

Facile Room Temperature Synthesis, Characterization, Molar Conductance and Magnetic Properties of Mixed Ligand Complexes of Vanadium (IV) Using 2, 2'-Bipyridine and Amino Acids as Ligands

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ABSTRACT

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Facile room temperature synthesis of four new mixed ligand complexes (C1-C4) of vanadium(IV) using 2, 2'-bipyridine as primary ligand and L-amino acids such as L-Alanine, L-Cystine, L-Methionine and L-Threonine as secondary ligands respectively is reported. The synthesized complexes were characterized using melting point/decomposition temperature determination, elemental analysis, solubility behaviour, IR spectra, molar conductance and magnetic susceptibility measurement. All the synthesized complexes are proposed to have square pyramidal geometry based on the results obtained.

Keywords: Amino acids, 2, 2'-Bipyridine, Magnetic property, Molar conductance, Square pyramidal and Vanadium.

I. INTRODUCTION

In recent time increased attention is paid to the research area dealing with synthesis, characterization and applications of mixed ligand complexes of transition metals. Mixed ligand complexes are found to exhibit better biological activities as compared to simple complexes which is devoted to the presence of more than one type of ligands coordinated to central metal atom [1, 2]. The literature survey reveals that mixed ligand oxovanadium(IV) complexes show modulating activities of various enzymes [3, 4]. These complexes also have biological activities such as antibacterial, antifungal, antiviral, and anticancer drugs [5-7]. The mixed ligand transition metal

complexes with benzoheterocyclic rings and some L-amino acids have been the focus of a considerable number of investigations for their good coordination ability with metal atoms [8]. A large number of mixed-ligand complexes involving heterocyclic bases such as pyridine, 2,2'-bipyridine, o-phenanthroline, etc. were reported by many researchers owing to their biological applications and thermal stability [9]. In present investigation we report facile room temperature synthesis of four mixed ligand complexes (C1-C4) of vanadium(IV) using 2,2'-bipyridine and L-amino acids such as L-Alanine, L-Cystine, L-Methionine and L-Threonine ligands respectively. All the synthesized complexes were characterized using melting point determination, elemental analysis,

solubility behaviour, IR spectra, molar conductance and magnetic susceptibility measurements. Based on the results obtained all the synthesized complexes were proposed to have square pyramidal geometry.

II. EXPERIMENTAL SECTION

Materials

All the chemicals used i.e. vanadyl sulphate, 2,2'-bipyridine and all the amino acids were from purchased from S.D. Fine Chemicals, Private Limited, Mumbai and were of AR grade. All the solvents were purified using recommended method before use [10]. Melting points or decomposition temperatures of all the synthesized compounds were measured using a simple capillary tube method and are uncorrected. Molar conductance values of all the synthesized complexes were measured by preparing 10^{-3} M solutions in DMF solvent using Equiptronics conductivity meter with an inbuilt magnetic stirrer (Model:Eq-664) at room temperature. Magnetic susceptibilities were determined on the SES Instrument's magnetic susceptibility Gouy's balance (Model: EMU-50) at room temperature using copper (II) sulphate as a standard. IR spectra of complexes were recorded as KBr pellets in the region of $4000-400\text{ cm}^{-1}$ on a SHIMADZU Spectrophotometer.

General procedure for the synthesis of mixed ligand complexes

To an aqueous solution (20 mL) of vanadyl sulphate (1.63 g, 0.01 Mole) add ethanolic solution (20 mL) of 2,2'-bipyridine (1.56 g, 0.01 Mole) was added drop wise with constant stirring. The mixture was stirred for 1 h at room temperature. To this reaction mixture was added an aqueous solution (20 mL) of respective amino acids (0.01 Mole) in drop wise manner with constant stirring. The reaction mixture was then allowed to stir for 5-7 h at room temperature. The colored complexes precipitated were filtered, washed with cold distilled water followed by ethanol. The

complexes were then dried at room temperature and used for further study. Figure 1 given below represents the synthesis scheme of mixed ligand complexes.

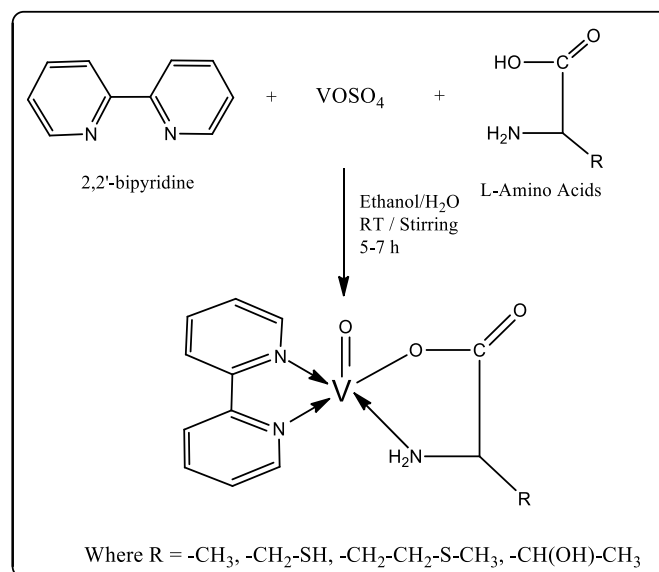


Figure 1 : Scheme representing synthesis of mixed ligand complexes

III. RESULT AND DISCUSSION

The complexes can be represented by general formula $[M(L_1)(L_2)]$ Where $M=V(IV)$, $L_1 = 2,2'$ -bipyridine and $L_2 = L$ -aminoacids such as L-Alanine, L-Cystine, L-Methionine and L-Threonine. The molecular weight and chemical formula are estimated for all the synthesized complexes using 'Chem Draw ultra 11.0' software.

Physicochemical data

Physical properties such as colour, melting points/decomposition temperatures, percentage yield were recorded for all the complexes. All the complexes are found to be decomposed at more than 260°C temperature indicating their thermal stability. Physicochemical data along with elemental analysis, molar conductance and magnetic properties recorded for all the synthesized mixed ligand complexes is represented in Table 1.

Table 1. Physicochemical data of synthesized mixed ligand complexes

Complex	Proposed Molecular Weight (g)	Colour	Yield (%)	Elemental analysis				Molar conductance (Ohm ⁻¹ ·Cm ² ·mol ⁻¹)	Magnetic susceptibility μ_{eff} (BM)
				C	H	N	S		
C ₁ C ₁₃ H ₁₄ N ₃ O ₃ V	311.21	Faint Brown	65	50.17 (50.19)	4.53 (4.54)	13.50 (13.48)	--	12	1.87
C ₂ C ₁₃ H ₁₄ N ₃ O ₃ SV	343.28	Black	68	45.49 (45.48)	4.11 (4.10)	12.24 (12.20)	9.34 (9.33)	15	1.91
C ₃ C ₁₅ H ₁₈ N ₃ O ₃ SV	371.33	Black	72	48.52 (48.50)	4.89 (4.87)	11.32 (11.32)	8.64 (8.63)	21	1.89
C ₄ C ₁₄ H ₁₆ N ₃ O ₄ V	341.24	Fait Green	73	49.28 (49.26)	4.73 (4.72)	12.31 (12.30)	--	23	1.92

Solubility behaviour

The solubility behaviour of all the synthesized mixed ligand complexes was checked with solvents such as water, methanol, ethanol, acetone, ethyl acetate, chloroform, DMSO, DMF etc. available in the

laboratory. The complexes were found to be insoluble in common organic solvents and were soluble in DMF, DMSO and CHCl₃. Table 2 represents the results obtained.

Table 2. Solubility of all the synthesized mixed ligand complexes

Complex	Water	Methanol	ethanol	Acetone	Ethyl acetate	Chloroform	DMSO	DMF
C ₁ C ₁₃ H ₁₄ N ₃ O ₃ V	I	I	I	I	I	S	S	S
C ₂ C ₁₃ H ₁₄ N ₃ O ₃ SV	I	I	I	I	I	S	S	S
C ₃ C ₁₅ H ₁₈ N ₃ O ₃ SV	I	I	I	I	I	S	S	S
C ₄ C ₁₄ H ₁₆ N ₃ O ₄ V	I	I	I	I	I	S	S	S

I= insoluble, S= soluble

IR Spectra

IR spectra of synthesized mixed ligand complexes were recorded and compared with those of free ligand molecules. The some major absorption bands observed are expressed here. The absorption band of the (C=N) group of free bipyridine ligand molecule observed at 1454 cm⁻¹ [13] was shifted to lower frequency in IR spectra of complexes in the range of

1435-1445 cm⁻¹ indicating bipyridine ligand is coordinated to metal atom via the nitrogen atom.

Similarly the asymmetric and symmetric (COO⁻) bands observed in the region 1580-1597 and 1402-1408 cm⁻¹ in case of free amino acids were observed to be shifted to lower wave number region of 1571-1575 and 1373-1377 cm⁻¹ respectively in the spectra of complexes. This indicates bonding of COO⁻ group with vanadium metal via oxygen atom of carboxylic

group of amino acids. The band observed between 940-950 cm^{-1} in the spectra of complexes indicates (V=O) stretching vibration. Finally the bands observed in the range of 440-450 and 620-635 cm^{-1} indicates $\nu(\text{M-N})$ and $\nu(\text{M-O})$ bonding in complexes respectively.

Molar conductance

Molar conductance values for all the synthesized complexes were recorded by preparing 10^{-3} M solutions in DMSO solvent. The observed molar conductance values in the range (12-23 $\text{Ohm}^{-1} \cdot \text{Cm}^2 \cdot \text{Mol}^{-1}$) for complexes indicated towards their non-electrolytic nature of complexes.

Magnetic properties

Magnetic susceptibility values for all the synthesized mixed ligand complexes were recorded at room temperature using copper (II) sulphate as an internal standard. These were then converted into magnetic moment values using spin only formula. The magnetic moment values of all the four complexes were found in the range of 1.87 to 1.92 BM indicating presence of one unpaired electron in these complexes. Thus confirming oxidation state of vanadium in complexes is +4 in all these complexes [14]. Thus all the four complexes exhibited square planar geometry around the vanadium metal atom.

IV. CONCLUSION

Four mixed ligand complexes of vanadium(IV) (**C₁-C₄**) were synthesized at room temperature using bipyridine and L-amino acids as ligands. The elemental analysis data reveals 1:1:1 (M:L:L) proportion in complexes. The decomposition temperature values recorded for all the complexes were $>260^\circ\text{C}$ indicating thermal stability of complexes. IR spectra, molar conductance and magnetic moment values of complexes reveal that vanadium is having +4 oxidation states with one unpaired electron present in

all these complexes. Thus all the complexes are having square pyramidal geometry.

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