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# Synthesis, spectral characterization and antimicrobial studies of nano-sized oxovanadium(IV) complexes with Schiff bases derived from 5-(phenyl/substituted phenyl)-2-hydrazino-1,3,4-thiadiazole and indoline-2,3-dione



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#### HIGHLIGHTS

- Oxovanadium(IV) complexes with Schiff bases containing 1,3,4thiadiazole ring have been synthesized and characterized.
- It was found that the oxovanadium(IV) complexes have monoclinic crystal system.
- The fungicidal screening test of all synthesized compounds and its Schiff bases are important in model compound studies.
- Maximum fungicidal activity shown by 2-Cl Substituted complex.

#### ARTICLE INFO

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# ABSTRACT

A new class of oxovanadium(IV) complexes with Schiff bases derived by the condensation of 5-(phenyl/ substituted phenyl)-2-hydrazino-1,3,4-thiadiazoles and indoline-2,3-dione have been prepared in ethanol in the presence of sodium acetate. Micro-analytical data, magnetic susceptibility, UV–Vis, IR, EPR and XRD spectral techniques were used to confirm the structures. Electronic absorption spectra of the complexes suggest a square-pyramidal geometry. The oxovanadium(IV) complexes have monoclinic crystal system and particle sizes were found to be in the range 18.0 nm to 24.0 nm (nano-size). *In vitro* antifungal activity of synthesized compounds was determined against fungi *Aspergillus niger, Collectori-chum falcatum* and *Colletotrichum pallescence* and *in vitro* antibacterial activity was determined by screening the compounds against Gram-negative (*Escherichia coli* and *Salmonella typhi*) and Gram-positive (*Staphylococcus aureus* and *Bacillus subtilis*) bacterial strains. The oxovanadium(IV) complexes have higher antimicrobial effect than free ligands.

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# Introduction

Schiff base ligands are able to coordinate many elements and to stabilize them in various oxidation states. Schiff bases have been known to be used in the preparation of many potential drugs

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http://dx.doi.org/10.1016/j.saa.2014.01.041 1386-1425/© 2014 Elsevier B.V. All rights reserved. and possess a broad spectrum of biological activities such as antimicrobial, anti-inflammatory [1–4]. Other useful applications are sensor [5,6], electrodes [7], conducting polymer [8,9], energy storage [10], enzymatic application [11], solar cell [12], antiviral [13], antifungal [14], anti-inflammatory [15], antitumor [16], antiparasitic [17], antibacterial [18], anti-HIV [19], anticancer [20], etc. Schiff bases derived from 1,3,4-thiadiazole have been synthesized and extensively studied because they have some typical properties such as manifestations of original structures, thermal stability, significant biological properties, high synthesis flexibility and therapeutic utility [21]. Previously, many reports that after complexation with transition metal ions the microbial activity was generally increased [22]. The coordination chemistry of vanadium has acquired renewed interest since the discovery of vanadium in organism such as certain ascidians and amanita mushrooms and as a constituent of the cofactors in vanadate dependent haloperoxidases and vanadium nitrogenase [23]. Recent advances in catalytic and medicinal properties of vanadium complexes have stimulated their design and synthesis [24]. It is also suggested that vanadium could be considered as a representative of a new class of nonplatinum metal antitumor agent [25].

In the present paper, the synthesis, characterization and antimicrobial (antifungal and antibacterial) activities of oxovanadium(IV) complexes with Schiff bases derived from 5-(phenyl/substituted phenyl)-2-hydrazino-1,3,4-thiadiazoles and indoline-2,3-dione are reported.

## Experimental

All the solvents and chemicals used were of reagent grade and used without further purification. Vanadyl sulphate was procured from Aldrich Chemical Co. England. The Schiff bases were synthesized as reported in the literature [26]. Elemental analysis was measured with Perkin-Elmer 1400C analyzer. Infrared spectra (4000–200 cm<sup>-1</sup>) of ligands and complexes were recorded as KBr pellets on a Nicolet-5700 FTIR Spectrophotometer. The magnetic susceptibility at room temperature was measured by Gouy's method using Hg  $[Co(NCS)_4]$  as callibrant. Electronic spectra of the complexes were recorded on Varian Cary-100 Bio UV-Vis-spectrophotometer using DMSO as a solvent. Conductance measurements were recorded in DMSO  $(10^{-3})$  using Elico conductivity bridge type CM-82, provided with a dip type conductivity cell fitted with Pt electrodes. EPR spectra of complexes were recorded at room temperature and liquid nitrogen temperature using Varian-E-4Xband. EPR spectrophotometer operating at microwave frequency  $\sim$ 9.1 GHz, field calibration was checked by using tetracyanoethylene (TCNE) free radical for which g = 2.00277 at room temperature. The particle size of the complexes has been calculated by analysis of X-ray diffraction pattern, obtained using an X-ray powdered diffractometer (Bruker AXS D8 Advance) with Cu K $\alpha 1$  $(\lambda = 1.54060 \text{ Å})$  source.

## Synthesis of Schiff base ligands

A mixture of 5-(phenyl/substituted phenyl)-2-hydrazino-1,3, 4-thiadiazole and indoline-2,3-dione in 1:1 molar ratio, respectively, was refluxed in ethanol (30 cm<sup>3</sup>) containing few drops of concentrated hydrochloric acid for 5–6 h. The product, separated on evaporation of the ethanol, was recrystallized from ethanol ether mixture (1:1).

Synthesis of oxovanadium(IV) complexes

Ethanolic solution  $(20 \text{ cm}^3)$  of vanadyl sulphate (1 mmol) was added to a refluxing ethanolic solution  $(20 \text{ cm}^3)$  of appropriate Schiff base (2 mmol) and sodium acetate (2 mmol). The reaction mixture was refluxed for  $\sim 10$  h. The compound separated in form of amorphous crystals from the clear solution of mixture was filtered, washed with ether and dried *in vacuo*. The empirical formulae, colour, percentage yield, elemental analysis and molar conductance values are listed in Table 1. Synthetic procedure of ligands and oxovanadium(IV) complexes are given in Scheme 1.

## **Results and discussion**

Newly synthesized oxovanadium(IV) complexes with Schiff bases derived from 5-(phenyl/substituted phenyl)-2-hydrazino-1,3,4-thiadiazole and indoline-2,3-dione are coloured, amorphous solids. The complexes are soluble in dimethylformamide and dimethylsulphoxide. The molar conductance values in DMF are in the range of  $16-20 \,\Omega^{-1} \,\mathrm{cm}^2 \,\mathrm{mol}^{-1}$  indicating non-electrolytic behaviour in solution.

#### Magnetic moment and electronic spectra

Magnetic moments of oxovanadium(IV) complexes were studied at room temperature and lie in the range of 1.72–1.78 B.M. These values are well suited for oxovanadium(IV) complexes having one unpaired electron [23]. The electronic spectra of the oxovanadium(IV) complexes in DMF shows three distinct bands in the region of 12,000–12,500, 15,600–16,400 and 22,350–25,000 cm<sup>-1</sup>. These transitions falls in the same range as reported for other five coordinated (C<sub>4v</sub> symmetry) oxovanadium(IV) complexes [27]. The electronic spectra of the two representative complexes are shown in Fig. 1. To interpret the electronic spectra of oxovanadium(IV) complexes, several scheme have been developed. However, the scheme developed by Ballhausen and Gray (the BG scheme) can account well for complexes confirming both to idealized and low symmetry system [28]. These bands can be assigned, on the basis of Ballhausen and Gray energy level scheme,  $b_2 \to e^{*\pi}({}^2B_2 \to {}^2E), \ b_2 \to b_1^*({}^2B_2 \to {}^2B_1) \ \text{and} \ b_2 \to a_1^*({}^2B_2 \to {}^2A_1)$ transitions in increasing order of energy.

#### Infrared spectra

The IR spectra provide valuable information regarding the nature of functional group attached to the metal ion. Schiff bases show a medium intensity band *ca.* 3250–3180 cm<sup>-1</sup> due to v(N-H) which remains almost at the same position in the complexes indicating the non-involvement of (N–H) group in the bond formation [29].The ligands show medium intensity band at *ca.* 1620–1590 cm<sup>-1</sup> assignable to v(C=N) which shifts to lower frequency (*ca.* 20–15 cm<sup>-1</sup>) in the complexes. This shift indicates the coordina-

#### Table 1

Physical properties and analytical data of oxovanadium(IV) complexes.

Complex	Empirical formula	Formula weight	Colour	Yield (%)	Conductance $(\Omega^{-1} \text{ cm}^2 \text{ mol}^{-1})$	Analysis % found (Calcd.)			
		mengine		(,3)	(	V	С	Н	Ν
$[VO(L^1)_2]$ $[VO(L^2)_2]$	$\begin{bmatrix} C_{32}H_{20}N_{10}O_{3}S_{2}V \end{bmatrix} \\ \begin{bmatrix} C_{32}H_{18}N_{10}O_{3}S_{2}CI_{2}V \end{bmatrix} \\ \begin{bmatrix} C_{11}H_{18}N_{10}O_{3}S_{2}CI_{2}V \end{bmatrix} \\ \begin{bmatrix} C_{11}H_{18}N_{10}O_{18}S_{18}CI_{18}V \\ \end{bmatrix} \\ \begin{bmatrix} C_{11}H_{18}N_{18}O_{18}S_{18}CI_{18}V \\ \end{bmatrix} \\ \begin{bmatrix} C_{11}H_{18}N_{18}O_{18}S_{18}V \\ \end{bmatrix} \\ \begin{bmatrix} C_{11}H_{18}N_{18}O_{18}S_{18}V \\ \end{bmatrix} \\ \end{bmatrix} \\ \end{bmatrix} \\ \begin{bmatrix} C_{11}H_{18}N_{18}O_{18}S_{18}V \\ \end{bmatrix} \\ \end{bmatrix} \\ \end{bmatrix} \\ \end{bmatrix} \\ \begin{bmatrix} C_{11}H_{18}N_{18}O_{18}V \\ \end{bmatrix} \\ \end{bmatrix} \\ \end{bmatrix} \\ \end{bmatrix} \\ \end{bmatrix} \\ \end{bmatrix} \\ \begin{bmatrix} C_{11}H_{18}N_{18}O_{18}V \\ \end{bmatrix} \\ $	707.673 776.543	Light green Shining green	71 55 76	19.4 18.8	6.93 (7.19) 6.38 (6.56)	54.12 (54.32) 49.38 (49.49)	2.68 (2.84) 2.26 (2.34)	19.58 (19.79) 17.92 (18.04)
$[VO(L^2)_2]$ $[VO(L^4)_2]$	$[C_{32}H_{18}N_{10}O_{3}S_{2}Cl_{2}V]$ $[C_{32}H_{20}N_{12}O_{7}S_{2}V]$	799.663	green Green	78	15.6	6.14 (6.37)	49.36 (49.49)	2.25 (2.34)	20.94 (21.02)

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