Egypt. J. Chem. **Vol. 65,** No. SI:13B pp. 263 - 271 (2022)

Egyptian Journal of Chemistry

http://ejchem.journals.ekb.eg/

Synthesis and study conductmetrically parameters of trans hydroxyl-4-proline with vandyl complex and its biological activity

Radwa T. Rashad*

Misr higher Institute for Engineering and Technology, Mansoura, Egypt

Abstract

This study aims to investigate the thermodynamic parameters for nano VO (II) sulfate alone and with amino acid (H2Prol) to form complexes in the solutions, and metal complexe have been characterized on the basis of elemential analysis, electronic spectra, IR spectra and various physico chemical study. The measurements of vandyl sulphate conductance have happened in binary mixed solvent with ethanol in different mass fraction 0.0, 0.2 and 0.4 (W/W) (EtOH-H2O) at four different temperatures from 298.15 to 313.15K (with a step of 5 K). The experimental data obtained by using the Fuoss-Shedlovsky extrapolation technique. All thermodynamic parameters for association were calculated, also we studied the effect of hydrogen bond formation in solution. Furthermore, biological activity measurements are executed, which benefits in determining the factors impacting the thermodynamic parameters

Keywords: Limiting molar conductance, Ion-pair association Constants, Binary Mixed solvents, Walden product, Association constant.

1. Introduction

Proline is an amino acid needed for the production of collagen and cartilage. It keeps muscles and joints flexible and helps reduce sagging and wrinkling that accompany UV exposure and normal aging of the skin. Thermodynamic properties are very useful study of the intermolecular interactions and geometrical effects in the systems, thermo-physical and bulk properties of solutions. Also its necessary in theoretical and applied areas of research and used in many other fields of industry [1-3]. Studying the information of the transport properties (conductance, viscosity, ionic mobility) of electrolytes in aqueous and partially aqueous media tell us all about ion-ion and ion-solvent interactions in these solutions [3-9]. The Fuoss-Shedlovsky is one of the mathematic equation of conductivity theories, which has been successfully used to investigate many electrolytes in solutions [9-15]. The physical properties of the binary mixed solvents like the viscosity and the relative permittivity can be varied and this making them more favor to solvent system for the study of ion association and ion mobility. Recently, a study of the properties of vandyl sulphate is essential in many fields such as biochemistry and in other different industry. The more uses of vandyl sulphate as a coloring ingredient in artworks, especially glasses and potteries.

The present article show the effect of all parameters on the transport properties of vandyl sulphate in a binary mixed solvent with alcohol mass fraction 0.0, 0.2 and 0.4 (W/W) (EtOH-H2O) at four different temperatures from 298.15 to 313.15K (with a step of 5K). By applying the Fuoss- Shedlovsky conductivity equation [16].

2. Experimental

2.1. Chemicals

All chemicals and all were exploited without any former purification. The used amino acids (ligands) are (H2L) supplied from BDH chemicals Ltd as solids. The metal salt used is purchased from Nice Laboratory, India. The water contents were determined through the (Mettler DL 18 Karl Fischer Titrator) and were shown to be smaller than 0.01%. All the glassware was left in the chromic mixture for a day and then cleansed many times with water, distilled water, and finally with bidistilled water and then kept in an electric oven to dry. Bidistilled water was obtained by redistilling the ordinary distilled water over KMnO4 and KOH. The first 25 ml were excluded. Measured specific conductance was found to be 2-5 x10-7S cm^{-1}.

*Corresponding author e-mail: radwa_tarek@engmet.edu.eg.
Receive Date: 07 February 2022, Revise Date: 23 March 2022, Accept Date: 29 March 2022
DOI: 10.21608/EJCHEM.2022.120565.5409

Solvents used are Ethanol (EtOH), pure water (H2O) and mixed solvent of Ethanol-water of different percentages (EtOH-H2O) which supplied from El-Goumhouria CO.

The structures and properties of material, ligand and solvent which used in this study are shown in Tables (1-3).

Table (1) Structure and properties of Vanadium oxide sulphate.

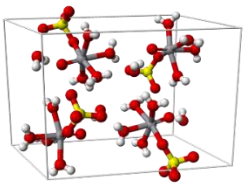
Chemical name	Vanadium oxide sulphate
Structure	 VOSO4.xH2O
Molar mass	163 g.mol-1
Odour	Odorless
Density	N/A
Melting point	105 °C (221 °F; 378 K) decomposes

Table (2) Structure and properties of ethanol.

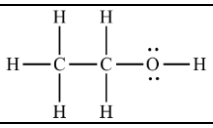
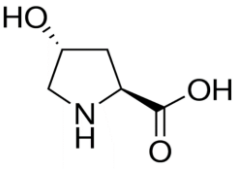
Chemical name	Ethanol
Structure	
Molar mass	46.07 g.mol-1
Odour	Pleasant
Density	0.7893 g/cm3
Melting point	-114.14 °C (-173.45 F –159.01K)
Boiling point	78.24 °C (172.83F –351.39K)

Table (3) Structure and properties of Trans-4-hydroxy proline.

Properties	Trans-4-hydroxy proline
Structure of Trans-4-hydroxy proline	
IUPAC name	(2S,4R)-4-hydroxypyrrolidine-2-carboxylic acid
Molar mass	131.131 g.mol-1
Density	1.907gm/cm3
Melting point	274°C
Boiling point	Decomposed

23. Results and discussion

3.1. *Association parameters of the bulk and nano metals in the presence of ligand (trans-4-hydroxy proline)*

The effects of metal salt concentration , type of solvent used, the temperature and the presence of ligand on the molar conductance (Λm) and hence limiting molar conductance (Λ_o) were studied. However, the influences of all the above-mentioned factors on the values of Walden product ($\Lambda_o \eta_o$), degree of dissociation (α), dissociation, association (KD, KA) and Gibbs free energies of association (Δ GA) were discussed.

The limiting molar conductance (**Λ**o) at infinite dilutions were estimated for a metal salt in water and absolute ethanol at different temperatures in the presence of the ligand by extrapolating the relation between (Λm) and (C½) to zero concentration.

The values of (Λ_o , Λ_m, $\gamma_{\pm}$, KA, KD, α and Δ GA) for the solutions (0, 20, 40%) EtOH-H2O were calculated and are reported in tables for VOSO4 in EtOH-H2O in the presence of amino acids (trans-4-hydroxy proline) at different temperatures (298.15, 303.15, 308.15 and 313.15K) were calculated by using equation below [17-27].

$$KA = \frac{\Lambda 0\ (\Lambda 0 - S(Z)\ \Lambda m\ )}{Cm\ \Lambda m2\ S(Z)2\gamma\pm2} \quad \ldots\ldots\ldots(1)$$

Where (Λm, Λ0) are the molar and limiting molar conductance of nano-VoSO4 in presence of ligand respectively, Cm is the molar concentration of nano-VoSO4, S(Z) is Fouss–Shedlovsky factor, equal with unity for strong electrolytes, γ± is the mean activity coefficient.

3.2 Relation between Λm and C1/2 for VOSO4 in presence of trans-4-hydroxy proline

The relation between Λm and C1/2 for $VOSO_4$ at 298.15K in presence of trans-4-hydroxy proline at (0%, 20% and 40%) EtOH-H2O.

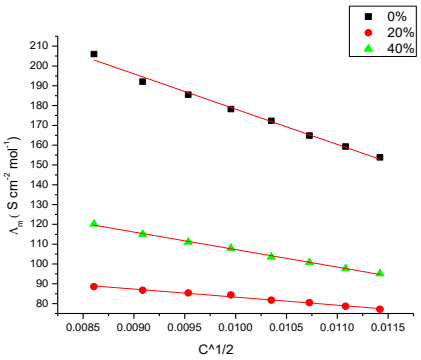


Fig. (1) The relation between molar conductance (Λm) and C^1/2 of VOSO4 at 298.15K temperature.

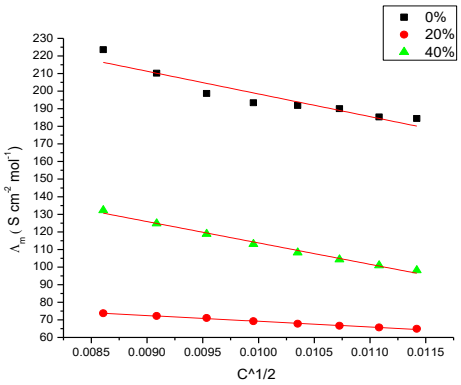


Fig. (2) The relation between molar conductance (Λm) and C^1/2 of VOSO4 at 303.15K .

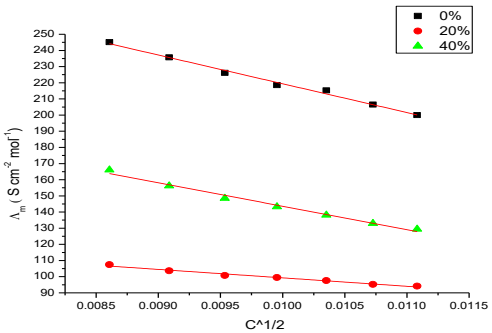


Fig. (3) The relation between molar conductance (Λm) and C^1/2 of VOSO4 at 308.15K temperature.

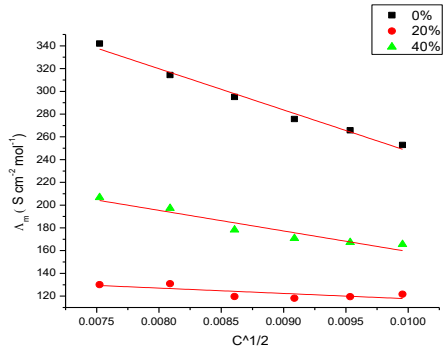


Fig. (4) The relation between molar conductance (Λm) and C^1/2 of VOSO4 at 313.15K

Table (4) The value of mole fractions (XS), the values of viscosity *(η*o), limiting molar conductance (Λ), molar conductance (Λm), Walden product (Λo*ηo*), Fuoss-Shedlovsky parameters (S, Z and S(z)), activity coefficient (*Y*±), and dissociation constant (KD) for nano VOSO4 in presence of trans-4-hydroxy proline in mixed solvent (EtOH-H2O) at four different temperatures.

T(K)	XS	102η	Λo	Λm	Λo*η*o	S	Z	S(z)	*Y*±	103 KD
298.15K	0	0.8921	256.35	185.46	2.2868	119.3848	0.0041	1.0041	0.959	0.19
	0.0717	0.9042	123.95	85.36	0.9570	85.1164	0.0073	1.0073	0.957	0.32
	0.1708	0.9209	195.84	111.18	1.7470	105.4742	0.0041	1.0042	0.964	0.07
303.15K	0	0.8001	**327.34**	198.66	2.6190	143.9993	0.0040	1.0040	0.957	0.12
	0.0717	0.8082	**141.69**	71.06	1.1336	100.7331	0.0059	1.0059	0.960	0.18
	0.1708	0.8193	**235.66**	118.89	1.8855	122.633	0.0043	1.0043	0.961	0.06
308.15K	0	0.7222	**397.36**	226.16	2.7819	171.837	0.0038	1.0038	0.958	0.09
	0.0717	0.7329	**151.55**	100.76	1.0610	113.6552	0.0072	1.0072	0.954	0.17
	0.1708	0.7478	**288.97**	148.52	2.0230	146.1817	0.0042	1.0042	0.959	0.06
313.15K	0	0.6211	**609.31**	265.76	3.6119	238.8199	0.0030	1.0030	0.962	0.04
	0.0717	0.6911	**164.60**	119.46	0.9757	131.9109	0.0080	1.0080	0.952	0.16
	0.1708	0.7092	**340.26**	167.26	2.0170	174.1398	0.0042	1.0042	0.960	0.06

Λ o in (S cm2.mol-1), Λ_m in (S cm2.mol-1)

Table (5) Degree of dissociation (α), association constant (KA), triple ion association constant (K3), Gibbs free energy of association (ΔGA), fluidity ratio (Rx), and transfer free energy of association (ΔGt) for VOSO4 in presence of trans-4-hydroxy proline in mixed solvent (EtOH-H2O) at 298.15K

VOL.% OF EtOH-H2O	105K3	α	KA	Δ GA	Rx	Δ Gt
0%	0.3749	0.4264	5257.64	-21.241	0.0960	0
20%	0.2226	0.5014	3147.38	-19.9688	0.0401	1.2722
40%	0.8851	0.5696	10120.57	-22.545	0.0733	-1.304

Table (6) Degree of dissociation (α), association constant (KA), triple ion association constant (K3), Gibbs free energy of association (ΔGA), fluidity ratio (Rx), and transfer free energy of association (ΔGt) for VOSO4 in presence of trans-4-hydroxy proline in mixed solvent (EtOH-H2O) at 303.15K.

VOL.% OF EtOH-H2O	105K3	α	KA	Δ GA	Rx	Δ Gt
0%	0.9300	0.4093	8326.056	-22.756	0.1099	0
20%	0.9034	0.5044	5285.61	-21.6105	0.0476	1.1455
40%	1.5092	0.5063	12122.41	-23.2604	0.0791	-0.4956

Table (7) Degree of dissociation (α), association constant (KA), triple ion association constant (K3), Gibbs free energy of association (ΔGA), fluidity ratio (Rx), and transfer free energy of association (ΔGt) for VOSO4 in presence of trans-4-hydroxy proline in mixed solvent (EtOH-H2O) at 308.15K

VOL.% OF EtOH-H2O	105K3	α	KA	Δ GA	Rx	Δ Gt
0%	1.1174	0.5713	10376.46	-23.6955	0.1168	0
20%	1.6883	0.6696	5862.509	-22.2324	0.0445	-1.4631
40%	1.8431	0.8160	14295.36	-24.5164	0.0849	-0.8209

Table (8) Degree of dissociation (α), association constant (KA), triple ion association constant (K3), Gibbs free energy of association (ΔGA), fluidity ratio (Rx), and transfer free energy of association (ΔGt) for VOSO4 in presence of trans-4-hydroxy proline in mixed solvent (EtOH-H2O) at 313.15K

VOL.% OF EtOH-H2O	105K3	α	KA	Δ GA	Rx	Δ Gt
0%	2.0129	0.4374	13001.4	-23.1528	0.1516	0
20%	3.3640	0.7316	6013.22	-22.6593	0.0409	0.4935
40%	4.5923	0.9342	16358.06	-25.2652	0.0846	-2.1124

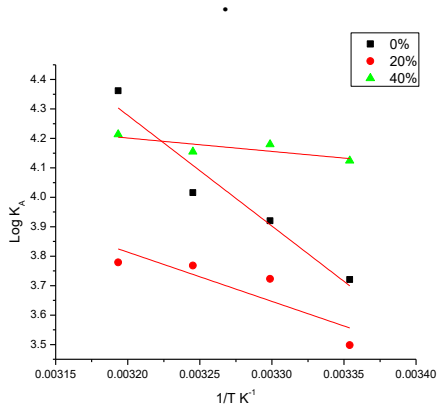


Fig (5) Relation between log KA and 1/T for VOSO4 in presence of trans-4-hydroxy proline at different temperatures.

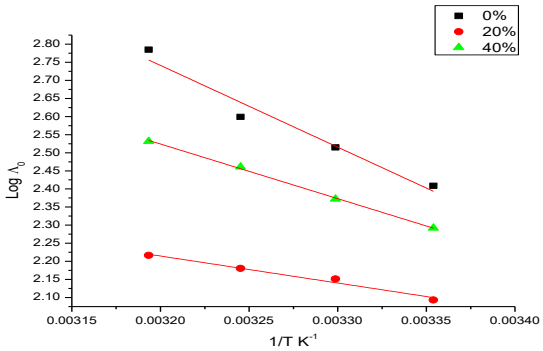


Fig (6) Relation between log Λ_o and 1/T for VOSO4 in different (EtOH-H2O) percentages

3.3 Activation energy of association of VOSO4 in presence of trans-4-hydroxy proline

The association constants, activation energy enthalpies and entropies of association for VOSO4 at different concentrations of ethanol–water at different temperatures are tabulated in Table (9) Association constants, activation energy, enthalpies and entropies of association for VOSO4 at different temperatures in presence of trans-4-hydroxy proline.

T (K)	Xs	Ea	ΔHA kJ.mol-1	TΔSA kJ.mol-1	ΔSA J.mol-1
298.15	0	9.9713	71.9884	93.2294	312.6931
	0.0717	4.6014	8.6922	29.6610	98.1296
	0.1708	7.3551	32.0119	55.5575	186.3411
303.15	0	9.9713	71.9884	94.7444	312.5334
	0.0717	4.6014	8.6922	30.3027	99.9596
	0.1708	7.3551	32.0119	56.2723	185.6254
308.15	0	9.9713	71.9884	95.6839	310.5109
	0.0717	4.6014	8.6922	30.9246	100.3558
	0.1708	7.3551	32.0119	56.5283	183.4443
313.15	0	9.9713	71.9884	98.1412	313.4001
	0.0717	4.6014	8.6922	31.3514	100.1165
	0.1708	7.3551	32.0119	57.2771	182.9064

3.4 Effect of H2L and its metal complexes toward S. aureus, E. coli, and C. albicans

H2L and its metal complexes were investigated for antibacterial activity against Gram-positive bacteria (*Staphylococcus aureus*), Gram-negative bacteria (*Escherichia coli*), and fungal (*Candida albicans*). Ampicillin (antibacterial) and clotrimazole (antifungal) were used as reference drugs [28-30]. The outcomes were recorded for all compounds under investigation as the average diameter of inhibition zones of bacterial or fungal growth around the discs in mm.

From the results obtained for the diameter of the inhibition zone of Proline and its metal complexes as represented in Figs (7) we concluded that the most of isolated complexes as bulk copper complexes and bulk and nano zinc complexes have an intermediate antimicrobial activity except vandyl complexes has the largest antimicrobial activity.

Complexes of proline showed selectivity against G- bacteria as in figure complex of VO(II) has the most potent (84%) followed by Zn(II) (48%).

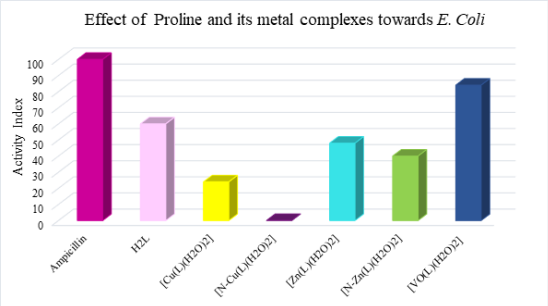


Fig. (7) Effect of trans-4-hydroxy proline and its metal complexes towards *Escherichia coli*

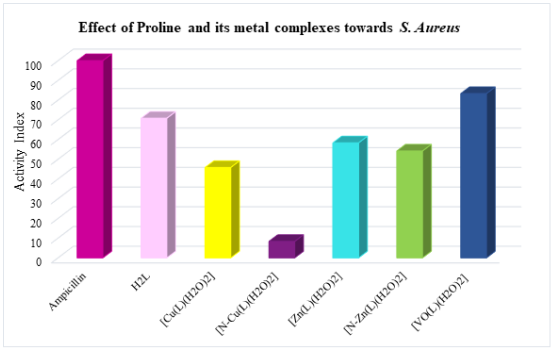


Fig. (8) Effect of trans hydroxyl proline (H2L) and its metal complexes towards *Staphylococcus aureus*

H2L showed antibacterial effect against G+ bacteria for VO(II) largest one*(83.3%) followed by Zn(II)complex* (58.2%) and nano Zn (II) complex (54.2%).

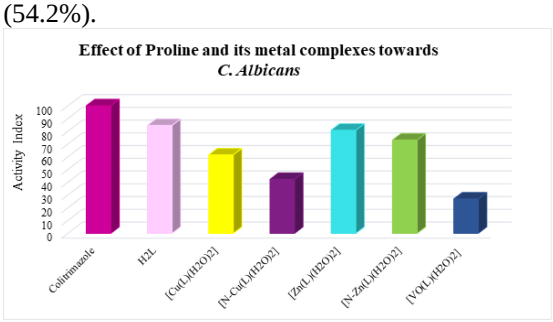


Fig. (9) Effect of trans -4-hydroxy proline (H2L) and its metal complexes towards *C. albicans*

Also showed towards *C. albicans* that Zn(II) complex has the most potent antibacterial (80.8%) followed by Cu(II) complexes (61.5%) but Vanadyl complex has the lowest antibacterial effect towards *C. albicans*

Isolation of metal complexes

The new complexes of (H2L), with VO(II) metal ions were performed and described by conventional chemical and physical methods. All the solid complexes were isolated in a pure state. The results of elemental analyses and some physical

properties of the metal complexes collected in tables (10-11).

The comparison, between the experimental and theoretical results showed that the compositions of the isolated complexes established with the suggested formula. There was one types of metal complexes were isolated from the reaction of hydrazones under examination with some metal salts where the ligands behaved in, bi dentate manner. All solid complexes were stable in air and insoluble in organic solvents however soluble in dimethyl formamide (DMF) and dimethyl sulphoxide (DMSO) easily.

Table (10) Elemental analysis and physical data of H2L and its metal complexes.

Compound	Formula	M.Wt	Yield	color	m.p	C	H	M
H2L	C5H9NO3	131.131	80	white	275	45.80 (45.83)	(6.92) (6.94)	_
[VO(L)(H2O)2]	VOC5H11NO5	232.094	70	Dark green	>300	25.87 (25.82)	(2.62) (3.12)	21.95 (21.90)

Table (11) Most important IR spectral bands of H2L and its metal complexes

Compound	υ(OH)	υ(NH)as	υ(C=O)s	υ(C=O)as	M-N	M-O
H2L	3285	3138	1591	1399	---	---
[VO(L)(H2O)2]	3565	---	1543	1340	452	539

Infrared and electronic spectra studies

A substantial part of knowledge concerning the mode of bonding in metal chelates can be gained by applying infrared spectroscopy [31-.35] To achieve an idea about the groups involved in complex formation as well as the influence of the electric field of the central metal ion on the charge distribution within the ligand, The difference between the spectra of the free ligands and those of the complexes can fall in the following types:

i. Band splitting **ii**. Intensity change **iii**. shifts in band position **iv**. Disapperance of bands and appearance of new bands

4.C.2.1 Infrared of H2L and its metal complexes

The most important infrared bands of H2L and its metal complexes are listed in table (11) and represented graphically in figures. The IR spectrum of H2L display four bands at 1591, 1399, 3138 and 3285 assigned to υ(C=O)as, υ(NH)s, υ(NH)as, and υ(OH) respectively[36].

In [VO(L)(H2O)2], complexe H2L acts as binegative bidentate coordinating via carbonyl oxygen υ(C=O)s. This mode of chelation is based on disappearance of υ(NH) and supported by shift of υ(C=O)s vibration to lower wave length (1543, 1562,1584 and 1563) respectively. Moreover, the IR spectra of all isolated complexes show new bands in the range of (510-539) and (434-452) cm-1 regions which may be attributed to υ(M-O) and υ(M-N)[37], respectively.

Eectronic spectra and magnetic moments

The importance of magnetic and spectra proprieties of the isolated solid complexes very useful to know the complexes configuration. Spectral proprieties were important in changing between ground and excited states of molecules while magnetic proprieties were concerned with energy in ground states.

As electronic spectra may be estimated easily in few minutes in this method, the data rapidly out comes. The differences between electronic spectra and infrared spectroscopy, bands in electronic was broad and not given any information about role group while infrared give details about function groups.

Both magnetic and spectral studies were used to describe the stereochemistry of metal ions in complexes that depend on the position and number d-d transition and could else indicated about extent of distortion present in the complex.

4.C.3.1 ***Electronic spectra and magnetic moment of H2L metal complexes***

The ligand field parameters, electronic spectral bands and magnetic moments of metal complexes collected in table (10-11) and described graphically in figure (12-13).

The electronic spectra of [VO(L)(H2O)2] complex showed a band at 17391 and 17544 cm-1, respectively assigned to 2B2 → 2E(υ2) in a square-pyramidal configuration[38].

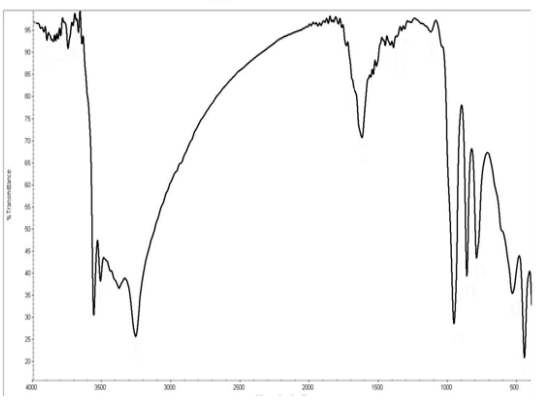


Fig. (10) IR spectrum of H2L ligand.

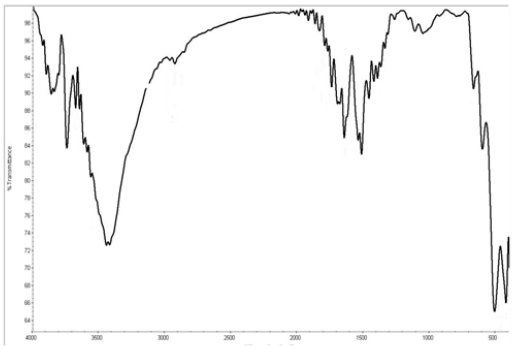


Fig. (11) IR spectrum of VO(L)(H2O)2 complex.

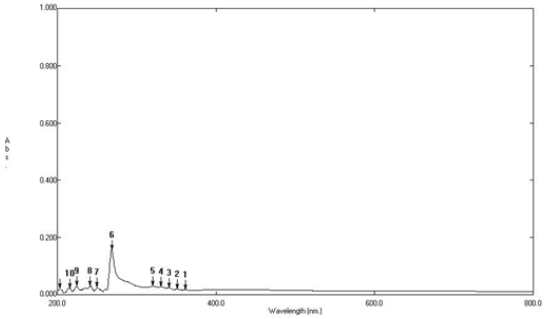


Fig. (12) UV-Vis of H2L ligand

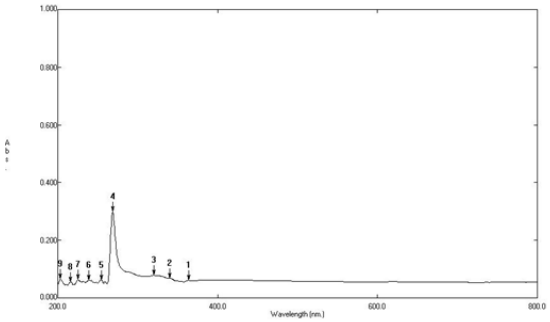


Fig. (13) UV-Vis of [VO(L)(H2O)2] complex.

Conclusion:

Although the complexation reaction is thermodynamically favoured for nano VOSO4 the stability constant for all stiochiometric ratios (L:M) of (1:1) and (1:2) is greater on using VOSO4 than other metals and the biological activity confirmed it. The conductivity measurements show thermodynamic parameters for solvation of bulk and nano- metal salts in absence and presence of amino acids Trans-4-hydroxy proline (H2Prol) in all mixed solvents (0%, 20% and 40% EtOH-H2O) at different temperatures (298.15, 303.15, 308.15 and 313.15 K) by using Fuoss-Shedlovesky method. It was found that the association parameters KA, ΔGA for bulk salts alone in absence of amino acids are increased by increasing temperature due to the increase in the kinetic energy. Also the association thermodynamic parameters for the solvation of nano VOSO4 are decreased by increasing the mole fraction of ethanol in mixed EtOH-H2O solvents.

For association thermodynamic solvation parameters for the interaction of both bulk and nano (VOSO4) in presence and absence of amino acids (Trans-4-hydroxy proline (H2Prol) in mixed EtOH-H2O and at different temperatures.

Two stoichiometric complexes{1:2 & 1:1[M]/[L]} (M/amino acid) are formed in all media and at the four different temperatures.

All complex formation parameters are increased with increase of temperature due to the increase in the kinetic energy.

Also the entropies of complex formation are greater for 1:2 stoichiometric complexes than that of 1:1 stoichiometric complexes.

References

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