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# OXOVANADIUM(IV) COMPLEXES WITH LIGANDS DERIVED BY CONDENSATION OF 1,2-DIACETYLBENZENE WITH 2-AMINOBENZAMIDE AND β-DIKETONES

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*The synthesis of a new parent oxovanadium(IV) complex, $[VO(L)]SO_4$, is achieved under in-situ experimental conditions where vanadyl ion acts as a kinetic template for the ligand derived by condensation of 1,2-diacetylbenzene with 2-aminobenzamide in 1:2 molar ratio in aqueous ethanol medium. The parent complex, $[VO(L)]SO_4$, has reacted with β-diketones to get macrocyclic complexes of the general formula, $[VO(mac)]SO_4$, where mac = macrocyclic ligands derived by reactions of $[VO(L)]SO_4$ with acetylacetone, benzoylacetone, thenoyltrifluoroacetone and dibenzoylmethane. These vanadyl complexes are characterized and their tentative structures are ascertained on the basis of elemental analyses, molar conductance, magnetic moments and spectral data of electronic, infra-red and X-band electron spin resonance spectroscopy. All the vanadyl complexes are five-coordinate with tetradentate chelating ligands. These vanadyl complexes were screened for their antifungal activities against Aspergillus niger and Aspergillus flavus, which showed promising antifungal activities.*

**Key words:** vanadyl ion, macrocyclic complexes, 1,2-diacetylbenzene, β-diketones.

## INTRODUCTION

The coordination chemistry of vanadium (Yuan et al., 2009; Rehder et al., 2003) and particularly its biological (Baran et al., 2008; Guiotoku et al., 2007) and catalytic activities (Selbin et al., 1966) have attracted huge interest in the synthesis of oxovanadium(IV) complexes with a variety of ligands with nitrogen and oxygen donor atoms. Recognition of vanadium in enzymatic systems such as in the haloperoxidases (Brink et al., 2001) found in marine fungi and algae (Meisch et. al., 1979) and in mushrooms (Adetutu et al., 2010); have induced enhanced curiosity to study vanadium role in biological systems and catalytic areas. Reports are available for vanadyl complexes in its +4 and +5 oxidation states showing insulin like activities (Shukla et. al., 2008), anticancer, antitumor, antibacterial and antifungal activities (Pasayat et al., 2012; Dash et al., 2012). The vanadyl complexes are reported to influence many enzymatic systems such as phosphatases, ATPases, peroxidases, ribonucleases, protein kinases and oxidoreductases (Wilkinson et al.,

1987). There are reports that vanadium enters into the organism by inhalation, the gastrointestinal tract and the skin, which is specifically stored in certain organs mainly in the liver, Kidney and bones (Cusi et al., 2001). In order to explore the pharmaceutical applications of vanadyl ion in complexed form, a series of oxovanadium(IV) complexes with ligands derived by condensation of 1,2-diacetylbenzene with 2-aminobenzamide in 1:2 molar ratio in aqueous ethanol medium and their cyclisation with β-diketones is reported, where vanadyl ion play a key role as kinetic template. The antifungal studies of these vanadyl complexes are carried out against Aspergillus niger and Aspergillus flavus.

## MATERIALS AND METHODS

### Materials

Reagent grade chemicals and solvents were used in the synthesis. Vanadyl sulphate, 1,2-Diacetylbenzene and 2-Aminobenzamide used were Aldrich products. The β-diketones such as acetylacetone, benzoylacetone, thenoyltrifluoroacetone and dibenzoylmethane were Sisco Research Laboratory products.

### Analytical and physical measurements

Standard gravimetric method was used for quantitative estimation of Vanadium as its sodium vanadate (Vogel et al., 1978). Estimation of Sulphur was made as barium sulphate (Vogel et al., 1978). The standard method for determination of melting point (uncorrected) was employed using sulphuric acid bath. Toshniwal conductivity bridge, model no. CLO102A was used for conductance measurements at room temperature. The magnetic susceptibility of the complexes in powder form was carried out at room temperature using Guoy's balance. Mercury tetrathiocyanatocobaltate(II), $Hg[Co(CNS)_4]$, ($X_g$ = 1644 x $10^{-6}$ c.g.s. unit at $20^0$ C) was used as calibrant. The electronic spectra of the complexes were recorded on Beckmann DU-2 spectrophotometer in the ranges 2000 – 185 nm using dimethylformamide as solvent. The room temperature and liquid nitrogen temperature E.S.R. spectra were recorded at SAIF, IIT Mumbai, India. The infrared spectra of the complexes were measured on IRAffinity-1, FTIR spectrophotometer, SHIMADZU using KBr pellets in the range 4000 $cm^{-1}$ – 200 $cm^{-1}$.

#### In-situ preparation of oxovanadium(IV) complex with ligand derived by condensation of 1,2-diacetylbenzene with 2-aminobenzamide ( 1:2 )

Vanadyl sulphate (2 mmol) was dissolved in methanol (25 mL), which was added into a refluxing solution of 1, 2-diacetylbenzene (2 mmol) and 2-aminobenzamide (4 mmol) in ethanol (25 mL). The reflux of the reaction mixture

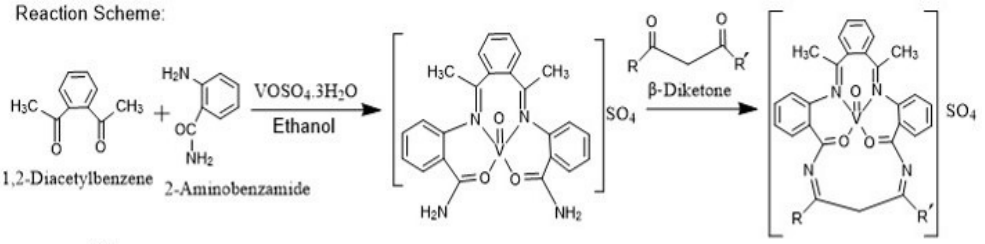
was continued for 5 hours, when the color of the solution turned green with little precipitation. A dark green color product was isolated after evaporating solvent under vacuum. The isolated complex was thoroughly washed with methanol / ethanol (1:1) mixture (10 mL). The yield was found to be 70%.

**In-situ preparation of macrocyclic complexes of oxovanadium(IV) using β-diketones as ring closing reagents**

Vanadyl sulphate (2 mmol) was dissolved in methanol (25 mL), which was added into a refluxing solution of 1, 2-diacetylbenzene (2 mmol) and 2-aminobenzamide (4 mmol) in ethanol (25 mL). The reflux of reaction mixture was continued for 5 hours, when the color of the solution turned green with little precipitation. To this reaction mixture, an ethanolic solution (10 mL) of acetylacetone (2 mmol) and glacial acetic acid (1 mL) were added. This reaction mixture was refluxed further for about 3 hours and a green precipitate was isolated after cooling the solution. The vanadyl complex was purified by washing with a methanol / ethanol (1:1) mixture (10 mL). The yield was found to be 60%. A similar procedure was followed for the synthesis of other oxovanadium(IV) macrocyclic complexes using benzoylacetone, thenoyltrifluoroacetone and dibenzoylmethane.

## RESULTS AND DISCUSSION

The physical and analytical data of the complexes are presented in Table 1 and the reaction scheme is presented in Scheme1.




Where,

| R | R′ | β-Diketone |
|---|---|---|
| $-CH_3$ | $-CH_3$ | Acetylcetone |
| $-C_6H_5$ | $-CH_3$ | Benzoylacetone |
| $-C_4H_3S$ | $-CF_3$ | Thenoyltrifluroacetone |
| $-C_6H_5$ | $-C_6H_5$ | Dibenzoylmethane |

**Scheme 1**

As shown in the reaction scheme 1, the parent oxovanadium(IV) complex was synthesized by in-situ method by refluxing the reaction mixture containing vanadyl sulphate, 1,2-diacetylbenzene and 2-aminobenzamide in 1:1:2 molar ratios in aqueous ethanol medium. The reaction appears to proceed as follows:

$(VOSO_4).3H_2O$ + 1, 2-Diacetylbenzene + 2-Aminobenzamide → $[VO(L)]SO_4 + 5\ H_2O$

**Scheme 2**

The oxovanadium(IV) complex was reacted with β-diketones in 1:1 molar ratio, which resulted formation of macrocyclic complexes, $[VO(mac)]SO_4$, by condensation of the terminal amino group with ketonic group of β-diketones. The elemental analyses of the oxovanadium(IV) complexes show a 1:1 metal to ligand stoichiometry.

**Table 1**

Physical and analytical data of the oxovanadium(IV) complexes

| Complex | | Decomposition temperature ($^0$C) | C (%) **Found** *Calcd.* | H (%) **Found** *Calcd.* | N (%) **Found** *Calcd.* | V (%) **Found** *Calcd.* | S (%) **Found** *Calcd.* | $\mu_{eff.\ BM}$ 300K |
|---|---|---|---|---|---|---|---|---|
| Formula | number | | | | | | | |
| $C_{22}H_{22}N_4O_7VS$ | $[VO(L)]SO_4$ | 260 | **51.28** *51.34* | **3. 90** *3.92* | **9.96** *9.98* | **9.00** *9.08* | **5.68** *5.70* | 1.71 |
| $C_{29}H_{26}N_4O_7VS$ | $[VO(mac^1)]SO_4$ | 264 | **55.65** *55.68* | **4.13** *4.16* | **8.93** *8.96* | **8.14** *8.15* | **5.10** *5.12* | 1.73 |
| $C_{34}H_{28}N_4O_7VS$ | $[VO(mac^2)]SO_4$ | 265 | **59.34** *59.39* | **4.00** *4.07* | **8.12** *8.15* | **7.39** *7.41* | **4.61** *4.65* | 1.72 |
| $C_{32}H_{23}N_4O_7VSF$ | $VO(mac^3)]SO_4$ | 264 | **51.35** *51.40* | **3.02** *3.07* | **7.46** *7.49* | **6.78** *6.81* | **8.52** *8.56* | 1.73 |
| $C_{39}H_{30}N_4O_7VS$ | $VO(mac^1)]SO_4$ | 265 | **62.46** *62.48* | **3.98** *4.00* | **7.45** *7.47* | **6.78** *6.80* | **4.24** *4.27* | 1.72 |

Where L = Ligand derived by condensation of 1, 2-Diacetylbenzene with 2-Aminobenzamide (1:2)

$mac^1$ = Macrocyclic ligands obtained by reaction of L with Acetylacetone.
$Mac^2$ = Macrocyclic ligands obtained by reaction of L with Benzoylacetone.
$Mac^3$ = Macrocyclic ligands obtained by reaction of L with Thenoyltrifluoroacetone.
$Mac^4$ = Macrocyclic ligands obtained by reaction of L with Dibenzoylmethane.

## INFRARED SPECTRA

The most relevant characteristic infrared bands of the oxovanadium(IV) complexes are listed below:

**Table 2**

Characteristic infrared spectral bands ($cm^{-1}$) of the oxovanadium(IV) complexes

| **Complex** | ***v*(>C=N)** | ***v* (V-N)** | ***v*(V=O)** | ***v*(C=O)** | ***v*(CN)+ *∂*(NH)** | **$SO_4^{2-}$** | **$SO_4^{2}$** | **$SO_4^{2}$** | **(N-H)** | **(N-H)** |
|---|---|---|---|---|---|---|---|---|---|---|
| | | | | **(Amide-)** | **(Amide-II)** | ***v*$_3$** | ***v*$_1$** | ***v*$_4$** | **$v_{asym}$** | **$v_{sym}$** |
| $[VO(L)]SO_4$ | 1610 | 304 | 980 | 1665 | 1650 | 1135 | 956 | 600 | 3352 | 3170 |
| $[VO(mac^1)]SO_4$ | 1605 | 302 | 981 | 1662 | 1648 | 1134 | 954 | 602 | | |
| $[VO(mac^2)]SO_4$ | 1605 | 304 | 982 | 1664 | 1650 | 1132 | 956 | 604 | | |
| $[VO(mac^3)]SO_4$ | 1610 | 302 | 981 | 1664 | 1648 | 1135 | 954 | 604 | | |
| $[VO(mac^4)]SO_4$ | 1605 | 301 | 980 | 1662 | 1648 | 1134 | 956 | 602 | | |

The IR spectrum of the $[VO(L)]SO_4$ complex shows bands at 1610 $cm^{-1}$, which may be assigned to the coordinated azomethine group (Sreeja et. al., 2005; Yadava et. al., 1987). The infrared bands appearing at 1665 $cm^{-1}$ and 1650 $cm^{-1}$ are assigned to the coordinated amide-I, v (C=O) and amide-II, v(CN) + *∂* NH (Maurya et al., 2006). The bands appearing at 3352 $cm^{-1}$ and 3170 $cm^{-1}$ may be assigned to asymmetrical and symmetrical N-H stretching modes of the non-coordinated terminal amino groups of the ligand, L (Nonoyama et al., 1975). A band at 304 $cm^{-1}$ further supports the coordination of nitrogen atom to vanadium, as it may be assigned to v (V-N) vibrations (Sakata et al., 1989). The presence of an intense band at 980 $cm^{-1}$ may be assigned to v (V=O) vibration (Samanta et al., 2003). The ionic sulfate group in the complex is indicated (Tsuchimoto et al., 2000) by three bands at 1135 $cm^{-1}$ ($v_3$), 956 $cm^{-1}$ ($v_1$) and 600 $cm^{-1}$ ($v_4$). The absence of $v_2$ band and non-splitting of $v_3$ band indicate that Td symmetry is retained. The infrared spectra of the other macrocyclic oxovanadium(IV) complexes as detailed in Table 2 have similar patterns except that $v_{asym.}$ and $v_{sym.}$ vibrations of the terminal $-NH_2$ group disappear after the ring closing reaction with β-diketones and infrared bands of uncoordinated *v*(>C=N) appear.

## MAGNETIC MOMENT AND ELECTRONIC SPECTRA

The magnetic moment values of all the oxovanadium(IV) complexes were found in the range 1.71 – 1.73 B.M. at room temperature, which are well within the range reported for vanadyl complexes with paramagnetic center (Sarkar et al., 2008). This data confirm the mononuclear nature of oxovanadium(IV) complexes. The electronic spectra show bands in the regions 11,130 – 11,900 $cm^{-1}$, 15,200 –

15,900 $cm^{-1}$, which are in accordance other reports for oxovanadium(IV) complexes involving nitrogen/oxygen donor atoms. According to Ballhausen and Gray scheme, these electronic bands have been assigned to $^2B_2 \rightarrow {}^2E$, $^2B_2 \rightarrow {}^2B_1$ and $^2B_2 \rightarrow {}^2A_1$ transitions respectively. One more electronic band observed in the region 35,200 – 35,700 $cm^{-1}$ may be assigned to the transition arising out of azomethine linkages.

## ESR SPECTRA

The X-band ESR spectra of the oxovanadium(IV) complexes were recorded at room temperature and liquid nitrogen temperature, which show eight lines due to hyperfine splitting originating basically from the interaction of unpaired electron with a $^{51}V$ nucleus having the nuclear spin no. I = 7/2 (Mishra et. al., 2005; Ando et. al., 2003). Anisotropy is not evidenced at room temperature because of rapid tumbling of molecules in solution and only g-average values are obtained.

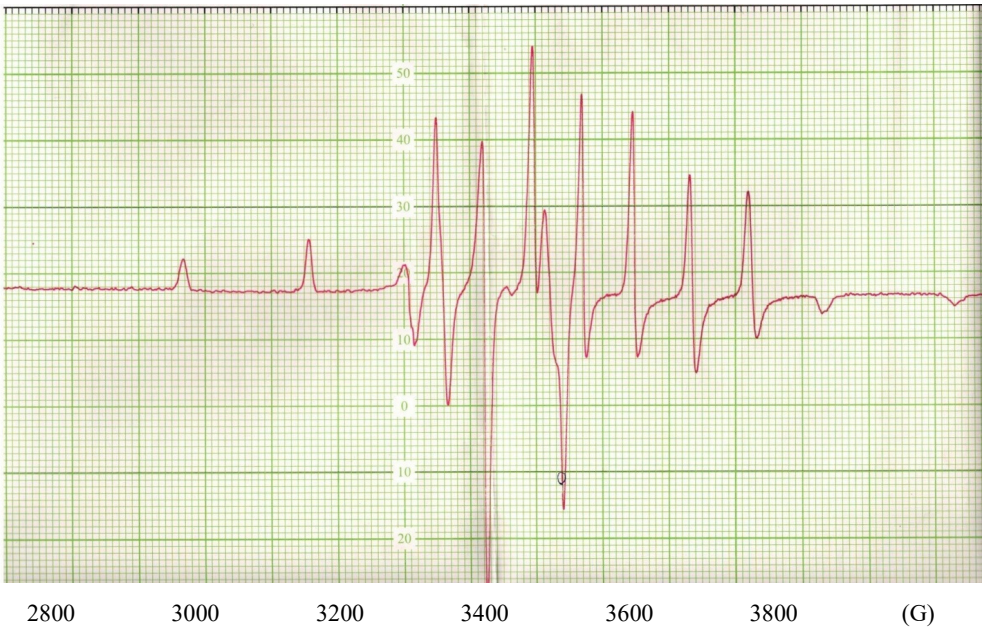



**Fig. 1.** X-Band ESR spectrum of $[VO(L)]SO_4$ at liquid nitrogen temperature (77 K).

However, as observed in Fig. 1, an isotropy is clearly visible in the spectra at liquid nitrogen temperature and eight lines each due to $g_{\parallel}$ and $g_{\perp}$ are observed separately showing $g_{\parallel} < g_{\perp}$ and $A_{\parallel} > A_{\perp}$. The $g_{\parallel}$, $g_{\perp}$, $A_{\parallel}$ and $A_{\perp}$ values, which are measured from the ESR spectra, are in good agreement for square pyramidal structure.

**Table 3**

X Band ESR spectral data of oxovanadium(IV) complexes

| Complex | Room Temp. | Liquid nitrogen temperature | | | | | |
|---|---|---|---|---|---|---|---|
| | ıg ı | $g_{ıı}$ | g⊥ | ıgı | $A_{ıı}$ | A⊥. | ıAı |
| $[VO(L)]SO_4$ | 1.980 | 1.930 | 1.970 | 1.956 | 190.70 | 66.48 | 107.88 |
| $[VO(mac^1)]SO_4$ | 1.98 | 1.932 | 1.973 | 1.959 | 190.80 | 65.90 | 107.53 |
| $[VO(mac^2)]SO_4$ | 1.981 | 1.931 | 1.972 | 1.958 | 190.60 | 65.88 | 107.45 |
| $[VO(mac^3)]SO_4$ | 1.982 | 1.932 | 1.973 | 1.959 | 190.72 | 65.90 | 107.50 |
| $[VO(mac^4)]SO_4$ | 1.982 | 1.931 | 1.972 | 1.958 | 190.71 | 65.92 | 107.51 |

## ANTIFUNGAL ACTIVITY

The oxovanadium(IV) complexes were tested for their antifungal activity against the fungi Aspergillus flavus and Aspergillus niger using standard methods (Rehman et al., 2013). All tested complexes showed significant antifungal activity but $[VO(mac^3)]SO_4$ was found to be the most active, which may be due to sulphur and fluorine atoms present in the thenoyltrifluoroacetone, a cyclising agent as β-diketone. The Fluconazole (75 µg/mL) was used as a standard drug. The stock solutions of the oxovanadium(IV) complexes (100 µg/mL) were prepared in dimethylformamide (DMF), and were added to potato dextrose sugar (PDA). This mixture was poured into sterile Petri dishes and allowed to solidify. Fungal spores were inoculated at the center of the medium. Finally, the Petri dishes were incubated at 303 K for 72 hours. The percentage inhibition was calculated by the equation:

$$\% \text{ Inhibition} = (C\text{-}T) \times 100 / C$$

Where C is the diameter of the fungal colony in control plate and T is diameter of fungal colony in test plate. The antifungal activity of the oxovanadium(IV) complexes are summarised below in Table 4.

**Table 4**

The antifungal activity of the oxovanadium(IV) complexes

| Complex | Zone of inhibition (in)* | | Conc. (µg/mL) |
|---|---|---|---|
| | Aspergillus flavus | Aspergillus niger | |
| $[VO(L)]SO_4$ | 64 | 68 | 100 |
| $[VO(mac^1)]SO_4$ | 63 | 69 | 100 |
| $[VO(mac^2)]SO_4$ | 66 | 68 | 100 |
| $[VO(mac^3)]SO_4$ | 76 | 80 | 100 |
| $[VO(mac^4)]SO_4$ | 67 | 69 | 100 |
| **Fluconazole | 100 | 100 | 75 |

**Standard Drug, * average of three replicates

The antifungal activities of oxovanadium(IV) complexes are attributed to the reduced polarity of metal ion after coordination by ligand molecule, which could enhance the lipophilic character of the central metal ion increasing its permeability through the lipid layer of the cell membrane. This significantly enhances the antifungal property of VO(IV) complexes.

## CONCLUSIONS

1,2-Diacetylbenzene has been used as precursor molecule having two reactive carbonyl groups capable of undergoing Schiff base condensation with 2-aminobenzamide in 1:2 molar ratio in aqueous ethanol medium. The parent complex, $[VO(L)]SO_4$ has been reacted with β-diketones to get macrocyclic complexes of the general formula, $[VO(mac)]SO_4$, where mac=macrocyclic ligands derived by reactions of $[VO(L)]SO_4$ with acetylacetone, benzoylacetone, thenoyltrifluoroacetone and dibenzoylmethane, which are thoroughly characterized. The oxovanadium(IV) complexes were tested for their antifungal activity against the fungi Aspergillus flavus and Aspergillus niger, which showed significant antifungal activity especially in the case of $[VO(mac^3)]SO_4$ (76 % and 80 %, respectively). This may be due to sulphur and fluorine atoms present in the thenoyltrifluoroacetone, a β-diketone used as a chelating agent.

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