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RESEARCH ARTICLE

SYNTHESIS, CHARACTERISATION AND ANTIBACTERIAL STUDIES OF OXOVANADIUM COMPLEXES OF BIDENTATE SCHIFF BASES

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ABSTRACT

Three vanadyl complexes of bidentate Schiff bases from vanillin with anthranilic acid (VA), 4-aminoantipyrine with vanillin (4AV) as well as 4-aminoantipyrine with furfural (4AF) have been synthesized and characterized using elemental analysis, FTIR, UV-Vis, ESR spectroscopic methods, magnetic susceptibility, molar conductivity measurements and thermal analysis. These complexes have the general molecular formula $[VO(VA)_2.H_2O]$, which is found to be a non-electrolyte, while the others with general molecular formula $[VO(L)_2.(H_2O)]SO_4$, where L= 4AV or 4AF were 1:1 electrolytes. The infra red spectra reveals the involvement of coordination of azomethine nitrogen and oxygen atom of carboxylate ion from VA to oxovanadium (IV) while azomethine nitrogen and oxygen atom from cyclic keto group present in pyrazoline ring are coordinated to VO (IV) from 4AV and 4AF. These complexes have a distorted octahedral geometry and yield V_2O_5 as a stable residue during thermal decomposition in static air atmosphere with loss of H_2O molecule in the first step. These complexes have been screened for their antibacterial activity against organisms like E.Coli, S.aureus, Serattai SP, P.aeruginosa and B.Cereus.

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INTRODUCTION

Schiff base are very important molecules in biological systems. They have wide range of application in bioinorganic, coordination and catalysis field (Kumar and Singh, 2006). They also have interesting properties and biological functions such as being models for mettalloproteins & oxygen carrier systems, in catalyzing organic oxidation ion reaction (Nagalakshmi, 2005). An ever increasing research interest in the coordination chemistry of vanadium has been aroused not only due to its exhibiting a range of oxidation states from +5 to -1 but also from the fascinating structural novelties and complexities exhibited by vanadium complex and their immense biological (Aureliano and Gandara, 2005; Shobu and Joseph, 2007) industrial (Hoppe and Limberg, 2007; Zhang and Yamamoto, 2007) and medicinal applications (Melchior *et al.,* 2001; Papaioannou *et al.,* 2004). Recently it has been shown that the involvement of periodic elements particularly vanadium (Ranjbar, 2004) with organic moieties having nitrogen and sulphur atoms, plays a crucial role in designing a potential molecule of specific use. Keeping the above facts in mind and in continuation of our research work on vanadium complexes (Gayathri and Suresh, 2017) with Schiff bases, the present paper describes the synthesis and characterization of vanadium complexes with Schiff base derived from condensation of vanillin with anthranilic acid, 4-aminoantipyrine with vanillin and 4-aminoantipyrine with furfural, and their characterization using physio-chemical methods, molar conductance and antibacterial properties.

Experimental

Vanillin, 4-aminoantipyrine, furfural, anthranilic acid, vanadylsulphate, ethylalcohol etc. were purchased from Merck and used as such without any further purification. The elemental analysis (CHNS) were performed using EL elemental analyser at Central Electrochemical Research Institute (CECRI). The FT infrared spectra were recorded in the range 4000-400cm^{-1} on a Schimadzu FTIR 8400S spectrometer using KBr pellet techniques. Electronic (UV-VIS) spectra were recorded on a Schimadzu UV-spectrophototmeter in the range 200- 1100 cm^{-1}using DMSO solvent. The thermal analyses were carried out using universal V4.5A Thermal analysis instrumentation in an atmosphere of static air with a heating rate of 10K/min. The ESR spectra of various Schiff base complexes were recorded on a JES-X_3 series in the scan range of 2300-4300 Gauss at LNT. The magnetic susceptibility measurements of the complexes were carried out using Guoy balance. The molar conductance was measured on ELICO-CM180 using DMSO as the solvent at

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room temperature. The antibacterial studies was carried out with disc diffusion method.

Synthesis of Schiff bases

a) From vanillin and anthranilic acid (VA) (Suresh and Prakash, 2010)

A ethanolic solution of vanillin (4.564g, 0.02mol,25mL) was added to an ethanolic solution of anthranilic acid (4.564g, 0.02mol,25mL). The contents were stirred for 10 minutes and refluxed on a water bath for 4-5 hours. Subsequently they were cooled and poured into crushed ice. The oily liquid over the water is allowed to stay aside for some time, as crystals of the Schiff base starts to appear. The resulting solid product is washed with distilled water and ethanol several times and is then recrystallised with ethanol (Brain *et al.,* 1966; Dubey *et al.,* 2006; Vogel's, 1978). The scheme of preparation is shown in Figure 1.

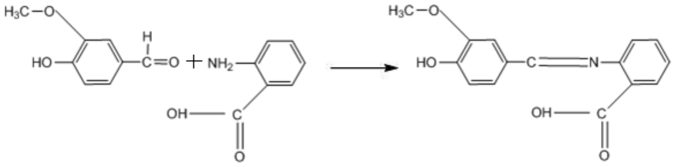

Figure 1. Schematic representation of the synthesis of ligand VA

b) From 4-aminoantipyrine and vanillin (4AV) (Suresh and Prakash, 2010)

The same method was adopted for this in which an ethanolic solution of 4-aminoantipyrine (1-phenyl-2,3-dimethyl-4-aminopyrazol-5-one) (6.09g, 0.03mol,25mL) was added to an ethanolic solution of vanillin (4.56g, 0.03 mol, 25mL). Its scheme is represented in Figure 2.

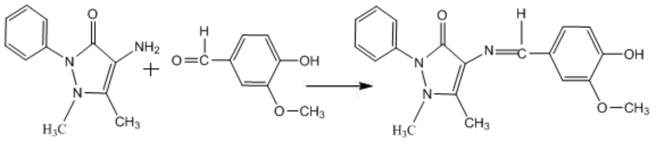

Figure 2. Schematic representation of the synthesis ligand 4AV

b) From 4-aminoantipyrine and furfural (4AF)

The same method was adopted for this in which an ethanolic solution of 4-aminoantipyrine (1-phenyl-2,3-dimethyl-4-aminopyrazol-5-one) (6.09g, 0.03mol,25mL) was added to an ethanolic solution of furfural (4.56g, 0.03 mol, 25mL). Its scheme is represented in Figure 3.

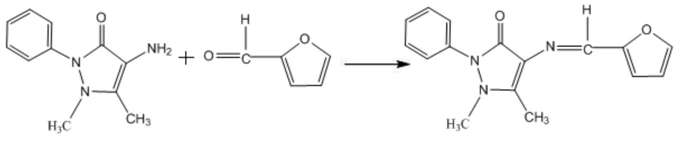

Figure 3. Schematic representation of the synthesis of ligand 4AF

Synthesis of Vanadyl complexes

- The vanadium complexes were prepared by refluxing 1:2 molar ethanolic mixture of the vanadium salt, $VOSO_4.5H_2O$ (2.530g, 0.01 mol) with the ligand VA (5.42g, 0.02 mol) for 5 hours. The contents were cooled and allowed to crystallize. The complex was collected by filtration and then washed several times with hot ethanol until the mother liquor become colourless. The resultant product were dried in air and stored in a dessicator over anhydrous calcium chloride under vacuum.
- The same method was adopted for other complexes in which the ligand 4AV (6.74g, 0.02 mol), 4AF (5.62g, 0.02 mol) was added to the vanadium salt $VOSO_4.5H_2O$ (2.530g, 0.01 mol) in ethanolic medium.

RESULTS AND DISCUSSION

Elemental Analysis and Molar Conductance studies

The Analytical data (Table 1) suggests 1:2 metal: ligand stoichiometry for all the three complexes with SO_4^{2-} as counter ion in case of 4AV and 4AF complexes. The synthesized complexes have the molecular formula $[VO(VA)_2.H_2O]$, $[VO(4AV)_2.H_2O]SO_4$ and $[VO(4AF)_2.H_2O]SO_4$.. These complexes are stable and dark green or orange in colour. The VA complex have molar conductance in the range of 26.5 S cm^2mol^{-1} indicating non-electrolytic nature whereas the complexes of 4AV and 4AF show molar conductance in the range 83.5-88 S cm^2mol^{-1} indicating they are 1:1 electrolytic in nature . The physical, analytical and conductivity data are presented in Table 1

Infrared spectra

The ν(OH) due to water absent in the spectrum of the ligand VA appears at 3441 cm^{-1} in the vanadyl complex $[VO(VA)_2.(H_2O)]$ indicates the presence of coordinated water in the complex. The spectrum of the ligand 1 (VA) shows $\nu_{C=N}$ bands observed at 1558 cm^{-1} was found to be shifted to lower frequencies 1503 cm^{-1}, attributed to the coordination of N atom of azomethine group to the metal ion. This is further substantiated by the presence of a new band around 450 cm^{-1} due to ν_{M-N}. The frequency corresponding to ν_{COO}^{-} present in the ligand 1481 cm^{-1} is shifted to lower wave number 1470 cm^{-1} in the complex, indicating the bond formation through the carboxylate COO^- anion (Gehad *et al.,* 2005; Kalagouda *et al.,* 2007). This is further substantiated by the presence of a new band around 591cm^{-1} due to ν_{M-O}. Another absorption band at 965 cm^{-1} corresponds to ν_{V-O} which was found to be absent in the IR spectrum of the ligand. A broad peak appears in the IR spectrum of $[VO(4AV)_2.H_2O]SO_4$ at 3230 cm^{-1} was due to coordinated water molecules and usually $\nu_{C=O}$ of cyclic keto group present in pyrazolone ring appears at 1670 cm^{-1}. In our compound it was found to be at 1650 cm^{-1} and shifted to 1602 cm^{-1} in the vanadyl complex and $\nu_{C=N}$ (azomethine group) at 1624 cm^{-1} in the free ligand was shifted to 1579 cm^{-1}. These data suggested that the coordination is through oxygen atom and azomethine group. The new band appears at 450 cm^{-1} was due to ν_{M-N} and 590 cm^{-1} was due to ν_{M-O}. Another absorption band at 970 cm^{-1} corresponds to $\nu_{V=O}$ which was found to be absent in the IR spectrum of the ligand. The peaks observed at 987 cm^{-1}(ν_1), 1131 cm-1 (ν_3) and 632 cm^{-1} (ν_4) are due to non-coordinated sulphato ions indicate the presence of SO_4^{2-} outside the coordination sphere (Nakamoto, 1986; Yadava *et al.,* 2015). The spectrum of the ligand 3 (4AF) shows $\nu_{C=N}$ bands observed at 1602 cm^{-1} was found to be shifted to lower frequencies 1593 cm^{-1}, attributed to the coordination of N atom of azomethine group to the metal ion.

Table 1. Physical , analytical and conductivity data of ligands and complexes

Compound	Mol.Wt	Colour	Yield	Elemental Analysis (%) Found (Calc)					Λ_m S cm^{2}mol^{-1}
				C	H	N	S	M	
$C_{15}H_{13}$ NO_4(VA)	271.26	Yellow	80%	67.11 (66.41)	4.900 (4.830)	5.188 (5.164)			
[VO(VA)$_2$.H_2O]	625.46	Dark Green	75%	59.73 (57.60)	5.551 (4.190)	5.295 (4.476)		11.2 (10.7)	26.5
$C_{19}H_{19}N_3O_3$(4AV)	337.37	Yellow	75%	68.05 (67.63)	12.87 (12.45)	5.890 (5.677)		-	
[VO(4AV)$_2$.H_2O]SO_4	855.64	Dark Green	70%	54.79 (53.33)	4.053 (4.476	9.993 (9.817)	4.166 (3.739)	8.100 7.823)	88
$C_{16}H_{15}N_3O_2$(4AF)	281.28	Orange	75%	68.79 (68.31)	5.421 (5.375)	15.30 (14.93)			
[VO(4AF)$_2$.H_2O]SO_4	743.16	Brown	70%	50.15 (51.71)	4.468 (4.069)	11.32(11.30)	4.394 (4.305)	9.234 (9.007)	83.5

Table 2. Infra-red spectral data of the Schiff bases and its vanadyl complexes

Compound	ν (OH) (H_2O)	ν(OH) (PHENOLIC)	$\nu_{C=O}$	$\nu_{C=N}$	ν_{COO^-}	ν_{M-O}	ν_{M-N}	$\nu_{V=O}$	ν_{SO4}^{2-}		
									ν_1	ν_3	ν_4
$C_{15}H_{13}$ NO_4(VA)	-	3239	-	1558	1481	-	-	-	-	-	-
[VO(VA)$_2$.H_2O]	3441	2955	-	1503	1471	591	450	965	-	-	-
$C_{19}H_{19}N_3O_3$(4AV)	-	3064	1650	1624	-	-	-	-	-	-	-
[VO(4AV)$_2$.H_2O]SO_4	3250	3063	1602	1579	-	590	450	970	987	1131	632
$C_{16}H_{15}N_3O_2$(4AF)	-	3045	1646	1602	-	-	-	-	-	-	-
[VO(4AF)$_2$$H_2O$]$SO_4$	3332	3046	1652	1593	-	591	445	970	950	1135	663

Table 3. UV-Visible Spectral Data and Magnetic moments of the complexes

Compound	Absorption Max (cm^{-1})	Assignments	μ (BM)
$C_{15}H_{13}$ NO_4(VA)	35460	n→π*	
	27397	π→π*	
[VO(VA)$_2$.H_2O]	23474,	$^2B_2$→$^2A_1$,	
	15822	$^2B_2$→$2B_1$	1.88 BM
	11312	$^2B_2$→^2E	
$C_{19}H_{19}N_3O_3$(4AV)	35460	n→π*	
	27397	π→π*	
[VO(4AV)$_2$.H_2O]SO_4	24630,	$^2B_2$→$^2A_1$,	
	15673	$^2B_2$→$2B_1$	1.79 BM
	11235	$^2B_2$→^2E	
$C_{16}H_{15}N_3O_2$(4AF)	35460	n→π*	
	27397	π→π*	
[VO(4AF)$_2$.H_2O]SO_4	24038	$^2B_2$→$^2A_1$,	1.73 BM
	16233	$^2B_2$→$2B_1$	
	12285	$^2B_2$→^2E	

This is further substantiated by the presence of a new band around 445 cm^{-1} due to ν_{M-N}. The frequency corresponding to $\nu_{C=O}$ of pyrazolone present in the ligand 1646 cm^{-1} is shifted to wave number 1652 cm^{-1} in the complexes, indicating the bond formation through the carbonyl group (Lever, 1984). This is further substantiated by the presence of a new band around 591 cm^{-1} due to ν_{M-O}. Another absorption band at 970 cm^{-1} corresponds to $\nu_{V=O}$ which was found to be absent in the IR spectrum of the ligand. A broad new peak observed at 3332 cm^{-1} in the vanadyl complex [VO(4AF)$_2$.(H_2O)]SO_4, shows the presence of coordinated water molecule in the complex. The peaks observed at 950 cm^{-1}(ν_1), 1135 cm^{-1} (ν_3) and 663 cm^{-1} (ν_4) are due to non-coordinated sulphato ions indicate the presence of SO_4^{2-} outside the coordination sphere sphere (Nakamoto, 1986; Yadava *et al.,* 2015). The IR data are presented in Table 2

UV-VIS Electronic spectra and Magnetic Susceptibility Measurement

The free ligands shows absorption maxima appearing around 35460 cm^{-1} and 27397 cm^{-1} for VA, 4AV and 4AF was due to n→ π* and π→π* transition respectively. In complex [VO(VA)$_2$.H_2O] three higher energy bands appears around 23474, 15822 and 11312 cm^{-1} assigned to $^2B_2$→$^2A_1$, $^2B_2$→$2B_1$ and $^2B_2$→^2E. In complex [VO(4AV)$_2$.H_2O]SO_4 three higher energy bands appears 24630,15673 and 11235 cm^{-1} due to $^2B_2$→$^2A_1$, $^2B_2$→$2B_1$ and $^2B_2$→^2E.

In complex [VO(4AF)$_2$.H_2O]SO_4 three higher energy bands appears around 24038, 16233 and 12285 cm^{-1} assigned to $^2B_2$→$^2A_1$, $^2B_2$→$2B_1$ and $^2B_2$→^2E respectively. These absorption bands are typical of distorted octahedral geometry (Gudasi and Goudar, 1994) VO^{2+} species. The magnetic moments of these complexes were seen to be in the range 1.73-1.88 BM which are also supporting the distorted octahedral geometry of VO^{2+} complexes, a 3d^1 system (Gudasi and Goudar, 1994). The electronic spectral details and magnetic moments were presented in Table 3.

Thermal Studies

The thermo gram of the vanadyl complex [VO(4AV)$_2$.(H_2O)]SO_4 is given in Figure 4. This compound is found to be stable only up to 60ºC. The first stage of decomposition is seen starting at 60ºC and progressing till 80ºC with a mass loss of 2.09%. The second stage of decomposition starts immediately at 80ºC and ends at 125ºC with a loss in mass corresponding to 6.66% only. The third stage of decomposition is seen initiating immediately at 125ºC and ending at 340ºC with a weight loss of 69.9%. The last stage of decomposition starts at 340ºC, progressing till 820ºC with a mass loss of 10.7% leaving behind a residue with a mass of 10.65%. Above 820ºC, the residue is seen stable in static air atmosphere. From the molecular structure and geometry of the complex from physic chemical methods, it is possible to explain the decomposition feature of the oxovanadium complex. The first stage of decomposition is due to the breaking of vanadium-water bond which eliminates one water molecule from the complex. The calculated value of 2.11% was in good agreement with observed value of 2.09%. During the second stage the breaking of bonds between metal and ligands takes place and which continues in the third step also. The total mass loss during the second and third stages corresponds to 76.56% which is in good agreement with the theoretical value of 78.85% due to the loss of two molecules of the ligands. In the next step the decomposition of sulphate ion takes place leaving behind a stable residue, V_2O_5. The calculated value of 10.8% is in good agreement with the observed value 10.65%. The decomposition stages was summarized as follows.

Stage 1 : $[VO(4AV)_2.(H_2O)]SO_4 \xrightarrow{60^\circ - 80^\circ C} [VO(4AV)_2]SO_4 + H_2O$

Stage 2 & 3: $[VO(4AV)_2]SO_4 \xrightarrow{80^\circ - 340^\circ C} VOSO_4$ + gaseous products

Stage 4 : $VOSO_4 \xrightarrow{340^\circ - 825^\circ C} \frac{1}{2} V_2O_5$ + gaseous products

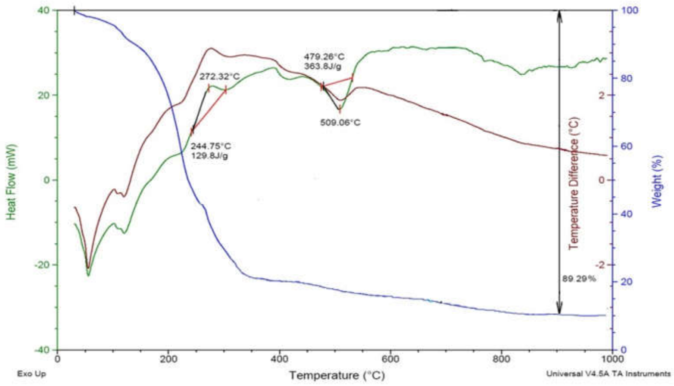

Figure 4. TG/DTA curve of $[VO(4AV)_2.(H_2O)]$ in static air

The thermogram of the $[VO(4AF)_2.(H_2O)]SO_4$ complex was given in Figure 5. The compound was formed to be stable only upto 70ºC, because the first stage of decomposition is seen starting at 70ºC and progressing till 130ºC with a mass loss of 2%. The second stage of decomposition starts only at 150ºC and ends at 260ºC with a loss in mass corresponding to 32%. The third stage of decomposition is initiating immediately at 260ºC, progressing till 320ºC with a mass loss of 41.3%. The final stage of decomposition starts at 320ºC and ends at 675ºC with a mass loss of 12%. Above 675ºC a horizontal plateau was obtained with the mass of the residue of 12.7%. From the physico chemical methods and its molecular structure, the first stage of decomposition is due to the loss of one molecule of water from the complex.

The calculated value of 2.42% is in good agreement with the observed value of 2%. During the second stage the breaking of bonds between vanadium and the schiff base takes place. The observed value of 32% is almost in agreement with the calculated value of 37.85% due to the loss of one molecule of the ligand. In the third stage the loss of another molecule of ligand takes place which also corresponds to 37.85%. The total value of mass loss for the second and third stage 73.3% is in good agreement with the calculated value of 75.7%. In the next stage the decomposition of sulphate takes place leaving behind a stable residue of V_2O_5. The calculated value of 12.24% is in good agreement with the observed value of 12.7%. The decomposition pathway was summarized as follows.

Stage 1 : $[VO(4AF)_2.(H_2O)]SO_4 \xrightarrow{70^\circ - 130^\circ C} [VO(4AF)_2]SO_4 + H_2O$

Stage 2 : $[VO(4AF)_2]SO_4 \xrightarrow{150^\circ - 260^\circ C} [VO(4AF)]SO_4$ + 4AF (gaseous products)

Stage 3 : $[VO(4AF)]SO_4 \xrightarrow{260^\circ - 320^\circ C} VOSO_4$ + 4AF (gaseous products)

Stage 4 : $VOSO_4 \xrightarrow{320^\circ - 675^\circ C} \frac{1}{2} V_2O_5$ + gaseous products

ESR Spectroscopy

The X-band ESR spectra of the oxovanadium complexes were recorded in DMSO solution at liquid nitrogen temperature. In the frozen state, the spectra show two types of resonance components, one set due to parallel while the other set due to perpendicular features, due to interaction between the electron and the vanadium nuclear spin(I = 7/2) (Sharma *et al.,* 2001; Yen, 1969). The observed spectra was not well resolved to calculate the ESR parameters given in Figure 6,7 & 8 and hence $g_{(iso)}$ was calculated for all the three complexes and was found to be 2.0051, 1.9989, 1.9922 respectively for these complexes.

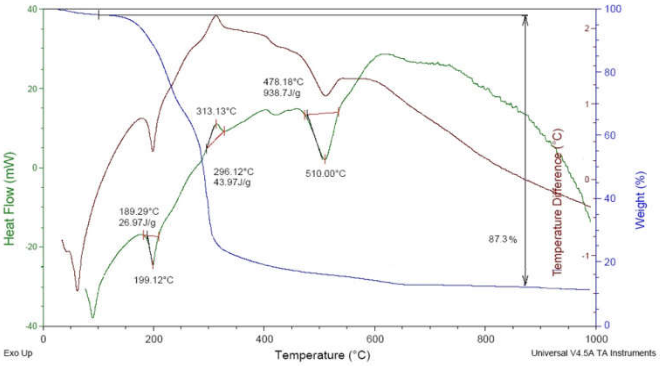

Figure 5. TG/DTA curve of $[VO(4AF)_2.(H_2O)]$ in static air

Antibacterial Studies

The ligands and the vanadyl complexes were assayed for their antimicrobial activities against E.Coli , S,aureus, P.aeruginosa, Serratia (sp) and Bacillus cereus by the disc diffusion method (Raman *et al.,* 2004; Shirin and Mukherjee, 1992). It was found that the complexes have better activity than the ligands explained by the theory of Tweedy. This is probably due to the greater lipophilicity nature of the complexes. Such increased acivity of metal chelates can be explained on the basis of overtones concept and chelation theory. According to overtone's concept of cell permeability the lipid membrane that surrounds the cell favours the passage of lipid soluble materials. On chelation, the polarity of metal ion will be reduced to a greater extent due to overlap of the ligand orbitals and partial sharing of positive charge of the metal ion with donor groups. Therefore the delocalisation of π electrons in the chelate ring increases and enhances the penetration of the complexes into lipid membranes. The antibacterial data are presented in Table 4.

Conclusion

In this work the synthesis and characterisation of Schiff base ligands derived from vanillin, anthranilic acid, furfural and 4-aminoantipyrine and its interaction with vanadium is reported. The metal complexes $[VO(VA)_2.H_2O]$ were non-electrolytic in nature whereas $[VO(4AV)_2.H_2O]SO_4$ and $[VO(4AF)_2.H_2O]SO_4$ are electrolytic in nature.

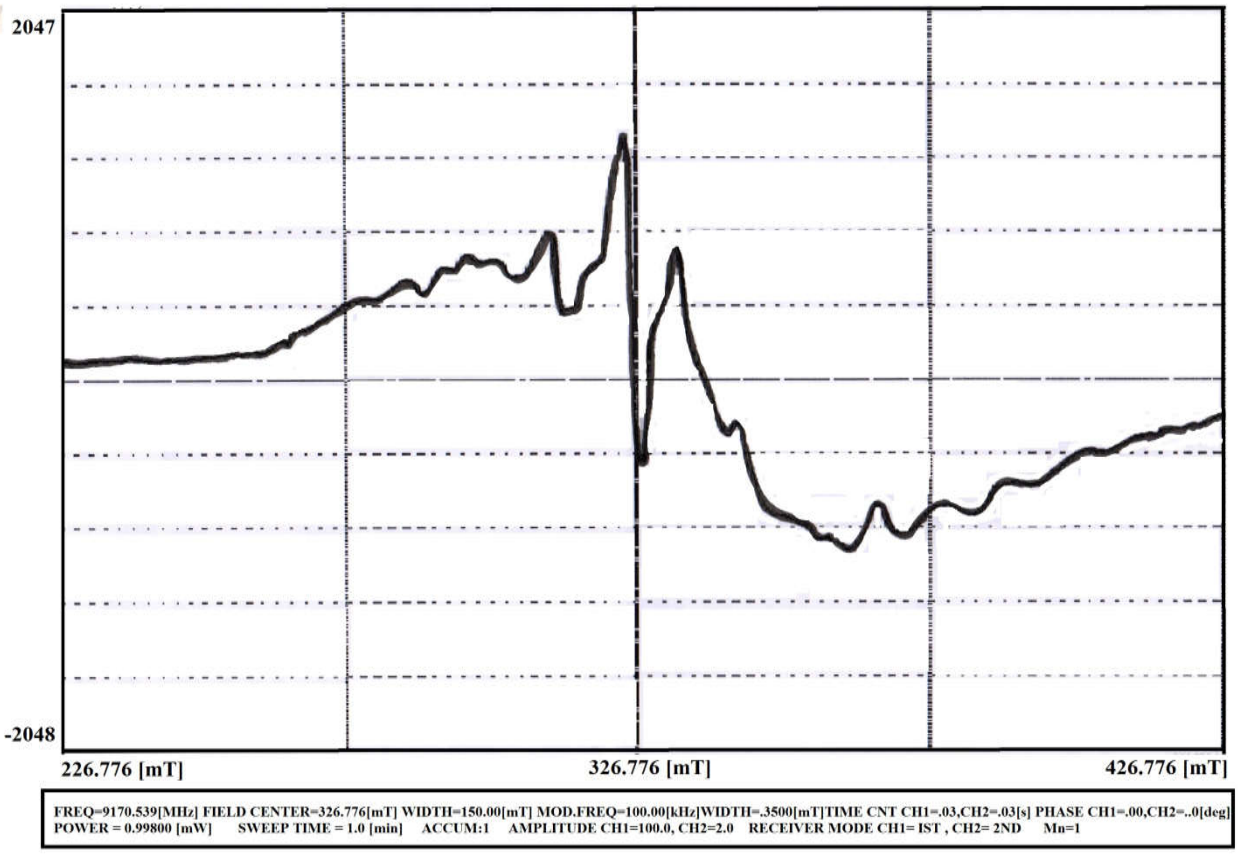

Figure 6. ESR spectrum of [VO(VA)$_2$.(H$_2$O)] at LNT

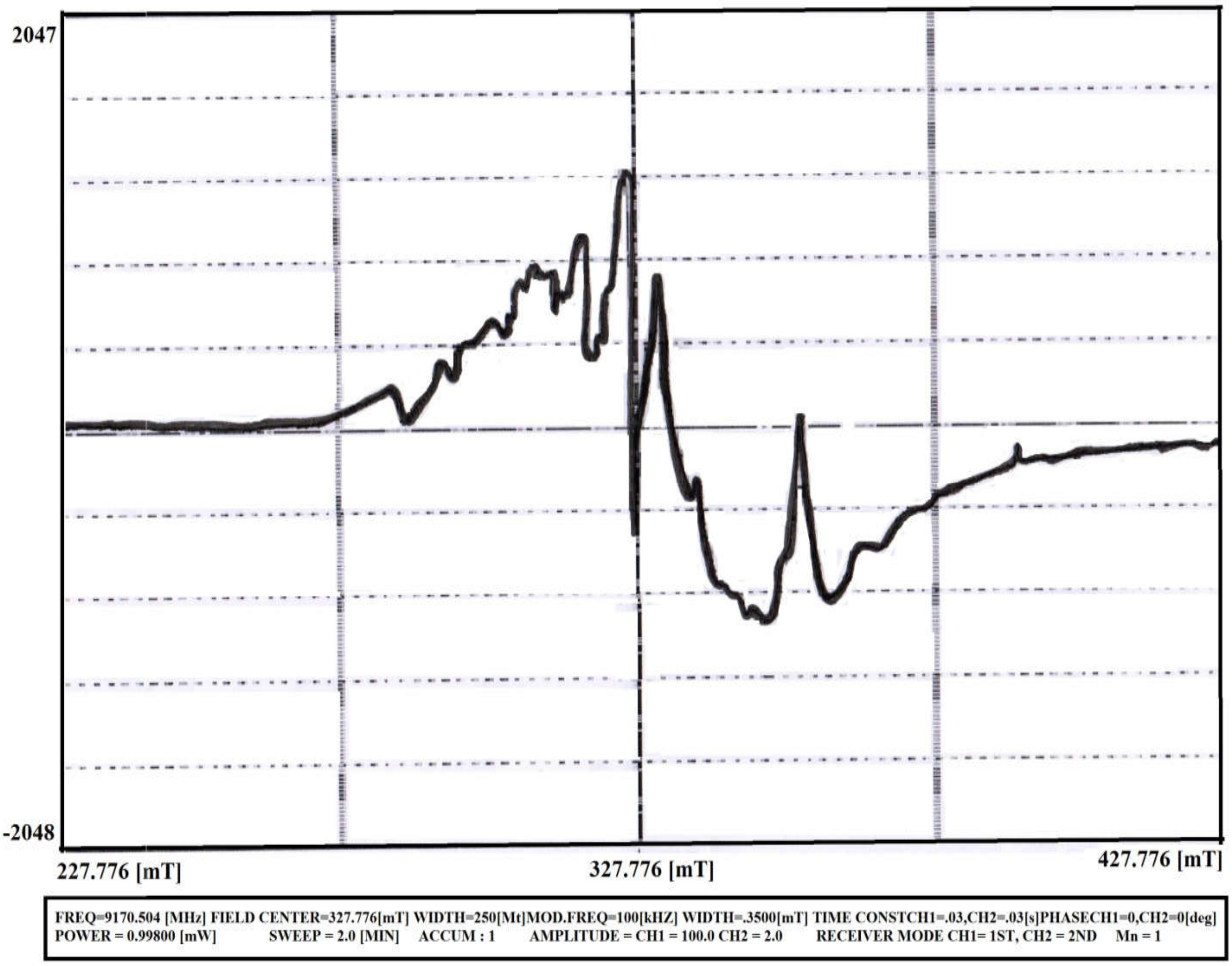

Figure 7. ESR spectrum of [VO(4AV)$_2$.(H$_2$O)] at LNT

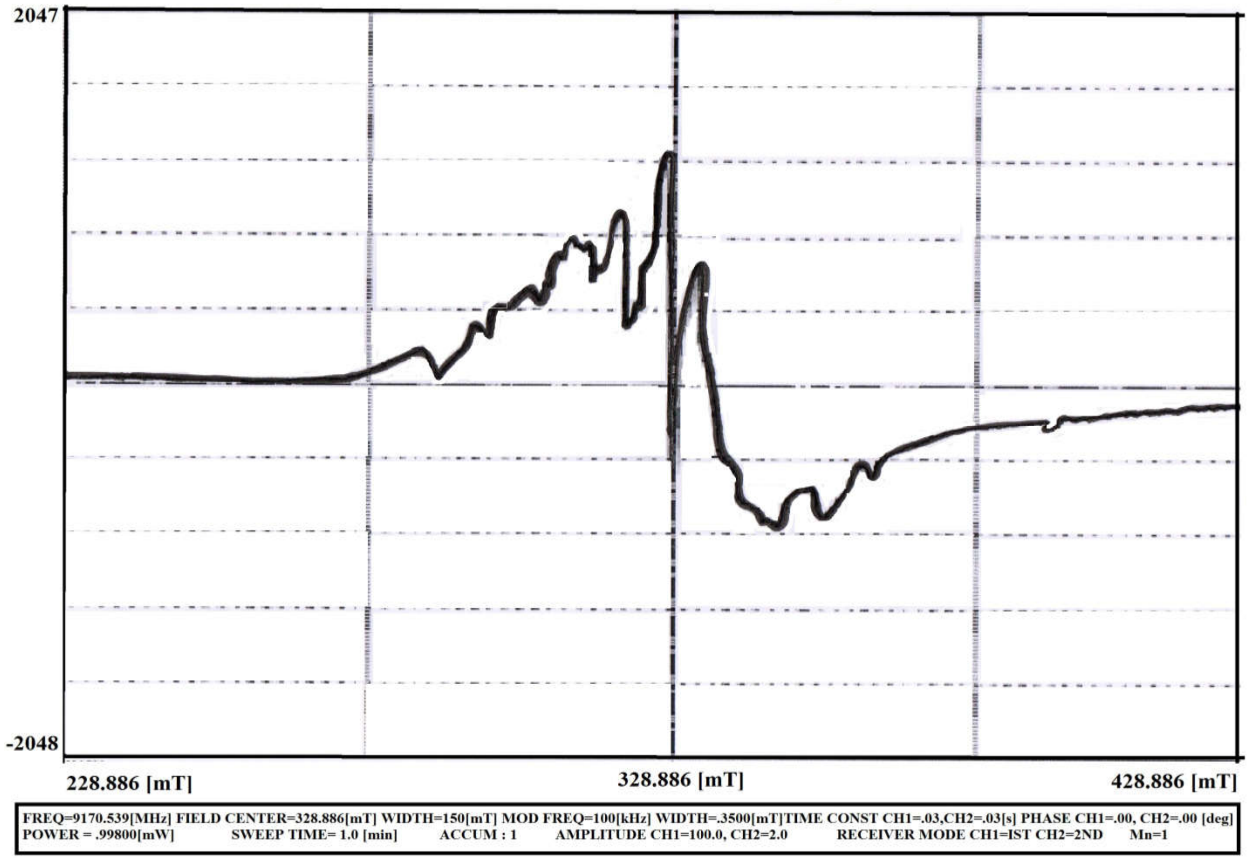

Figure 8. ESR spectrum of $[VO(4AF)_2.(H_2O)]$ at LNT

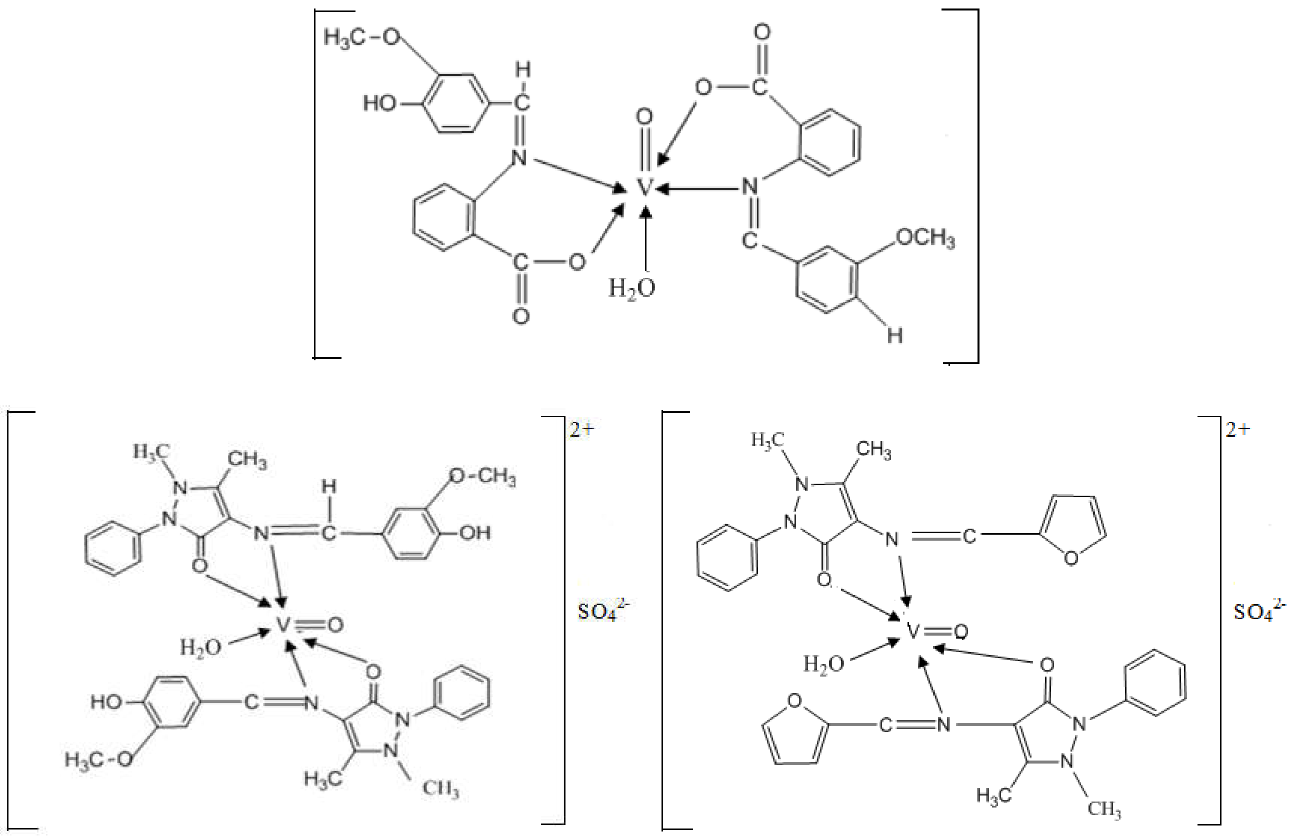

Figure 9. Structure of vanadium complexes

The synthesized systems were characterized by microanalysis, molar conductance values, FTIR, ESR, UV spectroscopy and TG analyses. Theses analytical and spectral data suggests that azomethine N atoms and carboxyl group coordinate around the metal ions in case of $[VO(4AV)_{2.}H_2O]SO_4$ and $[VO(4AF)_{2.}H_2O]SO_4$ where in case of $[VO(VA)_{2.}H_2O]$ coordination around the metal is through azomethine N atom and carboxylate anion generating a distorted octahedral structure. The antibacterial screening tests were also performed against bacteria which indicate that the complexes exhibit good antibacterial activity than the ligands. Based on the above results the structure of coordination compounds under investigation are formulated in Figure 9.

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