Article



A Vanadyl Complex Bearing an ONO Isonicotinohydrazone Ligand: XRD, Spectroscopy, and Catalytic Sulfoxidation

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Abstract: A new vanadium complex was prepared by reacting $[VO(acac)_2]$ with an ONO isonicotinohydrazone ligand. Various techniques including UV-Vis, FT-IR, CHN, and SC-XRD were utilized to characterize the synthesized complex. The data collected from the diffraction study showed that the vanadium ion is hexa-coordinated and has a distorted octahedral geometry. The V^V ion is coordinated to O,N,O donor sites of the liagnd and a methoxide O atom in the equatorial positions, while methanolic and oxo O atoms occupy the axial sites. Moreover, the catalytic activity of the vanadium complex was studied in the sulfoxidation reaction of different sulfides using hydrogen peroxide.



Keywords: Tridentate Schiff base, Vanadium, SC-XRD, Homogeneous catalysis, Sulfoxidation

1. INTRODUCTION

Transition metal complexes are highly versatile and important in various fields of chemistry. They exhibit a wide range of properties and reactivities due to their ability to undergo different oxidation states and coordination geometries.¹⁻⁴ In recent years, there has been a significant increase in research efforts focused on the synthesis and use of transition metal complexes with Schiff bases derived from substituted salicylaldehydes and amines.⁵⁻⁷ The chemistry of hydrazones remains an exciting field of study due to their adaptability, ease of synthesis, and resistance to hydrolysis.⁸ In general, they contain an acidic proton and deprotonation occurs when they coordinate with transition metals.^{9,10}

Aroylhydrazones are a well-known group of ONO donor ligands that play a crucial role in advancing the field of coordination chemistry as they can coordinate with most transition metals.¹¹ Moreover, the investigation of oxovanadium complexes chelated to tridentate aroylhydrazones ligands has sparked significant interest owing to their resemblance with the active sites of many vanadium-based enzymes.¹²

There are various chemical and biochemical reactions in which vanadium has played a role, and this has caused vanadium to have an attractive and dominant position among transition metals.¹³⁻¹⁸ Vanadium is less sensitive to moisture than many other transition metals. Other characteristic features of vanadium ion include easy switching of oxidation states, use of green oxidants for oxidation of sulfides, flexibility to exhibit numerous possible coordination numbers, affinity for complexation with N/O donor ligands, compatibility with various geometries, and its Lewis acid behavior are attractive to research groups.¹⁹⁻²⁴ Oxovanadium complexes have proven to be highly efficient catalysts for a series of organic reactions, including the oxidation of alkenes, alcohols, and sulfides.²⁵

Sulfoxidation is synthetically important and has consistently attracted the attention of researchers due to its environmental implications.²⁶⁻²⁹ Sulfoxides and sulfones act as synthetic intermediates in the preparation of biological compounds³⁰ and they are also important in organic syntheses as activating groups in C–C bond formation reactions,³¹ asymmetric syntheses,³² and enzyme activation.³³ The facile preparation of these sulfur-containing compounds is a crucial concern in their application for organic synthesis, which has increased their use significantly. In recent years, various transition metal complexes are used to catalyze the oxidation of sulfides to the corresponding sulfoxides and/or sulfones.³⁴⁻⁴⁰

In light of the catalytic importance of homogeneous catalysts and as an extension of our ongoing research on sulfoxidation reaction,⁴¹⁻⁴⁴ in this work, we report a new vanadium complex from 5-bromosalicylaldehyde isonicotinohydrazone Schiff base ligand. Furthermore, the catalytic activity of this complex was investigated in facilitating the green sulfoxidation of different sulfides using hydrogen peroxide in refluxed ethanol.

2. EXPERIMENTAL

Materials and Methods

All materials were purchased from reputable companies, namely Merck and Alfa Aesar. To determine the chemical constituents of the complex, we utilized the Heraeus CHN-O-FLASH EA 1112 equipment. The UV-Vis recorded the UV-1601PC spectra were by spectrophotometer. The FT-IR spectra were recorded the KBr IRPrestige-21 through pellet by the spectrophotometer.

Syntheses

Synthesis of ONO tridentate Schiff base ligand. The (E)-N'-(5-bromosalicylaldehyde isonicotino-hydrazone) ligand was synthesized similarly to a method described in the literature.⁴⁵ 5-Bromosalicylaldehyde (2.01 g, 10 mmol) and isonicotinohydrazide (1.37 g, 10 mmol) were dissolved in 100 mL of hot methanol. The reaction mixture was refluxed for 3 h. The formed yellow product was separated through filtration and washed thrice with cold methanol.

Synthesis of vanadium complex. A mixture of H_2L (3.20 g, 10 mmol) and [VO(acac)₂] (2.65 g, 10 mmol) in 100 mL MeOH was subjected to reflux for 3 h. Following this, the solution was allowed to cool down to room temperature, and the resulting solid product was filtered. The solid product was then washed separately with 10 mL of H_2O , MeOH, and Et₂O, separately.

Yield 73 % (3.27 g). *Anal.* Calc. for $C_{15}H_{15}BrN_3O_5V$: C, 40.20; H, 3.37; N, 9.38, Found: C, 40.37; H, 3.41; N, 9.23%. FT-IR (KBr, cm⁻¹): 3429 (ν_{O-H}) (coordinated methanol); 1604 ($\nu_{C=N}$); 1454 ($\nu_{C=N-N=C}$); 1350 (ν_{C-O}); 1037 (ν_{N-N}); 891 ($\nu_{V=O}$); 575 (ν_{V-O}); 463 (ν_{V-N}).

Crystallographic data of vanadium complex

Using the Mo–K α radiation by a STOE IPDS–II diffractometer, the X-ray diffraction analysis of the vanadium complex was conducted. Table 1 represents the crystallographic data and the refinement parameters.

Typical method for sulfoxidation catalyzed by [VOL(OMe)(MeOH)]

Firstly, a solution containing diphenyl sulfide (0.186 g, 1 mmol) and 30 % aqueous H_2O_2 (2 mmol) in ethanol (5 mL) was prepared. Subsequently, 1 mol% of the complex was added to the solution and refluxed with vigorous stirring. The progress of the reaction was monitored by TLC using *n*-hexane:diethyl ether (70:30). After completion, 3 mL of hot ethanol was added and the mixture was stirred for 10 min. The catalyst was subsequently recycled through filtration and washed three times with hot ethanol. Finally, the product was obtained by evaporating the solvent and subjecting it to purification *via* chromatography over silica gel, utilizing a mixture of

 Table 1. Crystallographic data and structure refinement parameters for vanadium complex

vanadiani eompiex			
Identification code	[VOL(OMe)(MeOH)]		
Empirical formula	C15H15BrN3O5V		
Formula weight	448.15		
Temperature	293(2) K		
Wavelength	0.71073 Å		
Crystal system	Monoclinic		
Space group	P2(1)/c		
	a = 10.8691(12) Å, b = 7.9702(6) Å,		
Unit cell dimensions	c = 20.115(2) Å		
	$\alpha = 90^{\circ}, \beta = 104.661(9)^{\circ}, \gamma = 90^{\circ}$		
Volume	1685.8(3) Å ³		
Z	4		
Density (calculated)	1.766 Mg/m ³		
Absorption coefficient	2.990 mm ⁻¹		
F(000)	896		
Crystal size	$0.31 \times 0.15 \times 0.11 \text{ mm}^3$		
Theta range for data collection	1.94 to 26.00°		
Index ranges	-13<=h<=13, -8<=k<=9, -24<=l<=19		
Reflections collected	7736		
Independent reflections	3256 [R(int) = 0.0629]		
Completeness to theta = 26.00°	98.2 %		
Max. and min. transmission	0.7344 and 0.4575		
Refinement method	Full-matrix least-squares on F ²		
Data / restraints / parameters	3256 / 0 / 228		
Goodness-of-fit on F ²	1.031		
Final R indices [I>2sigma(I)]	R1 = 0.0448, wR2 = 0.0800		
R indices (all data)	R1 = 0.0714, $wR2 = 0.0870$		
Largest diff. peak and hole	0.396 and -0.386 e.Å ⁻³		

n-hexane and diethyl ether in a 70:30 ratio.

3. RESULTS AND DISCUSSION

Synthesis

The reaction between ONO tridentate Schiff base ligand and $[VO(acac)_2]$ in refluxing methanol afforded the related vanadium complex (Scheme 1). Once the complex was successfully prepared and characterized, its catalytic potential was explored by employing H₂O₂ in refluxing ethanol to oxidize different sulfides (Scheme 2).



Scheme 1. Synthesis of vanadium complex



Scheme 2. Vanadium-catalyzed oxidation of sulfides using H2O2

FT-IR spectra

Figure 1 represents the FT-IR spectra of 5bromosalicylaldehyde isonicotinohydrazone and its vanadium complex. The stretching vibrational bands for the hydrazoneic (NH) and carbonyl (C=O) groups were observed at 3421 and 1674 cm⁻¹ in the ligand spectrum. These eliminated in the complex spectrum due to tautomerization and involvement in complexation. The vibrational band of azomethine chromophore, which appeared in the ligand spectrum at 1616 cm⁻¹, was shifted to 1604 cm⁻¹ upon complex formation and the binding of iminic nitrogen to the vanadium ion. Also, the oxovanadium complex gave characteristic V=O, V–O, and V–N bands at 891, 575, and 463 cm⁻¹, respectively. These values align with those reported for similar complexes.⁴⁶⁻⁵⁰



Figure 1. FT-IR spectra of H_2L ligand and [VOL(OMe)(MeOH)] complex.

UV-Vis spectral details

The electronic spectra of both the Schiff base ligand and its vanadium complex were recorded from 270 to 500 nm in dimethylformamide (DMF) at room temperature (Figure 2). The Schiff base ligand exhibited four distinct bands at 285, 300, 330, and 400 nm attributed to $\pi \rightarrow \pi^*$ and $n \rightarrow \pi^*$ electronic transitions. Notably, upon coordination with the vanadium metal, the UV-Vis spectrum of the complex displayed a noticeable blue shift. The lowest energy transition band is observed near 415 nm, which is attributed to a LMCT transition as a charge transfer from a *p*-orbital on the lone-pair of ligand oxygen atoms to the empty *d*-orbital of the vanadium atom. The other strong band in the region 329 is the same as the absorption band in the ligand, so it is attributed to the intraligand $\pi \to \pi^*$ absorption peak of the Schiff base. The other mainly LMCT and to some extent $\pi \to \pi^*$ band appears at about 275 nm, and this is due to oxo, methoxylato and L oxygen atoms bound to vanadium(V).51-54



Figure 2. UV-Vis spectra of H_2L ligand and [VOL(OMe)(MeOH)] complex.

Crystal structure determination

The asymmetric unit of the title complex comprises a vanadium hydrazone complex in which the vanadium center is coordinated by a tridentate hydrazone ligand (ONO), a methoxide, and a coordinated methanol with distorted octahedral geometry (Figure 3). The axial V2–O4 [2.375(3) Å] bond is significantly longer than the equatorial V2–O5 [1.758(2) Å] due to the strong π -donor character of the oxo (O3) ligand. The crystal packing of the complex is consolidated by the intermolecular O–H…N, C–H…O, and C–H…N interactions. The intermolecular O–H…N, C–H…O, and C–H…N interactions form centrosymmetric dimers in the solid state (Figure 4).



Figure 3. The ORTEP of the title complex with 40% ellipsoid probability and selected atom numbering.

Catalytic activity studies

After synthesizing and characterizing the oxovanadium complex of (E)-N'-(5-bromosalicyladehyde isonicotinohydrazone), its catalytic activity was studied in the sulfoxidation reaction. The oxidation of diphenyl sulfide was chosen as the model reaction to obtain the optimal conditions. Preliminary experiments revealed that the most favorable outcomes were obtained by utilizing 2 mmol H_2O_2 as the oxidant in refluxing ethanol and incorporating 1 mol% of the catalyst (Figure 5).



Figure 4. The centrosymmetric dimer formation by the intermolecular O-H^{...}N, C-H^{...O}, and C-H^{...N} interactions.



Figure 5. Effect of various factors on the sulfoxidation of diphenyl sulfide using [VOL(OMe)(MeOH)] complex.

To explore the versatility of this procedure, a range of aromatic and aliphatic sulfides were subjected to a reaction with 2 mmol H_2O_2 and 1 mol% catalyst in refluxed ethanol to produce the corresponding sulfoxides and sulfones (Table 2). Remarkably, most sulfides underwent complete conversion into their respective sulfoxides as the sole products. In some instances, a small amount of sulfone was also obtained. Investigations show that in the reaction conditions, sulfoxide is produced first, which gradually turns into sulfone as the reaction time increases due to overoxidation.

No	Substrate	Time (min)	Yield (%) (Sulfoxide/Sulfone) ^a	
1	<u>~^\$</u> ~	15	92 (100/0)	
2	~~ ^{\$} ~~	20	91 (100/0)	
3	~~~s~~~	10	89 (100/0)	
4	S-CH3	15	90 (85/15)	
5	√−s−√	30	94 (78/22)	
6	S-CH ₂	20	89 (90/10)	
7	S-CH ₂ -CI	20	93 (87/13)	
8	S-CH ₂ -CH ₃	30	90 (91/9)	
9		15	88 (92/8)	

^aEntries 1-4: GC yield and entries 5-9: isolated yield.

Table ? Vanadium catalyzed sulfavidation using H.O.

A potential mechanism for the oxidation of sulfides using hydrogen peroxide catalyzed by **[VOL(OMe)(MeOH)]** complex is proposed and illustrated in Scheme 3, based on relevant literature on oxidation reactions mediated by oxometal complexes.⁵⁵⁻⁵⁷



Scheme 3. Plausible mechanism for vanadium-catalyzed sulfoxidation using $\mathrm{H}_{2}\mathrm{O}_{2}$

Table 3 presents a comparison of the catalytic efficiency of **[VOL(OMe)(MeOH)]** complex in the sulfoxidation of diphenyl sulfide to the corresponding products, along with several protocols previously documented in other manuscripts. The results of this study show that the present method has shorter reaction time and higher yield of products in the presence of lower amounts of catalyst.

Entry	Catalyst	Conditions	Time (min.)	Yield (%)	Ref.
1	cis-[MoO2(phox)2]	UHP, CH2Cl2/MeOH, r.t.	20	62	[58]
2	MoO ₂ L(C ₂ H ₅ OH)	UHP, CH ₂ Cl ₂ /MeOH, r.t.	30	55	[59]
3	V(IV)O-MCM-41	UHP, EtOH, r.t.	12 days	89	[60]
4	VO(acac)@Sb@SMNPs	TBHP, DCE, 85 °C	60	71	[61]
5	[VOL(OMe)]/MNPs	H ₂ O ₂ , EtOH, reflux	30	90	[41]
6	MoO ₂ (L)(CH ₃ OH)	H ₂ O ₂ , EtOH, reflux	60	92	[43]
7	[MoO ₂ L(CH ₃ OH)]/MNPs	H ₂ O ₂ , EtOH, reflux	60	91	[44]
8	[VOL(OMe)(MeOH)]	H ₂ O ₂ , EtOH, reflux	30	94	This work

Table 3. Comparison of catalytic activity of [VOL(OMe)(MeOH)] complex with some other reported catalysts in the sulfoxidation of diphenyl sulfide

4. CONCLUSIONS

A novel vanadium complex, incorporating an ONO tridentate Schiff base ligand, was successfully characterized using advanced prepared and spectroscopic and physicochemical techniques. The spectroscopic evidence unequivocally demonstrates that the ligand acts as a tridentate, forming a highly stable complex with the vanadium ion through the azomethine N-atom, as well as the O-atoms of the enolic and phenolic groups. This synthesized vanadium complex was employed as a homogeneous catalyst in oxidizing various sulfides using H₂O₂. The results obtained from these experiments are highly promising, as most sulfides underwent complete conversion into their respective sulfoxides, serving as the sole products. In some cases, a minor amount of sulfone was also observed. Further investigations have revealed that, under the specific reaction conditions employed, the sulfoxide formation occurs initially. However, as the reaction time increases, overoxidation takes place, leading to the gradual transformation of sulfoxide into sulfone.

CONFLICTS OF INTEREST

The authors hereby affirm that they do not possess any conflicts of interest.

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