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MICROWAVE SYNTHESIS, REACTIVITY, ELECTRICAL AND SPECTRAL STUDIES OF OXOVANADIUM (IV) COMPLEXES SCHIFF BASE LIGANDS DERIVED FROM DIPHENYLGLYOXAL MOIETY

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ABSTRACT

Microwave-assisted synthesis has emerged as a powerful technique in modern chemistry, offering a sustainable and efficient alternative to conventional thermal methods. Microwave irradiated reactions under solvent free or less solvent conditions are attractive offering shorter reaction times, improved yields, and cleaner products with simplicity in processing and handling. Vanadium containing compounds have their utility in various physico-chemicals, biochemical physiological, enzymatic and catalytic processes. Novel solid complexes of oxovanadium(IV) with the Schiff bases *viz.* diphenylglyoxalidene - α, α′-bis (2-aminophenol) (DAPh), diphenylglyoxalidene-α,α′-bis (2-amino-4-chlorophenol) (DACp), diphenylglyoxalidene-α, α'-bis (2-aminopyridine) (DAPy) and diphenylglyoxalidene - α, α'-4-chloro-1,3-phenylenediamine (DCPa) have been synthesized by conventional as well as microwave methods. These compounds have been characterized by elemental analyses, thermal data, FAB mass, FT-IR, molar conductance, electronic spectra, ESR and magnetic measurements. FAB mass and thermal data give information about degradation pattern of complexes. The Schiff base ligands DAPh, DACp, DAPy and DCPa behave as tetradentate and bidentate coordinating through N_2O_2/ N_4 and N_2 donor, respectively. Molar conductance (10^{-3} M in MeOH) values show that VO-DAPh/DACp complexes are non-electrolytes and VO-DAPy/DCPa complexes have 2:2 electrolytic in nature. The proposed geometry of oxovanadium (IV)- Schiff base complexes are square pyramidal. The reactivity and substitution behaviour of the synthesized complexes have also been studied. Solid state AC-electrical conductivity studies reflect semiconducting nature of the complexes.

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INTRODUCTION

The coordination chemistry of oxovanadium (IV) with multidentate ligands received attention of researchers due to its growing applications in catalysis and therapeutics. Vanadium has multiple biological roles as essentiality in traces, therapeutic effect in small doses and toxicity in excess. Vanadium containing compounds have their utility as insulin mimetic and antiamoebic agent. Increasing biological and catalytic significance of vanadium, a lot of attention has been focussed on it, over the past decade. Vanadium constitutes 0.015% of earth's crust which is nearer to abundance of zinc. Biochemical role of vanadium has now become a widely chosen topic of bioinorganic chemistry. Though, the requirement of vanadium in mammals is at nano to picomolar level, however, its involvement in promotory and inhibitory biochemical processes *viz.* enzymes– nitrogenases and haloperoxidases, phosphatase and ATPase inhibitor, lowering of hyperglycemia and hyperlipidemia etc., have enough driving force for intensive research with an approach to establish its role as a micronutrient in human beings. The Schiff base transition metal complexes are a family of atractive oxidation catalysts for a variety of organic substrates because of their cheap & easy synthesis and their chemical & thermal stability (Mishra, *et al.*2005; Singh, *et al.* 2008; Mishra, *et al.* 2012; Mishra, *et al.*2008; Shukla,2008; Mishra, *et al.* 2009). Considering the relevance and significance of the work, we have synthesized and characterized some novel oxovanadium(IV) Schiff base complexes [VO(DAPh)].$3H_2O$ (I), [VO(DACp)].$3H_2O$ (II), [VO(DAPy)].$SO_4.4H_2O$ (III) and [VO(DCPa)].$SO_4.4H_2O$ (IV); where DAPh = diphenylglyoxalidene - α, α′-bis (2-aminophenol); DACp = diphenylglyoxalidene - α, α′-bis (2-amino-4-chlorophenol); DAPy = diphenylglyoxalidene- α, α′-bis (2-aminopyridine) and DCPa = diphenylglyoxalidene - α, α′-4-chloro-1,3-phenylenediamine. The reaction was carried out by both conventional and microwave methods. The application of microwave-assisted synthesis in organic, organometallic and coordination chemistry continues to develop at an astonishing pace. The salient features of microwave approach are shorter reaction times, simple reaction conditions and enhancements in yields(Mohanan,*et al.*2008;

Sharma, *et al.* 2010; Mahajan, *et al.* 2009; Polshettiwar, *et al.* 2009; Sun, *et al.* 2010).

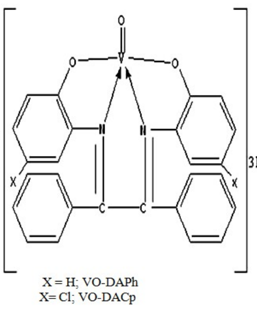
EXPERIMENTAL

Analyses and Physical Measurements: Elemental analysis were carried out using a Heraeus Elemental Analyzer and the FAB mass spectra were recorded at room temperature on a JEOL SX 102/DA-6000 mass spectrometer/data system using argon/xenon (6 kV, 10 mA) as the FAB gas (acclerating voltage 10 kV) at SAIF, CDRI Lucknow. Approximate molecular weight of compounds were determined by the Rast method.[17] The complexes were analysed for vanadium and sulphate content by the standard methods.[18] Magnetic measurement was made by Gouy's method. Electronic spectra (in MeOH) were recorded on Perkin Elmer Lambda-2B-spectrophotometer. Molar conductance ($10^{-3}M$ in Methanol) was measured on Elico-CM82 -Conductivity Bridge at room temperature. FT-IR spectra (in KBr disc) were recorded on Perkin Elmer RX-I spectrophotometer at SAIF, Panjab university Chandigarh. X-band EPR spectra were recorded at room temperature in a Varian E-112 spectrophotometer using TCNE (g = 2.0027) as the standard at SAIF, IIT Mumbai. The solid state AC-electrical conductivity have been measured by impedance spectroscopic method using HIOKI 3532-50 LCR Hitester at room temperature. Microwave assisted synthesis were carried out in open glass vessel on a modified microwave oven model 2001 ETB with rotating tray and a power source 230 V, microwave energy output 800 W and microwave frequency 2450 MHz. A thermocouple device was used to monitor the temperature inside the vessel of the microwave. The microwave reactions were performed using on/off cycling to control the temperature.

Conventional synthesis of Schiff bases (Ligands): The three Schiff bases (DAPh/DACp/DAPy) have been synthesized by adding the methanolic solution of diphenylglyoxal (0.01 mol) with the methanolic solution of 2-aminophenol/2-amino-4-chlorophenol/2-aminopyridine (0.02 mol) in 1:2 molar ratio. The reaction mixture was then refluxed on a water bath for about 4-6 hours. The condensation product was filtered, thoroughly washed with ethanol and ether, re-crystallized with ethanol and dried under reduced pressure over anhydrous $CaCl_2$. The purity of the synthesized compounds was monitored by TLC using silica gel (yield: DAPh - 74%; DACp - 71%; DAPy -70%).

Microwave method for the synthesis of Schiff bases: The 1:2 ratio of diphenylglyoxal with 2-aminophenol/2-amino-4-chlorophenol/2-aminopyridine were mixed thoroughly in a grinder. The reaction mixture was then irradiated by the microwave oven by taking 3-4 mL of dry ethanol as a solvent. The reaction was completed in a short time (3-5 min) with higher yields. The resulting product was then recrystallized with ethanol, finally dried under reduced pressure over anhydrous $CaCl_2$ in a desiccator. The progress of the reaction, purity of the product was monitored by TLC using silica gel G (yield: DAPh -90%; DACp - 87%; DAPy -88%).

I,II



III

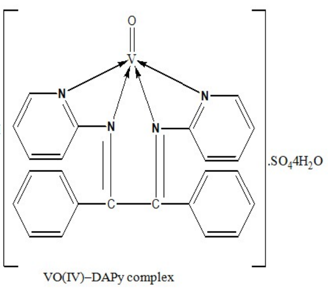


Figure 1. Proposed structure of VO(IV)–DAPh/DACp/DAPy complexes

Conventional preparation of VO (IV) Schiff base Complex: The VO(IV)–DAPh/DACp/DAPy complexes (fig. 1) have been prepared by mixing the methanolic solution of $VOSO_4.5H_2O$ (0.01 mol) to the methanolic solution of Schiff bases (DAPh/DACp/DAPy) (0.01 mol) in equimolar ratio. The resulting mixture was refluxed on water bath for 8-10 hours. A coloured product appeared on standing and cooling the refluxate. The precipitated complexes were filtered and washed repeatedly with ether. The complexes were re-crystallised twice with ethanol, finally washed with diethyl ether and dried under reduced pressure over anhydrous $CaCl_2$ in a dessicator. The complexes were further dried in an electric oven at 50-70°C (yield: 65-69%).

Microwave method for the Synthesis of metal complexes: The ligand and the metal salts were mixed in 1:1 VO(IV)–DAPh/DACp/DAPy and 1:2 VO(IV)–DCPa (metal:ligand) ratio in a grinder. The reaction mixture was then irradiated by the microwave oven by taking 3-4 mL of dry ethanol as a solvent. The reaction was completed in a short time (7-10 min) with higher yields. The resulting product was then recrystallized with ethanol and ether and finally dried under reduced pressure over anhydrous $CaCl_2$ in a desiccator. The progress of the reaction and purity of the product was monitored by TLC using silica gel G (yield: 80-84%).

Preparation of VO(IV)-DCPa Schiff base complex (by Template method): Because of poor yield of DCPa Schiff base by simple condensation, the template synthetic method has been used.VO(IV)–DCPa complex (fig. 2) has been prepared by mixing the methanolic solution of $VOSO_4$-$5H_2O$ (0.01 mol) to the methanolic solution of diphenylglyoxal (0.02 mol) in 1:2 molar ratio. The reaction mixture was then refluxed on a water bath for about 4-5 hours. A change in colour of reaction mixture occurs then a methanolic solution of 4-chloro-1,3-phenylenediamine (0.02 mol) was added. The resulting mixture was again refluxed on a water bath for about 5-6 hours. A coloured product appeared on standing and cooling the refluxate. The precipitated complex was filtered and washed repeatedly with ether. The complex was re-crystallized twice with ethanol, finally washed with diethyl ether and dried under reduced pressure over anhydrous $CaCl_2$ in a dessicator. The complex was further dried in an electric oven at 50-70°C (yield: 68%).

IV

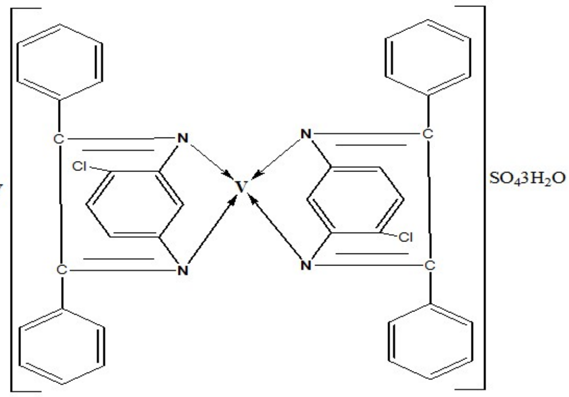


Figure 2. Proposed structure of VO(IV)–DCPa complex

RESULTS AND DISCUSSION

As a result of microwave-assisted synthesis, it was observed that the reaction was completed in a short time with higher yields compared to the conventional method. In the microwave method homogeneity of reaction mixture was increased by the rotating of reaction platform tray. The confirming of the results was also checked by the repeating of the synthesis process. All the metal complexes are coloured, solid and stable towards air and moisture at room temperature. They decompose on heating at high temperature, more or less soluble in common organic solvents. Analytical data of the compounds and together with their proposed molecular formula are given in Table 1. The metal complexes exhibit 1:1 (VO-DAPh/DACp/DAPy) and 1:2 (VO-DCPa) metal to Schiff base ligand stoichiometry. Molar conductance values show that VO-DAPh/DACp complexes are non-electrolytes and the VO-DAPy/DCPa complexes have 2:2 electrolytic in nature (Chandra, *et al.* 2009).

Reactivity: The solutions of complexes were made in methanol (99%) to check the reactivity and substitution behaviour against aquo, ammine, chloro, hydroxo and thiocynato ligands. The reaction was monitored by observing change in colour or precipitation (Mishra and Pandey, 2005).

Reaction with H_2O: VO-DAPh/DACp/DCPa complexes react slowly while VO-DAPy complex reacts rapidly on addition of slight amount of water at room temperature (1 hour). All the complexes decompose on heating with water.

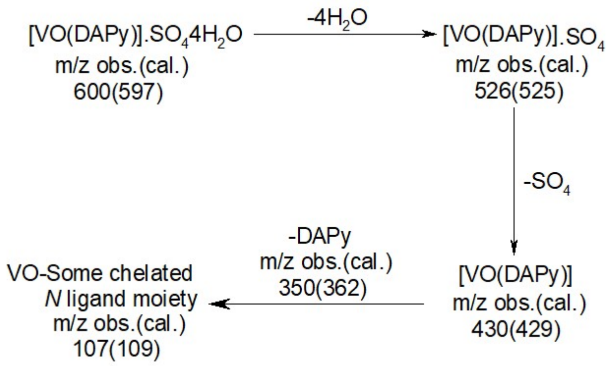
Reaction with dil. NH_3 (aq.):VO-DAPh/DACp/DCPa complexes react slowly with dil. aq. ammonia on keeping for 1 hour at room temperature, but VO-DCPy complex reacts fast. All the complexes react appreciably on heating.

Reaction with dil. HCl (aq.): VO-DAPh/DACp/DCPa complexes react slowly but VO-DAPy reacts rapidly with dil. hydrochloric acid, on keeping for 1 hour at room temperature. On heating the complexes decompose quickly.

Reaction with dil. NaOH (aq.): VO-DAPh/DACp complexes react slowly while VO-(DAPy/DCPa) complexes react rapidly with dil. alkali at room temperature (1 hour). All the complexes react to sufficient extent on heating.

Reaction with dil. KSCN (aq.): VO-DAPh/DACp/DCPa complexes react slowly with dil. potassium thiocynate, even on retaining for 1 hour at room temperature but VO-DAPy complex react fast under similar conditions. On heating the complexes react appreciably.

FAB mass spectra: The FAB-mass spectra suggested that all the complexes have a monomeric nature. These complexes show molecular ion peaks in good agreement with the empirical formula suggested by elemental analyses. The FAB mass spectra of [VO(DAPy)]SO_4.4H_2O complex has been studied as one of the representative case. The peaks of appreciable intensity have been observed at *m/z* values 600, 526, 430, 350 and 107 a.m.u., which suggest the proposed formula and the fragmentation pattern (Scheme 1). The *m/z* value 600 corresponds to nearest composition [VO(DAPy)]SO_4.4H_2O, 526 to [VO(DAPy)]SO_4, 430 to [VO(DAPy)], 350 to ligand alone and 107 to VO with some chelated-N-ligand moiety. This further indicate that oxovanadium (IV) at the centre of complex is coordinated with N_4 donor neutral Schiff base ligand and the resulting complex is monomeric (Mishra, *et al.*2012).



Scheme 1. FAB mass fragmentation pattern of VO-DAPy complex

Infrared spectral studies: The data of the IR spectra of Schiff base ligands and their complexes are listed in Table 2. The IR spectra of the complexes were compared with those of the free ligand in order to determine the involvement of coordination sites in chelation. Characteristic peaks in the spectra of the ligand and complexes were considered and compared. The IR spectra of Schiff base ligands (DAPh, DACp, DAPy and DCPa) give strong intensity band at about 1590$\pm$5 due to υ(C=N) (azomethine) group. This band shifts lower side by 20-30 cm^{-1} in complexes, suggesting coordination through azomethine nitrogen.

Table 2. Selected IR frequencies (cm^{-1}) of ligands and complexes

Compound	υ(C=N) (azomethine)	υ(C…C), (C…N) (pyridine ring)	υ(C-O)	υ(O-H)Phenolic	υ(V=O)	υ(V-O)	υ(V-N)
DAPh	1587	-	1263	1343	-	-	-
[VO(DAPh)].3H_2O	1565	-	1312	-	984	540	488
DACp	1592	-	1270	1354	-	-	-
[VO(DACp)].3H_2O	1560	-	1323	-	980	535	485
DAPy	1594	1578,1490, 1449	-	-	-	-	-
[VO(DAPy)].SO_4.4H_2O	1573	1544,1476, 1421	-	-	975	-	509
DCPa	1590	-	-	-	-	-	-
[VO(DCPa)$_2$].SO_4.3H_2O	1570	-	-	-	999	-	502

Table 3. Electronic spectral, magnetic moment and ESR data of complexes

Complexes	Transitions Band (cm^{-1})			μ_{eff} (B.M.)	*ESR Parameters*			
	$^2B_2 - {}^2E$	$^2B_2 - {}^2B_1$	$^2B_2$-$^2A_1$		$g_{\|\|}$	$g_\perp$	g_{av}	Δg
[VO(DAPh)].3H_2O	13700	19980	24150	1.76	1.8918	1.9843	1.9534	0.0925
[VO(DACp)].3H_2O	13695	19960	24160	1.77	1.8863	1.9874	1.9537	0.1011
[VO(DAPy)].SO_4.4H_2O	13706	20100	23130	1.74	1.8890	1.9783	1.9485	0.0893
[VO(DCPa)].SO_4.3H_2O	14203	19000	23250	1.78	1.9029	1.9753	1.9511	0.0724

Table 4. Composition and %weight obs. (calcd.)

Complexes (Room temperature)	100°C– 275°C	375°C	600°C
[VO ($C_{26}H_{18}O_2N_2$)]3H_2O	[VO ($C_{26}H_{18}O_2N_2$)] 90.12 (89.43)	VO- some chelated ligand moiety 40.12	V_2O_5 17.15 (17.80)
[VO($C_{26}H_{16}O_2N_2Cl_2$)]3H_2O	[VO($C_{26}H_{16}O_2N_2Cl_2$)] 91.26 (90.68)	VO- some chelated ligand moiety 42.78	V_2O_5 15.20 (15.68)
[VO($C_{24}H_{18}N_4$)]SO_4.4H_2O	[VO($C_{24}H_{18}N_4$)].SO_4 86.76 (87.93)	VO- some chelated ligand moiety 44.12	V_2O_5 15.02(15.24)
[VO($C_{20}H_{10}N_2Cl)_2$]SO_4.3H_2O	[VO($C_{20}H_{10}N_2Cl)_2$].SO_4 94.56 (93.60)	VO- some chelated ligand moiety 45.79	V_2O_5 10.10(10.78)

A strong band in DAPh and DACp ligand spectrum at 1343 and 1354 cm^{-1} due to phenolic-OH (deformation) has been found absent in complexes. This suggests deprotonation of phenolic-OH in VO-DAPh/DACp complexes. The chelation of phenolic oxygen in VO-DAPh/DACp complexes is supported by upward shift of υ(C-O) (phenolic 1265 ± 5 cm^{-1}). The ligand DAPy gives bands at 1578, 1490 and 1449 cm^{-1} due to υ(C…C), (C…N) (Aromatic ring). These bands shift lower side by 15-25 cm^{-1} in VO-DAPy complex indicating chelation with nitrogen of pyridine ring. The appearance of broad band around 3400-3200 cm^{-1} in the spectra of complexes is due to associated water molecules. A characteristic non-ligand sharp band in the spectra of complexes at 999-975 cm^{-1}, has been assigned to υ(V=O). Some new bands of low intensity in the region 540-535 cm^{-1} and 485-509 cm^{-1} in the spectra of complexes have been attributed to υ(V-O) and υ(V-N) modes, respectively. This indicates that ligands DAPh/DACp/DAPy and DCPa behave as tetradentate and bidentate, respectively (Abdallah, *et al.*2010; Nakamoto, 1998; Garg, *et al.* 2006; Raman, *et al.* 2007).

Magnetic moment and electronic spectra: Room temperature magnetic moment values of oxovanadium(IV) complexes lie in the range 1.74-1.78 B.M. The values are well suited for oxovanadium (IV) monomeric complexes with one unpaired electron. The electronic spectra (in MeOH) of $[VO(DAPh)].3H_2O$, $[VO(DACp)].3H_2O$, $[VO(DAPy)]SO_4.4H_2O$ and $[VO(DCPa)_2]SO_4.3H_2O$ complexes exhibit bands in the region 13695-14203, 19000-20100 and 23130-24150 cm^{-1}; which are assignable to $^2B_2 - {}^2E$, $^2B_2 - {}^2B_1$ and $^2B_2 - {}^2A_1$, transitions respectively Table 3. The geometry of these five coordinated mononuclear complexes (fig.1) has been ascribed to be trigonal bipyramidal (tbp) or square pyramidal (sp) (Lever, 1984; Soliman, *et al.*2004; Dubey, *et al.* 2008; Dutta and Syamal, 1993).

ESR spectral studies: The X-band EPR spectra of oxovanadium (IV) (d^1, ^{51}V, I = 7/2) complexes are not so resolved at room temperature to exhibit all eight hyperfine lines. The calculated values of $g_{||}$, $g_\perp$, g_{av} and **Δ**g for all the four complexes are given in Table 3. Here $g_{av}={}^1/_3\,[2g_\perp+g_{||}]$. The values are typical of the spectra displayed by tbp or spoxovanadium (IV) complexes with one unpaired electron in an orbital of mostly dxy character. The g_{av} values determined from the spectra are nearer to spin only; a slight variation may be accounted to spin-orbital coupling. In square pyramidal complexes with C_{4V} symmetry, the V=O bond is along z and the other four donor atoms (N_4) are along the x, y axes. An anisotropic EPR spectrum is expected exhibiting two g values ($g_z=g_{11}<g_\perp=g_x=g_y$) (Hathaway, 1987; Jain, *et al.*2012).

Thermal properties: The thermal degradation behaviour of the complexes has been noted on heating for 30 minutes in a muffle furnace at four temperature range (100° C, 275° C, 375° C and 600°C) in the atmosphere of air. The weight of the complex sample after each heating was recorded. The percentage remaining weight and the approximate nearby composition of the pyrolytic product corresponding to each temperature have tentatively been given in Table 4. The complexes are stable (w.r.t. Schiff base) in general upto 275°C; above this temperature some part of the chelated Schiff base start decomposing. The ultimate pyrolytic product (600°C) in oxygenated atmosphere for all the cases is supposed to be vanadium pentaoxide (Mohamed, *et al.* 2006; Ourari, *et al.* 2006; Makode, *et al.*2009; Jain, *et al.*2012).

Solid State electrical conductivity: The solid state electrical conductivity of the synthesized complexes in compressed pellet form (3 ton cm^{-2}) at room temperature (35°C) has been measured. The electrical conductivity value of the complexes was found to lie in the range 10^{-5} S cm^{-1}-10^{-4} S cm^{-1} Table 1. Thus the values correspond to semiconducting nature (Sujamol, *et al.* 2010; Mishra, *et al.* 2012). The electrical conductivity of chelates follows the order:

$[VO(DAPy)]SO_4.4H_2O$> [VO $(DCPa)_2]SO_4.3H_2O$>$[VO(DAPh)].3H_2O$>[VO $(DACp)].3H_2O$

CONCLUSION

In the present research studies, our efforts are synthesized some Oxovanadium (IV)-N_4, N_2O_2 and N_2 donor Schiff base complexes from conventional as well as microwave methods. These synthesized compounds characterized by various physicochemical and spectral analyses. In the result of microwave assisted synthesis, it has been observed that the reaction time decreased from hours to minutes and availability of the product within better yields compared to the classical method. The reactivity and substitution behaviour of synthesized complexes against aquo, ammine, chloro, hydroxo and thiocyanato ligands, have also been studied. These complexes show semiconducting nature. The synthesized ligands and complexes may prove to be potential chemical entities for various physico-chemical, physiological and biochemical reactions. The Schiff base transition metal complexes involving multidentate ligands are being considered now a days as attractive oxidation catalysts for a variety of organic substrates because of their cheap & easy synthesises and their chemical thermal & stability; thus they are of great commercial interest. Thermal data show degradation pattern of the complexes. Thermogravimetric studied of the complexes also helped to characterize of the complexes. Electrical conductivity data suggest that all the complexes fall in the semiconducting range.

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