal atom by dative bonds which is also known as coordinate bonds. Werner's Coordination Theory put forward by Alfred Werner in 1893 was the first attempt to explain the bonding in coordination complexes and he got Nobel prize in 1913 for his worthful contribution. The theory explains the nature of bonding in complexes. As per the theory the metal shows two different sorts of valencies - primary valency is the number of charges on the complex ion and secondary valency is number of ligand atoms coordinated to the metal atom (coordination number).

Metal complexes often have spectacular colors. These colors are caused by electronic transitions by the absorption of light. Most transitions that are related to colored metal complexes are either d-d transitions or charge transfer bands. There are certain methods used to verify the presence of complex ions. This can be achieved by observing the colour, solubility, absorption spectrum, magnetic properties etc. The properties of the complex is entirely different from that of the free atoms.

1.1. SCHIFF BASES

Schiff base is a class of organic compounds which are also known as azomethine, anilino or imines. The Schiff bases were act as an excellent ligands to form complexes with various transition metal ions due to its ability to donate proton/electron and number of bonding sites which guides the different stereo- chemical structures and also provides the information related to the kinetic stability and thermodynamic stabilities of the complexes.

Schiff bases are the special types of organic compounds, named after the work done by a German chemist Hugo-Schiff , he will condensed carbonyl compounds with primary amines to get azomethine group (>CH = N-) as follows:

$$>C = O + H_2N - R \rightarrow C\,H = N - R + H_2O$$

1.2.Formation of Schiff Base

A Schiff base is a nitrogen analog of an aldehyde or ketone in which the C=O group is replaced by C=N-R group. It is usually formed by the condensation of an aldehyde or ketone with a primary amine according to the following scheme :

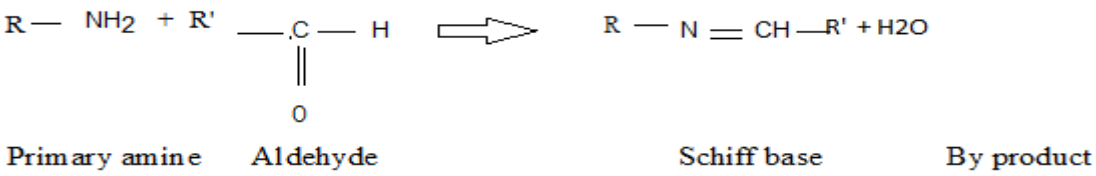


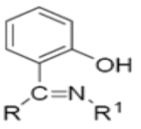
fig 1.2a. Process of condensation of aldehyde and primary amine

1.3.GENERAL PROPERTIES OF SCHIFF BASES

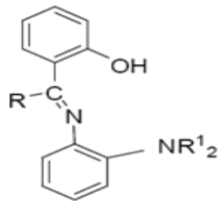
Schiff bases are generally obtained as pale-yellow or orange needles having melting point in the range 160-240^{o}c. Above this temperature, they will undergo decomposition. Schiff bases are insoluble in water but soluble in aqueous alkaline solution and fairly soluble in benzene, dioxane and chloroform. They are more soluble in hot methanol and ethanol. Hence these solvents are used for recrystallization. [5]IR spectra of the Schiff's bases normally shows C=N stretching frequency between 1562-1650 cm^{-1}. In metal complexes, this band is shifted towards a lower frequency due to the coordination of metal with nitrogen of azomethine group of the Schiff base.

1.4.SCHIFF'S BASE AS LIGAND

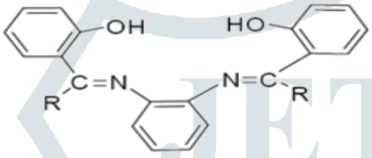
Ligands which possess two or more donating groups will share more than one pair of electrons with a single metal ion by coordinating around the central metal ion. These ligands are called as multidentate ligands, usually bidentate, tridentate etc.



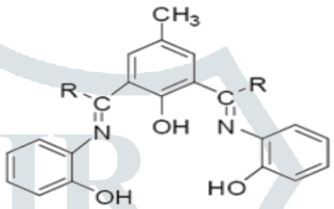
Bidentate (1)



Tridentate (2)



Tetradentate (3)



Pentadentate (4)

1.6.CHOICE OF METAL IONS.

Transition metals are generally used for the synthesis of Schiff base complexes. The 3d metal are generally used for the synthesis of metal complexes in the field of coordination chemistry. This is due to the following reasons:

1. Cations of transition metals are unique in their tendency to form complexes with several ions.

2. Due to smaller size and high positive charge density, cations can easily accept lone pair of electrons from the molecules.

3. Transition metals have lone pair of electrons to accept lone pair of electrons. Also, the d orbitals are projected out of the periphery of the atom, so the electrons occupy them strongly influence the environment. This is why d orbitals are less shielded than f orbitals.

1.8.APPLICATION OF SCHIFF BASES

Schiff bases are tremendously used in different fields such as agriculture, industries, Pharmaceuticals, medicine and so on.

- Schiff Bases used as Catalysts
- Antimicrobial activity
- Anti -Fungal And Anti-Viral action
- Anti-Oxidant Action
- Polymer and Dyes
- Anti-Fertility and Enzymatic Action

- Synergistic Action on Insecticides
- Plant Growth Regulator
- In Non-Linear Optical Devices
- In Medicinal Chemistry
- Photography
- Antitumour and Cytotoxic Activities

MATERIALS AND METHODS

4.1: MATERIALS.

All reagents used for the synthesis of ligand and complexes are of commercial grade and they are directly used without further purification. 4-nitrobenzaldehyde is used as aldehyde and 1-naphthylamine is used as the amine and the solvent used is methanol.

4.2: INSTRUMENTS

Instruments used in this investigation are given below:

1. Schimadzu IR prestige-20 spectrometer

2. Schimadzu UV-2450 A Spectrometer

3. Systronies conductivity meter 304

4. Gouy type magnetic balance

5. Vario-III CHN elemental analyser

The purity of the compounds were checked by Thin Layer Chromatography (0.5 mm thickness) using silica gel-G and spots were visualized by exposing the dry plates to iodine vapours.

4.3: METHODS

4.3.1: SYNTHESIS OF N-(4-NITROBENZYLIDENE)-1-NAPHTHYLAMINE

4-nitrobenzaldehyde (0.151g, 0.001M) dissolved in 20 ml methanol and 1-naphthylamine in 20 ml methanol was mixed well. The resulting mixture was refluxed for about four hours. On cooling, yellow crystals were separated from the solution. These crystals were filtered and dried.

4.3.2: SYNTHESIS OF METAL COMPLEXES

(a) Synthesis of Vanadium complex

Ammonium metavanadate has been used as a synthetic intermediate for the preparation of V(IV) complex. The methanolic solution of the ligand (0.001M) was just heated to dissolve the ligand and to this, methanolic solution of the metal (0.0005M) was added so that the ratio will be 1:2 and the mixture is refluxed for four hours. The pH is maintained between 6-7. Then the volume was reduced to half its initial volume. After concentration, the solution was cooled and the dark yellow complex formed is separated out. It is filtered, washed with methanol and dried in vaccum.

(b) Synthesis of Nickel complex

Nickel Chloride has been used as a synthetic intermediate for the preparation of Ni(II) complex. The methanolic solution of the ligand (0.001M) was just heated to dissolve the ligand and to this, methanolic solution of the metal (0.001M) was added so that the ratio will be 1:1 and the mixture is refluxed for four hours. The pH is maintained between 6-7. Then the volume was reduced to half its initial volume. After concentration, the solution was cooled and the complex formed is separated out. It is filtered, washed with methanol and dried in vaccum.

table 4.3 : physical properties of ligand and complexes.

Compound	Colour	Solubility	Yield(%)
Ligand	Yellow	DMSO	76.3%
Ni Complex	Dark Yellow	DMSO	78%
V Complex	Dark Yellow	DMSO	80%

4.4: INFRARED SPECTRA

IR spectroscopy is a spectroscopic technique used to identify chemical compounds and to investigate sample composition. A comparison of IR stretching frequency of the ligand and its metal complexes gives an idea about the mode of linking in complexes. On complexation with metal ions, the characteristic IR frequencies of the coordinating groups were influenced by the force constant of the metal-ligand bond resulting in shifting of the group frequencies. The shift were useful in identifying the coordination sites.

The IR spectra of the solid samples were recorded in Schimadzu IR prestige - 20 spectrometer in the range of 4000-400 cm^{-1}. Potassium bromide disc method was employed for sample preparation.

4.5: ELECTRONIC ABSORPTION SPECTROSCOPY

Electronic absorption spectroscopy is used to study the stereochemistry of the complexes. By using d-d transitions of the metal ions and their absorption spectra, it is possible to determine the ligand field splitting of the d orbitals of the metal ions. Metal-ligand interaction can be studied from the UV-Visible spectra of free ligand and its metal complexes.

The UV-Visible spectra of the samples in DMSO solution were recorded in Schimadzu UV-2450 A spectrometer in the range of 200-800 nm.

4.6: MOLAR CONDUCTANCE

Molar conductance of transition metal complexes were determined in DMF and N,N'-dimethyl formamide at room temperature using a systolic conductivity Meter 304. The cell constant of the conductivity cell was 1 cm^{-1}. The concentration of the solution was around 1x 10^{-3} M. The molar conductance is measured by the equation,

$$M = 1000\ k/c$$

Where c = concentration of the solution in mol/L

k = conductivity (specific conductance)

4.7 : CHN ANALYSIS

CHN analysis was done in Vario-III CHN elemental analyser at the Saif, Cochin University of Science and Technology, Kochi.

4.8: MAGNETIC SUSCEPTIBILITY

Magnetic susceptibility measurements of the metal complexes were studied at room temperature (300K) by using Magway MSB Mk1 magnetic susceptibility balance. Diamagnetic corrections were computed using Pascal's constant by adding the diamagnetic contribution of various atoms and structural units. Gram susceptibility was calculated using the formula,

$$X_g = (\alpha + \beta F) / W$$

Where α = Air Displacement Constant

β = Tube Constant

F = change in weight in milligram

W = weight of sample in gram

The effective magnetic moment μ_{eff} was calculated using the formula,

$$\mu_{eff} = 2.84\sqrt{X_m T}$$

where X_m = molar susceptibility corrected for diamagnetism and T = Temperature, 293 K

4.9: ESTIMATION OF VANADIUM

The most convenient method for the estimation of Vanadium is a volumetric process. The Vanadium is first obtained in acid solution as vanadate, reduced to the tetravalent state by one of several reducing agents which are available. The solution is then titrated in the presence of sulphuric acid with potassium permanganate solution, which quantitatively oxidises the lower Vanadium salt to Vanadate. Diphenylamine sulfonic acid is used as indicator. Using sulfur dioxide to effect the reduction, the following reaction take place.

$$2VO_3 + SO_2 + H_2SO_4 \rightarrow 2VOSO + 2H_2O$$

$$10VOSO_4 + 2KMnO_4 + 12H_2O \rightarrow 10HVO_3 + K_2SO_4 + 2MnSO_4 + 7H_2O$$

Excess of sulphur dioxide is removed before the titration by boiling the reduced solution in an atmosphere of CO_2. The titration is completed when a pink end point stable atleast for one minute.

4.10 : ESTIMATION OF NICKEL

The nickel is precipitated as nickel dimethyl glyoxime by adding alcoholic solution of dimethyl glyoxime and then adding a slight excess of aqueous ammonia solution.

$$NiSO_4 + 2C_4H_8O_2N_2 \longrightarrow Ni(C_4H_7O_2N_2)_2 + H_2SO_4$$

The complex solution was first digested with HCl – H_2SO_4 mixture and is made upto 25 mL and 10 mL is pipetted out into a 100 ml beaker. Then it is diluted to 50 mL The solution is heated to 80^0C and 10 mL of 1% dimethyl glyoxime in rectified spirit is added immediately followed by dilute ammonia solution drop wise directly to the solution. The precipitate is heated for 20 – 30 minutes and the supernatant liquid is tested for complete precipitation. The precipitate is allowed to cool and then filtered through a previously weighed sintered crucible (IG_4). The precipitate is washed with cold water and dried at 110-120^{0}C. It is allowed to cool in desicator and weighed.

4.11: ANTIBACTERIAL ACTIVITY

AGAR- WELL DIFFUSION METHOD

PRINCIPLE

The antimicrobials present in the samples are allowed to diffuse out into the medium and interact in a plate freshly seeded with the test organisms. The resulting zones of inhibition will be uniformly circular as there will be a confluent lawn of growth. The diameter of zone of inhibition can be measured in **millimeters**.

PROCEDURE

Petriplates containing 20ml Muller Hinton Agar Medium were seeded with bacterial culture of *E.coli, Pseudomonas aeroginosa, Streptococcus mutans a*nd *Staphylococcus aureus* (growth of culture adjusted according to McFards Standard, 0.5%). Wells of approximately 10mm was bored using a well cutter and different concentrations of sample such as 250µg/mL, 500µg/mL and 1000µg/mL were added. The plates were then incubated at 37°C

for 24 hours. The antibacterial activity was assayed by measuring the diameter of the inhibition zone formed around the well (NCCLS, 1993). Streptomycin was used as a positive control.

Reference: National Committee for Clinical Laboratory Standards. (1993a). Performance Standards for Antimicrobial Disk Susceptibility Tests—Fifth Edition: Approved Standard M2-A5. NCCLS, Villanova, PA.

4.12: ANTIFUNGAL ACTIVITY

AGAR- WELL DIFFUSION METHOD

PRINCIPLE

In order to access the biological significance and ability of the sample, the antifungal activity was determined by Agar well diffusion method. The antifungals present in the samples are allowed to diffuse out into the medium and interact in a plate freshly seeded with the test organisms. The resulting zones of inhibition will be uniformly circular as there will be a confluent lawn of growth. The diameter of zone of inhibition can be measured in millimeters.

PROCEDURE

Potato Dextrose agar plates were prepared and overnight grown species of fungus, *Aspergillus niger and Candida albicans* were swabbed. Wells of approximately 10mm was bored using a well cutter and samples of different concentrations such as 250µg/mL, 500µg/mL and 1000µg/mL were added. The zone of inhibition was measured after overnight incubation at room temperature and compared with that of standard antimycotic (Clotrimazole). (NCCLS, 1993).

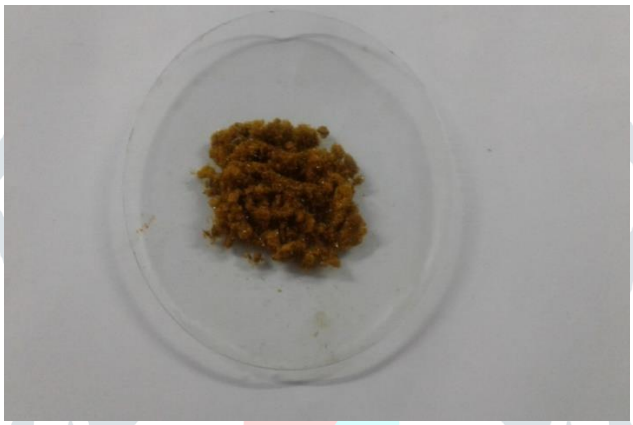
RESULT AND DISCUSSION

Schiff base derived from 4-nitrobenzaldehyde and 1-naphthyl amine has been examined as ligand for Nickel in +2 oxidation state and vanadium in +4 oxidation state.

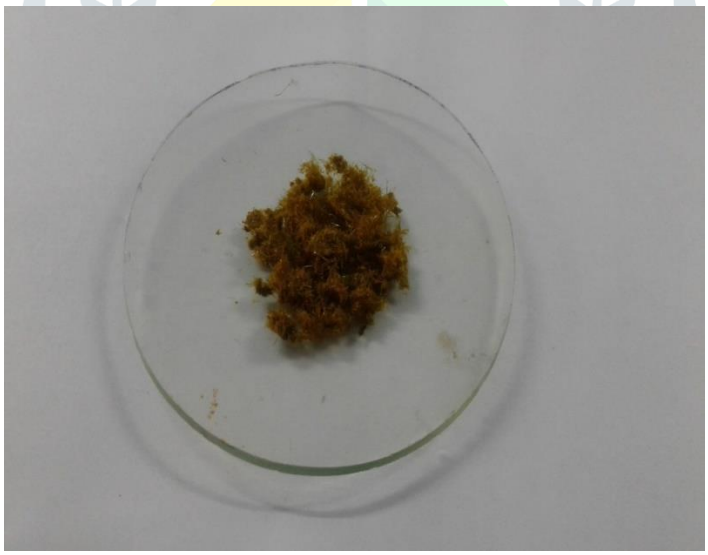
5.1: GENERAL PROPERTIES

Both V(IV) and Ni(II) complexes are dark yellow coloured and are stable in air. Both the complexes are sensitive to light and decomposes when exposed to light. The two complexes are insoluble in water and readily soluble in methanol, ethanol, Dimethyl Sulphoxide.

Ligand



Vanadium Complex



Nickel Complex

5.2: ANALYSIS

Metal content in the complexes were determined by standard method after decomposing the complexes with hydrochloric acid-sulphuric acid mixture. The metal complexes of Schiff base ligand were prepared by the stoichiometric reaction of the corresponding metal and ligand in 1:2 ratio. The gravimetric analysis of Nickel complex and

volumetric analysis of Vanadium complex are found to be successful and from these, the metal content in the complexes are determined.

5.3: MOLAR CONDUCTANCE

Molar conductance of 10^{-3} M solutions of the metal complexes at 25^0C were measured in DMF and N,N'-dimethyl formamide. The molar conductance values of Ni(II) and V(IV) complexes under investigation are found to be 158.5 $\Omega^{-1}cm^2mol^{-1}$ and 159 $\Omega^{-1}cm^2mol^{-1}$ respectively. The molar conductance value shows that the Ni(II) and V(IV) complex was electrolytic in nature. Because there is no charged species in the complex to neutralize the charge of the central metal ion.

Table 5.3(a) : Properties of complexes

Complex	colour	Yield(%)	Molecular weight	Magnetic moment (BM)	Molar conductance ($\Omega^{-1}cm^2mol^{-1}$)
Ni(II) complex	Dark yellow	78%	611.3114	3.84	158.5
V(IV) complex	Dark yellow	80%	635.5615	1.8	159

5.4: MAGNETIC MEASUREMENTS

Magnetic susceptibility of the complexes were determined using Magway MSB Mk1 magnetic susceptibility balance. The measurements were made at room temperature. Table 5.3 (a) shows the effective magnetic moments calculated from the magnetic susceptibility which is corrected for diamagnetic corrections. Some indications about the structure, geometry and coordination of the complexes can be obtained from magnetic moment values.

The Ni(II) and V(IV) complexes are paramagnetic in nature.

The magnetic moment values of Ni(II) and V(IV) complexes are found to be 3.84 BM and 1.91 BM. From this, it is clear that the Ni(II) complex has tetrahedral and V(IV) complex has distorted triagonal bipyramidal structure.

5.5 : CHN ANALYSIS

The experimental percentage values of carbon, hydrogen and nitrogen in the two complexes found out by CHN analysis were similar to the percentage calculated from the

theoretical aspects. So from CHN analysis, the structure and denticity of the ligand and complex can be confirmed.

Table 5.5 (a) : CHN Analysis

COMPLEX	C%		H%		N%	
	experimental	calculated	experimental	Calculated	experimental	calculated
Ni complex	50.22%	52.48%	5.75%	4.6639%	7.38%	7.201%
V complex	66.76%	62.29%	4.16%	4.3050%	9.60%	8.546%

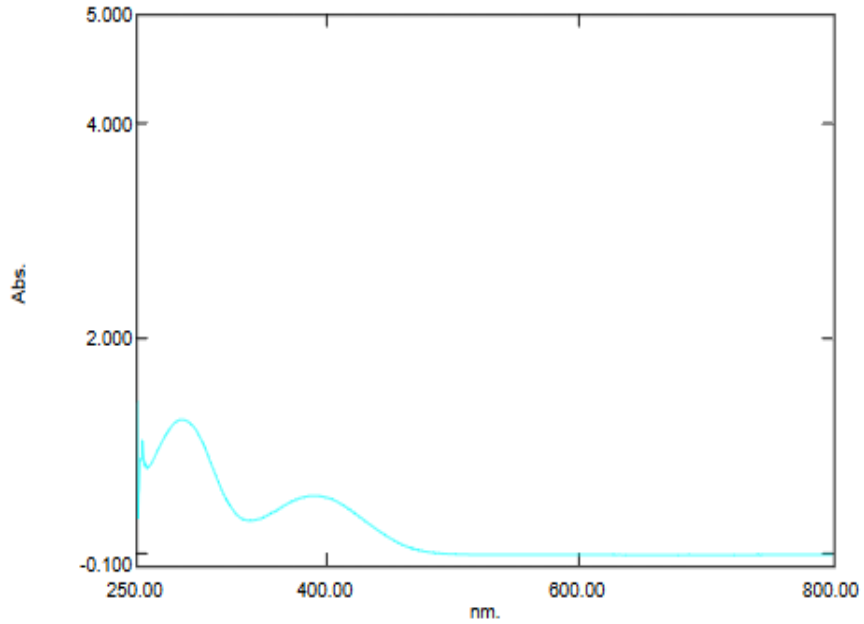
Here, the CHN analysis values are in good agreement with the calculated percentage of carbon, hydrogen and nitrogen in the complex. So by using CHN analysis, it is easy to confirm that the ligand is monodentate.The structures of the complexes can also be confirmed from CHN analysis value. The structure of Ni complex is found to be tetrahedral and that of Vanadium complex is triagonal bipyramidal.

5.6: ELECTRONIC SPECTRA

The electronic spectra are often helpful in the evaluation of results furnished by other methods of analysis. The electronic spectral bands of the ligand and complexes was recorded over the range of 200-800 nm in DMSO .

5.6 a : ELECTRONIC SPECTRUM OF LIGAND

DST FIST UV Vis
spectrophotometer Department of
Chemistry
Sample. 4 nitro ligand
5.10.2018



No.	P/V	Wavelength	Abs.	Description
1	↑	390.00	0.542	
2	↑	286.50	1.247	
3	↑	226.50	1.204	
4	↑	213.50	1.712	
5	↑	204.50	2.988	
6	↓	339.50	0.313	
7	↓	237.00	0.137	
8	↓	224.00	-4.000	
9	↓	211.50	-0.142	

Ultraviolet spectra of the ligands recorded in DMSO showed strong bands around 286.5 nm and at 339.5 nm region which confirms the presence of benzenoid and azomethine linkages, which are characteristic of Π—›Π* and n—›Π* transition respectively.

ELECTRONIC SPECTRA OF COMPLEXES.

5.6 b : Nickel Complex

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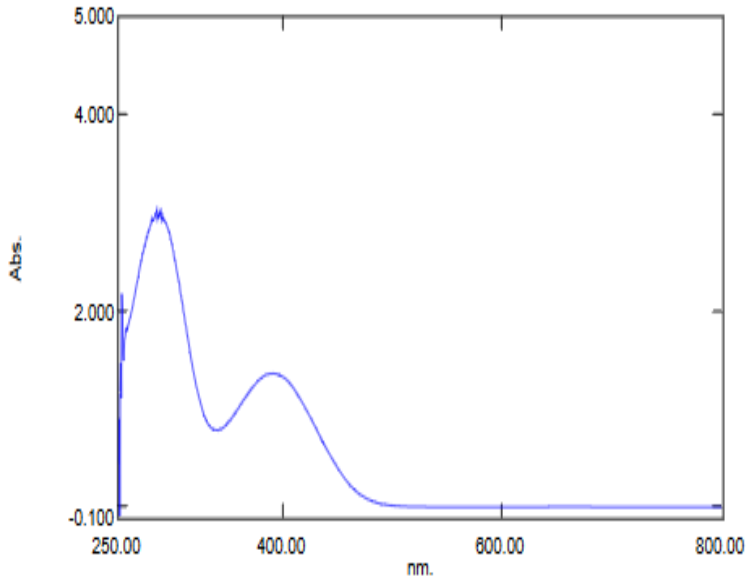
spectrophotometer Department of
Chemistry
Sample. nickel
5.10.2018

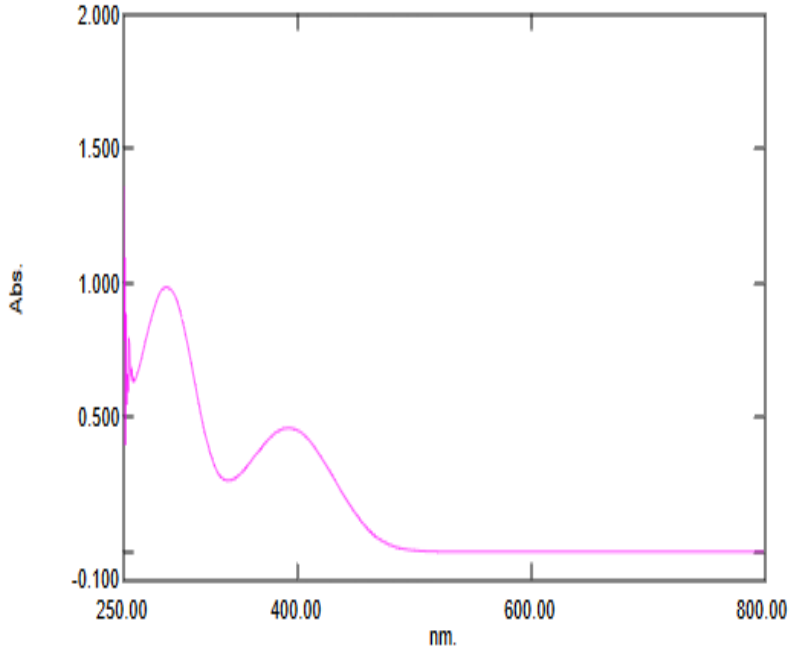


No.	P/V	Wavelength	Abs.	Description
1	↑	391.50	1.362	
2	↑	285.50	3.032	
3	↑	217.50	3.168	
4	↓	340.50	0.782	
5	↓	251.50	-0.263	

Ultraviolet spectra of the Nickel complex recorded in DMSO showed strong bands around 285.5 nm and at 340.5 nm region which confirms the presence of benzenoid and azomethine linkages, which are characteristic of Π—›Π* and n—›Π* transition respectively.

5.6 c : Vanadium complex

DST FIST UV Vis spectrophotometer Department of Chemistry
Sample. vanadium 4
5.10.2018



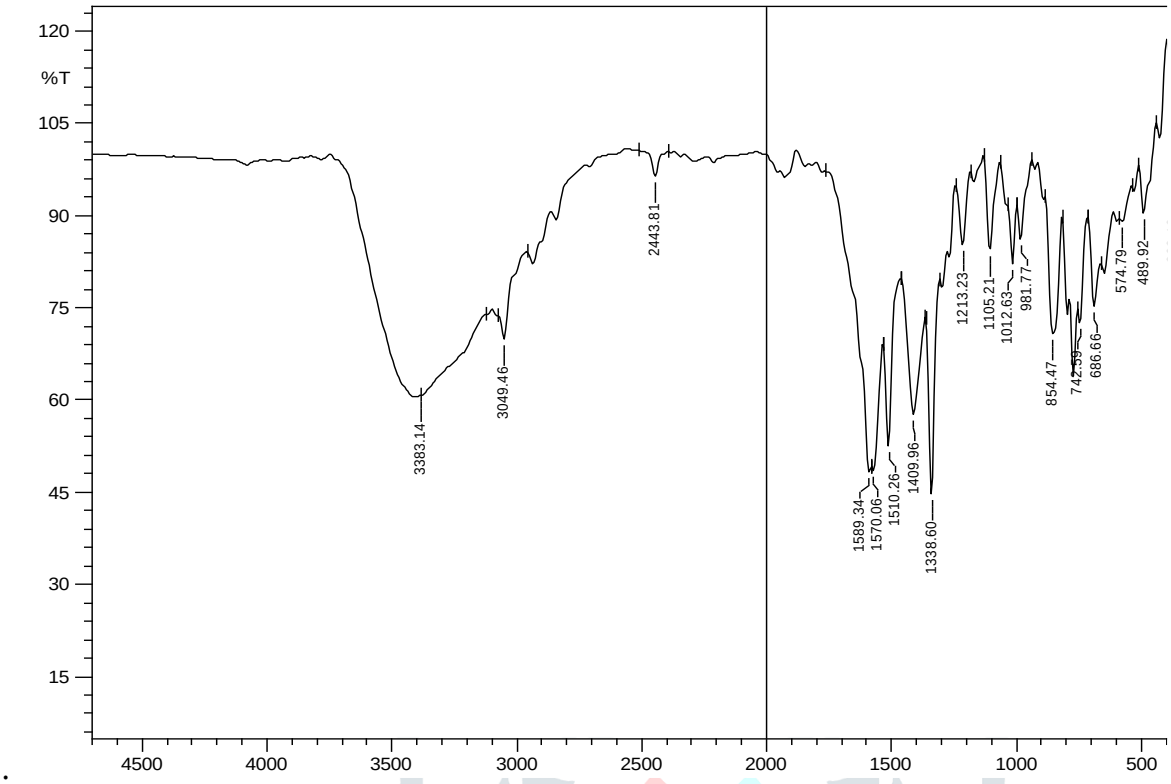
No.	P/V	Wavelength	Abs.	Descripti
1	↑	392.00	0.460	
2	↑	286.50	0.986	
3	↓	339.50	0.265	
4	↓	259.00	0.634	

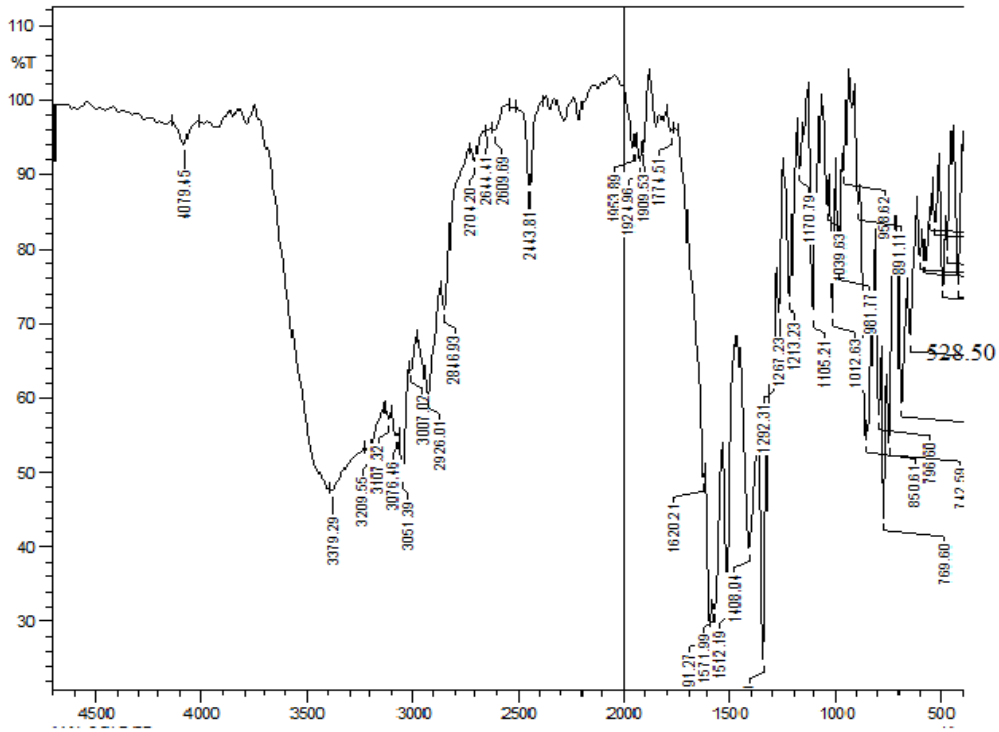
Ultraviolet spectra of the Vanadium complex recorded in DMSO showed strong bands around 286.5 nm and at 339.5 nm region which confirms the presence of benzenoid and azomethine linkages, which are characteristic of Π—›Π* and n—›Π* transition respectively.

5.7: INFRARED SPECTRA

The IR spectral data of the Schiff base ligand and its metal complexes are presented in table 5.6(a) and 5.6(b). The spectra of the complexes were compared with that of the free ligand to determine the coordination sites which involve in chelation

(a) IR SPECTRUM OF LIGAND





(b) IR SPECTRUM OF NICKEL COMPLEX

Table 5.7(b) : Comparison of IR spectra of ligand and Ni Complex

Ligand	Ni Complex	Assignment
-	3379	Coordinated water molecule
3049	3007	=CH
1589.34	1591.27	C=N
1510.26	1512.19	C=C
1570 1338.60	1571 1292.31	N=O (Sym. Bending) N=O (asym. Bending)
742.59	742.59	monosubstituted
-	528.50	Ni-N

In the IR spectrum of the ligand, a medium strength band is observed at 3049 cm^{-1} which corresponds to =CH group. In the complex, it is shifted to a lower frequency of 3007 cm^{-1}. The intense band near 1589 cm^{-1} can be attributed to υ (C=N) of the ligand. It is shifted to a higher frequency of 1591.27 cm^{-1} which indicates the presence of coordination. Two peaks at 1570 cm^{-1} and 1338.60 cm^{-1} corresponds to the symmetric and asymmetric bending of NO_2 group. In Ni complex, a strong band is obtained in 3379 cm^{-1} which indicates the presence of coordinated water molecule. The peak at 528.50 cm^{-1} can be attributed to Ni-N bond which is absent in the free ligand. This peak at 528.50 cm^{-1} confirms the coordination in Ni complex.

(c) IR SPECTRUM OF VANADIUM COMPLEX

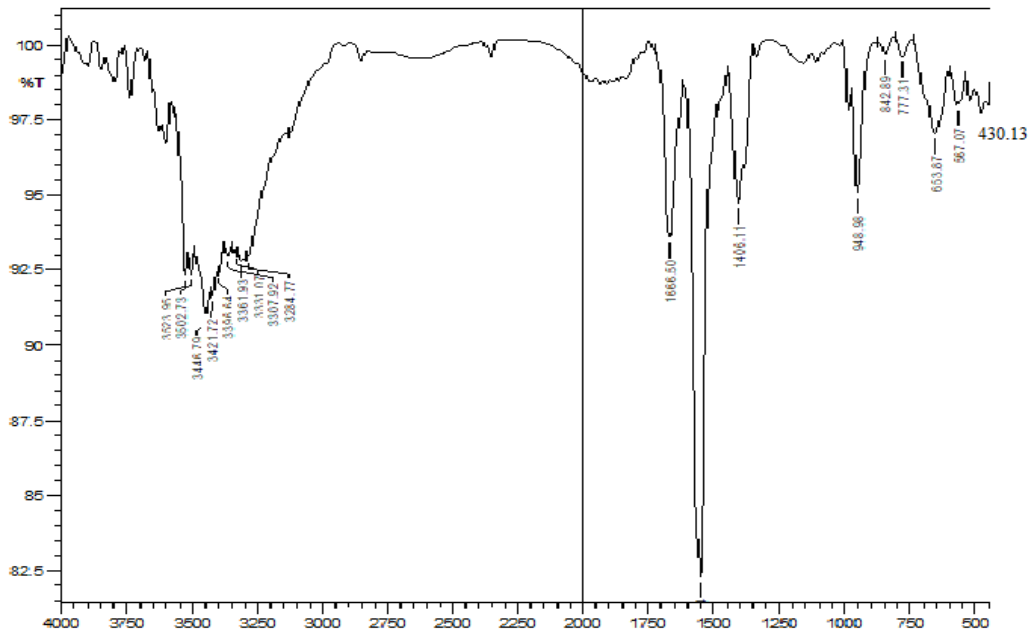


Table 5.7(c) : Comparison of IR spectra of ligand and Vanadium Complex

Ligand	V Complex	Assignment
-	3412	Coordinated water molecule
3049	3016	=CH
1589.34	1672.28	C=N
1510.26	1526.28	C=C
1338.60	1340.53	N=O (asym.bending)
742.59	713.66	monosubstituted
-	430.13	V-N
-	918.12	V=O

In the IR spectrum of the ligand, a medium strength band is observed at 3049 cm^{-1} which corresponds to =CH group. In the complex, it is shifted to a lower frequency of 3016 cm^{-1}. The intense band near 1589.34 cm^{-1} corresponds to υ (C=N) of the ligand. It is shifted to a higher frequency of 1672.28 cm^{-1} which indicates the presence of coordination. The peak at 1338.60 cm^{-1} indicates the asymmetric bending of NO_2 group.The peak at 3412 cm^{-1} confirms the presence of coordinated water molecule. The strong bands at 430.13 cm^{-1} and 918.12 cm^{-1} attribute to V-N and V=O bonds which are absent in the free ligand. These peaks confirms the formation of metal complexes.

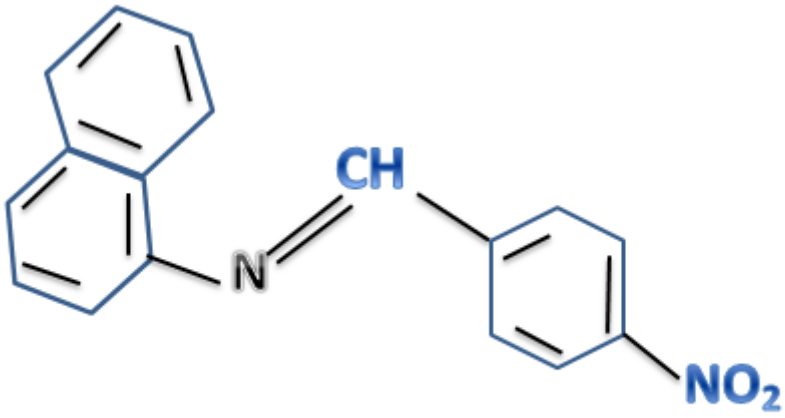


Fig 1 : Structure of Ligand

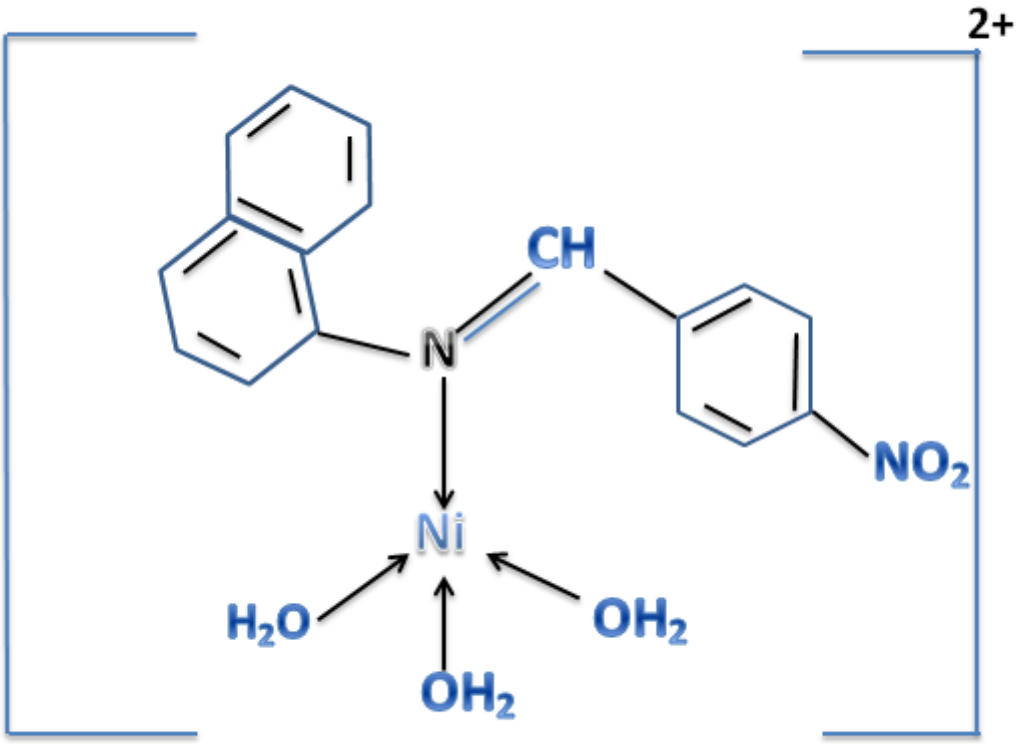


Fig 2 : Structure of Ni complex

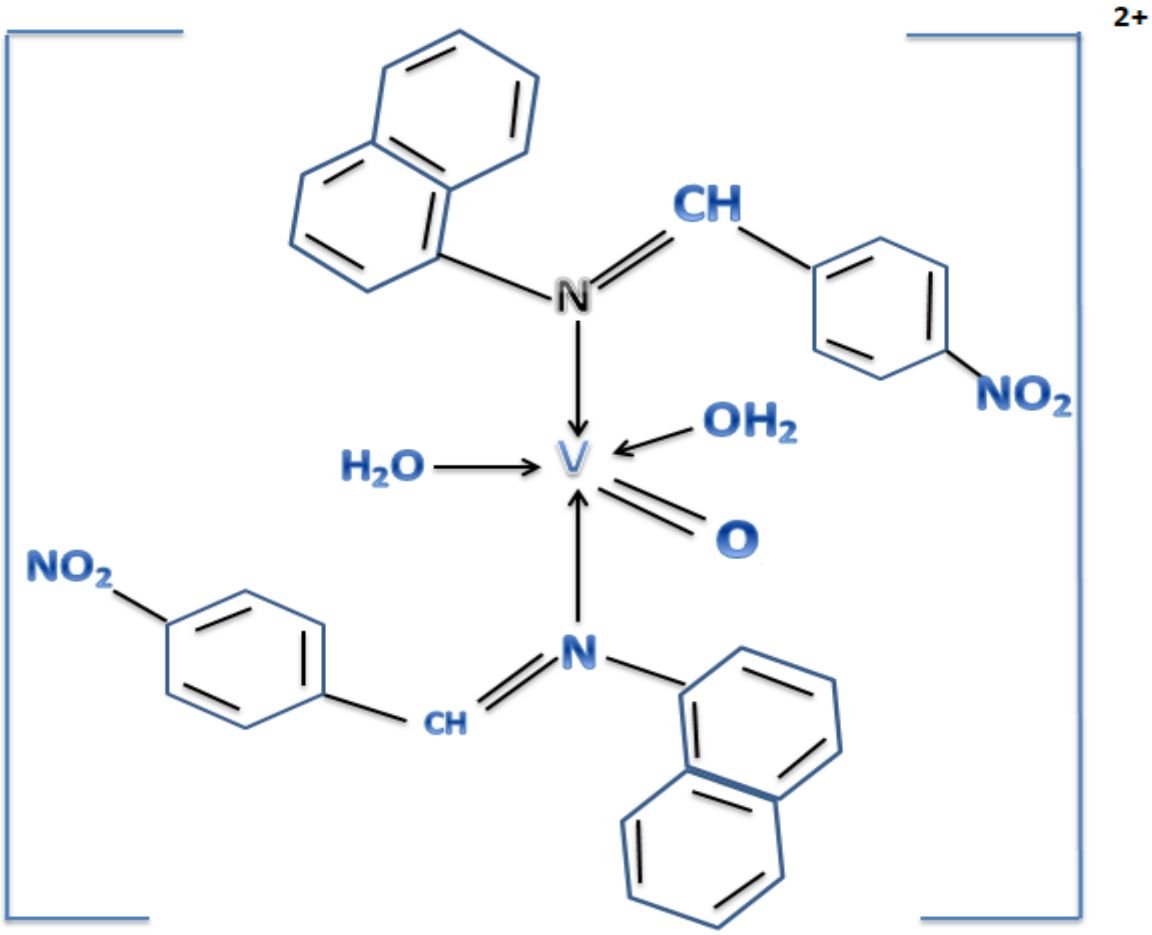


Fig 3 : Structure of V Complex

5.8: ANTIBACTERIAL AND ANTIFUNGAL STUDIES.

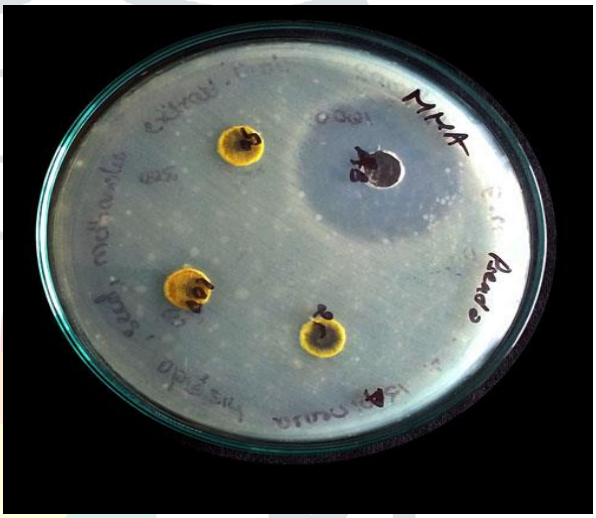
Antibacterial studies

The antibacterial and antifugal studies were conducted by comparing the zone of inhibition of a standard bacterial agent with the complexes and their extend of antimicrobial activity is measured.

5.8 (a): ANTIBACTERIAL STUDIES OF VANADIUM COMPLEXES

E COLI

PSEUDOMONAS AERGINOSA



STREPTOCOCCUS MUTANS

STAPHYLOCOCCUS AUREUS

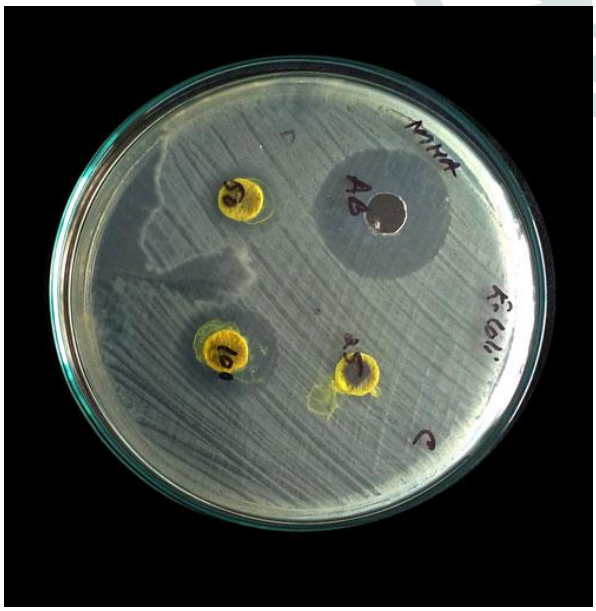
Table 5.8 (a) : Antibacterial Studies Of Vanadium Complexes

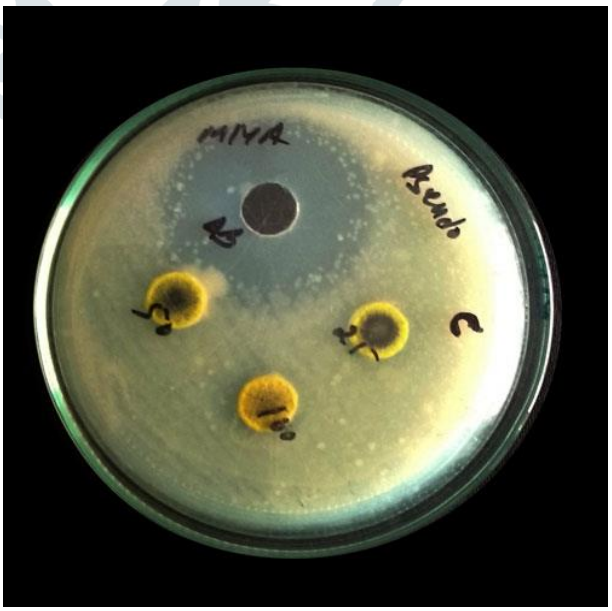
ORGANISM	ANTIBACTERIAL AGENT	ZONE OF INHIBITION			
		AGENT	250	500	1000
E Coli	Streptomycin	26	-	-	-
Pseudomonas aerginosa	Streptomycin	30	-	-	-
Streptococcus mutans	Streptomycin	26	-	-	11
Staphylococcus Aureus	Streptomycin	27	11	11	12

4-nitro vanadium complex is inactive for Gram negative Bacteria like E Coli and Pseudomonas aerginosa.The vanadium complex is inactive for Gram positive stain Streptococcus mutans at the concentration of 250 and 500. But it is slightly active at the concentration of 1000. The complex shows slight activity for the Gram positive stain Staphylococcus aureus at 250, 500 and 1000 concentrations.

5.8(b) :ANTIBACTERIAL STUDIES OF NICKEL COMPLEXES

E COLI **PSEUDOMONAS AERGINOSA**





STREPTOCOCCUS MUTANS **STAPHYLOCOCCUS AUREUS**

Table 5.8 (b) : Antibacterial Studies Of Nickel Complexes

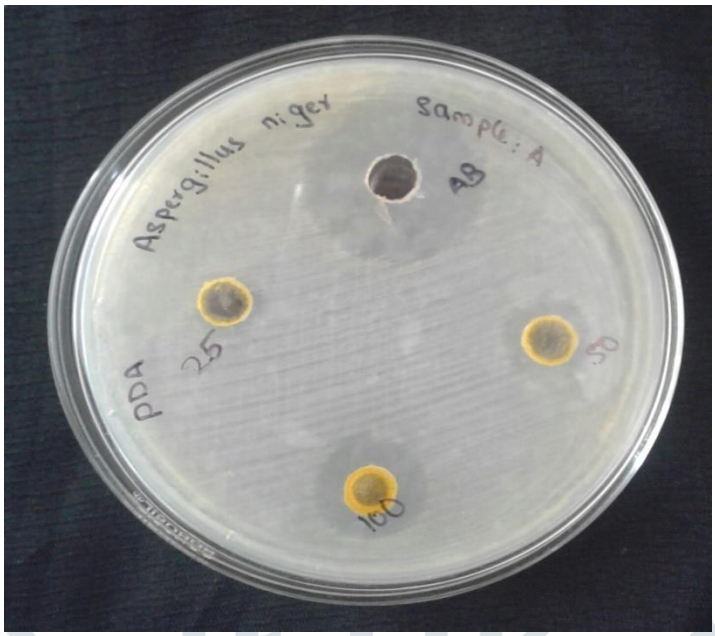
ORGANISM	ANTIBACTERIAL AGENT	ZONE OF INHIBITION			
		AGENT	250	500	1000
E Coli	Streptomycin	20	-	12	18
Pseudomonas Aerginosa	Streptomycin	30	-	10	11
Streptococcus mutans	Streptomycin	26	-	-	11
Staphylococcus aureus	Streptomycin	27	-	-	11

4-nitro nickel complex is inactive for Gram negative Bacteria like E Coli at the concentration of 250 and shows shows moderate activity at the concentrations of 500 and 1000. The complex is inactive for Pseudomonas aerginosa at 250 concentration and shows slight activity at 500 and 1000 concentration. The nickel complex is inactive for Gram positive stain Streptococcus mutans at the concentration of 250 and 500. But it is slightly active at the concentration of 1000. The complex is inactive for the Gram positive stain Staphylococcus aureus at 250, 500 and shows slight activity at 1000 concentrations.

Antifungal studies

5.8(c) : ANTIFUNGAL RESULTS OF VANADIUM COMPLEX

ASPERGILLUS NIGER



CANDIDA ALBICANS

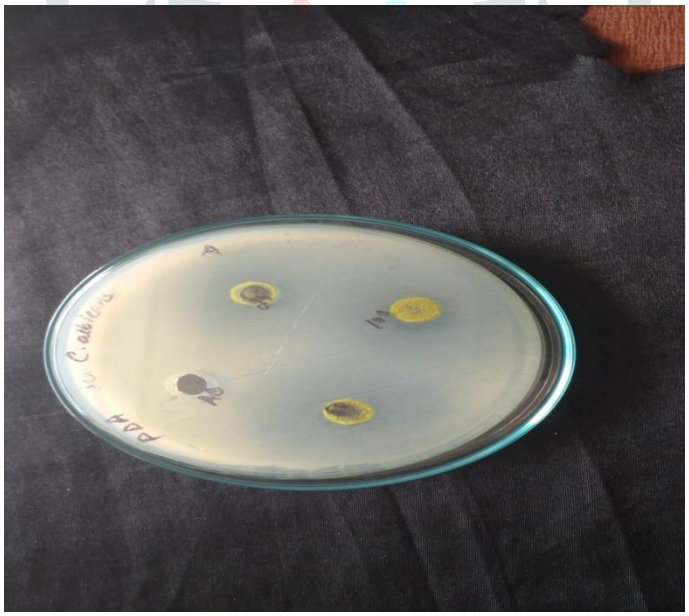


Table 5.8(c) : Antifungal Results Of Vanadium Complex

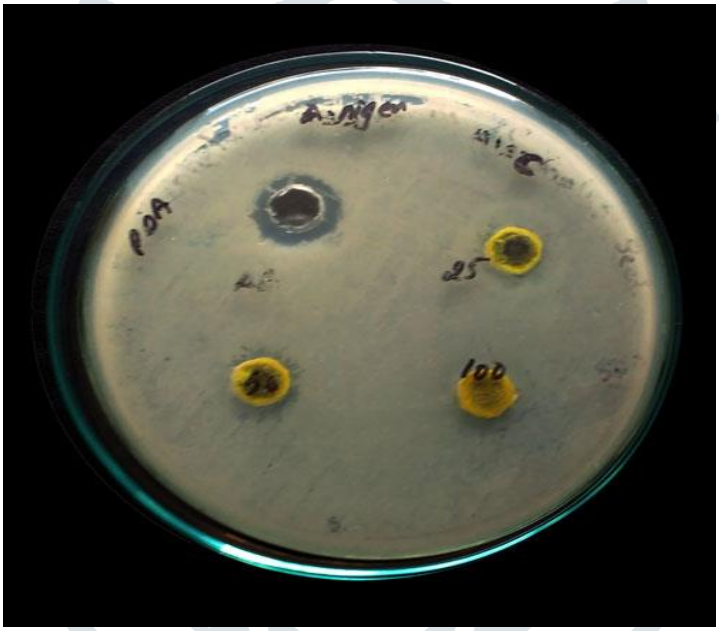
ORGANISM	ANTIFUNGAL AGENT	ZONE OF INHIBITION			
		AGENT	250	500	1000
Aspergillus	Clotrimazole	30	_	13	19

niger					
Candida albicans	Clotrimazole	15	_	11	12

The standard antifungal agent used for the antifungal studies is Clotrimazole. 4 nitro – vanadium complex is inactive for Aspergillus niger fugus at the concentration of 250. But the complex is slightly active at 500 and 1000 concentration.

5.8 (d) : ANTIFUNGAL RESULTS OF NICKEL COMPLEX

ASPERGILLUS NIGER



CANDIDA ALBICANS

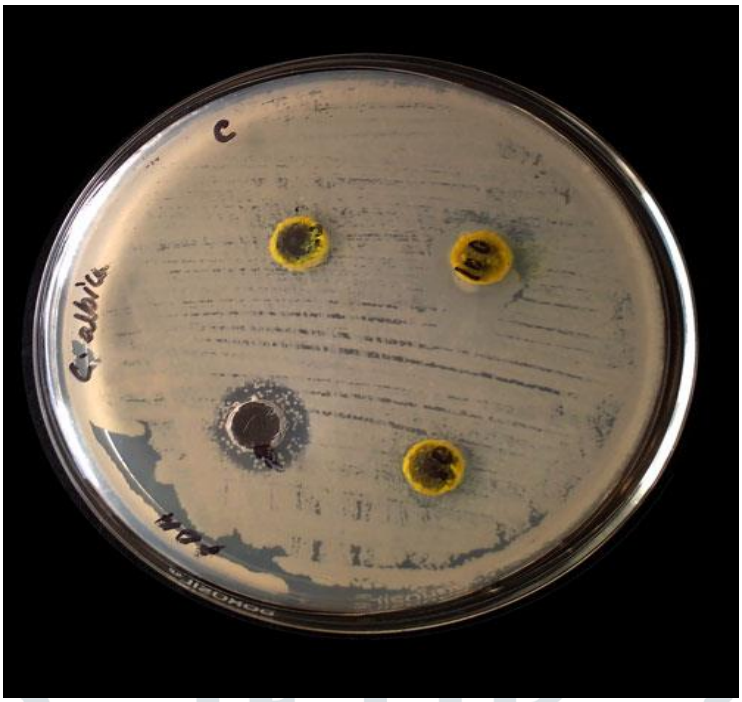


Table 5.8(d) : Antifungal Results Of Nickel Complex

ORGANISM	STANDARD ANTIFUNGAL AGENT	ZONE OF INHIBITION			
		AGENT	250	500	1000
Aspergillus niger	Clotrimazole	15	_	_	_
Candida albicans	Clotrimazole	15	_	_	_

The standard antifungal agent used for the antifungal studies is Clotrimazole. The 4 nitro – nickel complex is inactive for both Aspergillus niger and Candida albicans at all concentrations of 250, 500 and 1000.

SUMMARY AND CONCLUSION

The Schiff base ligand, (4- nitrobenzylidene)-1-naphthyl amine has been synthesized by the condensation of 4-nitrobenzaldehye and 1-naphthyl amine. It is yellow in colour and having 76.3% yield and soluble in dimethyl sulphoxide. The spectral data of the ligand concluded that the ligand is monodentate.

The Nickel complex of this ligand is dark yellow in colour and having 78% yield and soluble in DMSO. The molar conductance value of this complex were found to be 158.5 $\Omega^{-1}cm^2mol^{-1}$. This value indicates that the complex is electrolytic in nature. The magnetic moment value of this complex is 3.84 BM which suggests that the complex has tetrahedral structure. The appearance of new bands at 528.50 cm^{-1} in the IR spectrum is assigned to Ni-N stretching vibration. The spectral data of the ligand concluded that the ligand is monodentate.

The oxovanadium complex of this ligand is dark yellow in colour and having 80% yield and soluble in DMSO. The molar conductance value of this complex were found to be 159 $\Omega^{-1}cm^2mol^{-1}$. This value indicates that the complex is electrolytic in nature. The magnetic moment value of this complex is 1.91 BM which suggests that the complex has triagonal bipyramidal structure. The appearance of new bands at 430.13 cm^{-1} and 918.12 cm^{-1} in the IR spectrum are assigned to V-N and V=O stretching vibrations.

The UV spectra of ligand and complexes shows characteristic absorption at the range of 285 nm and at 340 nm region which confirms the presence of benzenoid and azomethine linkages, which are characteristic of Π—›Π* and n—›Π* transition respectively.

The antibacterial and antifugal studies of complexes were conducted by comparing the zone of inhibition of a standard bacterial agent with the complexes and their extend of antimicrobial activity is measured. 4-nitro vanadium complex is inactive for Gram negative Bacteria like E Coli and Pseudomonas aerginosa.The vanadium complex is inactive for Gram positive stain Streptococcus mutans at the concentration of 250 and 500. But it is slightly active at the concentration of 1000. The complex shows slight activity for the Gram positive stain Staphylococcus aureus at 250, 500 and 1000 concentrations.

4-nitro nickel complex is inactive for Gram negative Bacteria like E Coli at the concentration of 250 and shows shows moderate activity at the concentrations of 500 and 1000. The complex is inactive for Pseudomonas aerginosa at 250 concentration and shows slight activity at 500 and 1000 concentration. The nickel complex is inactive for Gram positive stain Streptococcus mutans at the concentration of 250 and 500. But it is slightly active at the concentration of 1000. The complex is inactive for the Gram positive stain Staphylococcus aureus at 250, 500 and shows slight activity at 1000 concentrations.

The standard antifungal agent used for the antifungal studies is Clotrimazole. 4 nitro – vanadium complex is inactive for Aspergillus niger fugus at the concentration of 250. But the complex is slightly active at 500 and 1000 concentration. The 4 nitro – nickel

complex is inactive for both Aspergillus niger and Candida albicans at all concentrations of 250, 500 and 1000.

The experimental percentage values of carbon, hydrogen and nitrogen in the two complexes found out by CHN analysis were similar to the percentage calculated from the theoretical aspects. So from CHN analysis, the structure and denticity of the ligand and complex can be confirmed.

ACKNOWLEDGEMENT

I take this opportunity to express my deep sense of gratitude to my supervising guide **Dr.A.MARIA STARVIN,** Professor, Department of Chemistry, Christian College,Kattakada for his valuable suggestions, immense guidance, constant encouragement and constructive criticism throughout this project work.

I take this opportunity to express my deep sense of gratitude to **Sri.CHARLES FINNY S.L**, Head of the Department of Chemistry, Christian College,Kattakada for his valuable suggestions and constant encouragement throughout this project work.

I also express my thanks to **Dr. DINESH BABU**, Professor, Department of Chemistry, Women's College, Vazhuthacaud, for helping me to take IR and UV spectra.

I also thank **BIOGENIX RESEARCH CENTRE MUDAVANMUGAL, POOJAPURA AND SOPHISTICATED TEST AND COMPUTATION CENTRE, CUSAT, COCHIN** for helping me to conduct Antimicrobial (antibacterial and antifungal) studies and CHN Analysis.

I acknowledge the **Department of Science and Technology (DST)** for providing fund under the scheme of **"Fund for Improvement of S & T infrastructure in universities and higher education institutions (FIST)"** for providing fund to purchase equipment in instrumentation lab and also for computer facilities in Computer and Networking Lab.

We also acknowledge **Kerala State Council For Science And Technology** for giving fund to the Department of Chemistry under the scheme of Selective Augmentation of Research and Development.

I affectionately remember the valuable and timely help rendered by the non-teaching staffs of our department and all those who have helped us directly or indirectly while carrying out the project work.

I am extending my immense thanks to my parents, classmates and friends for their support and encouragement during the course of work.

Above all, I immensely thank God Almighty for giving me the strength and peace for carrying out the work and also for a successful completion.

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