Spectrochimica Acta Part A: Molecular and Biomolecular Spectroscopy 107 (2013) 145–150

Contents lists available at SciVerse ScienceDirect

Spectrochimica Acta Part A: Molecular and Biomolecular Spectroscopy

journal homepage: www.elsevier.com/locate/saa

Synthesis, spectral, thermal and thermodynamic studies of oxovanadium(IV) complexes of Schiff bases derived from 3,4-diaminobenzoic acid with salicylaldehyde derivatives

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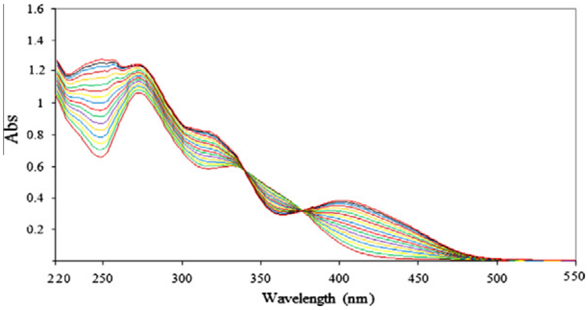
HIGHLIGHTS

- Three new oxovanadium(IV) complexes have been synthesized and characterized.
- From TG curves, the order of thermal stability for the complexes is $VOL^3 > VOL^1 > VOL^2$.
- The trend of formation constants of the complexes are as follow: $VOL^3 > VOL^2 > VOL^1$.

GRAPHICAL ABSTRACT

The synthesis and evaluation of three new oxovanadium(IV) complexes, formed by the interaction of vanadyl acetylacetonate and the Schiff bases, Khosro Mohammadi, Mahmood Niad and Amene Irandoost.



ARTICLE INFO

Article history:
Received 1 September 2012
Received in revised form 28 December 2012
Accepted 11 January 2013
Available online 23 January 2013

Keywords:
Oxovanadium(IV)
Schiff base
Thermogravimetry
Formation constant

ABSTRACT

Synthesis and evaluation of three new oxovanadium(IV) complexes, formed by the interaction of vanadyl acetylacetonate and the Schiff bases: 3,4-bis((E)-2-hydroxybenzylideneamino)benzoic acid (L^1), 3,4-bis((E)-2-hydroxy-3-methoxybenzylideneamino)benzoic acid (L^2) and 3,4-bis((E)-2,4-dihydroxybenzylideneamino)benzoic acid (L^3) in methanol. The complexes have been characterized and studied by IR spectra, UV–Vis spectroscopy and thermogravimetry in order to evaluate their thermal stability and thermal decomposition. According to the results discussed from TG curves, the order of thermal stability for the complexes is $VOL^3 > VOL^1 > VOL^2$. Their formation constants (K_f) were obtained by UV–Vis spectroscopic titration at 15, 25, 35 and 45 °C in methanol by SQUAD software. The trend of formation constants of the complexes as follows: $VOL^3 > VOL^2 > VOL^1$.

Introduction

Schiff bases are the condensation products of primary amines with carbonyl compounds. The common structural feature of these compounds is the azomethine group with a general formula $RHC{=}N{-}R'$ where R and R′ are alkyl, aryl, cyclo alkyl or heterocyclic groups which may be variously substituted. These compounds are also known as anils, imines or azomethines. Several studies [1] showed that the presence of a lone pair of electrons in an sp^2 hybridized orbital of nitrogen atom of the azomethine group is of considerable chemical and biological importance. Because of the relative easiness of preparation, synthetic flexibility, and the special properties of C=N group, Schiff bases are generally excellent chelating agents [2,3] especially when a functional group like —OH or —SH is present close to the azomethine group so as to form a five or six member ring with the metal ion. Versatility of Schiff base ligands

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http://dx.doi.org/10.1016/j.saa.2013.01.035

and biological, analytical and industrial applications of their complexes make further investigations in this area highly desirable.

Transition metal complexes of vanadium with Schiff bases have been amongst the most widely studies of coordination compounds in the past few years, since they are found to be of importance as analytical and antimicrobial agents [4,5]. Considerable efforts have been made to study the behavior and functions of vanadium in biological system [6,7] as well as in catalytic and pharmaceutical applications [8,9].

Vanadium is recognized as an essential trace element for different organism [10]. Vanadium complexes are also of major concern because of their adverse effect on the hydroprocessing catalysts used in the refining crude oil [11,12].

In this study, we have described synthesis, spectroscopic and formation constant measurements of three new oxovanadium(IV) complexes, formed by the interaction of vanadyl acetylacetonate and the Schiff bases: 3,4-bis((E)-2-hydroxybenzylideneamino)benzoic acid (L^1), 3,4-bis((E)-2-hydroxy-3-methoxybenzylideneamino)benzoic acid (L^2) and 3,4-bis((E)-2,4-dihydroxybenzylideneamino) benzoic acid (L^3) which have been characterized by IR, Mass, UV–Vis spectroscopy, thermogravimetry and kinetic studies.

Experimental

Materials and reagents

All chemicals used were of the analytical reagent grade (AR), and of highest purity available. They included vanadyl acetylacetonate, 3,4-diaminobenzoic acid, 2-hydroxybenzylaldehyde, 2-hydroxy-3-methoxybenzylaldehyde and 2,4-dihydroxybenzylaldehyde. All reagents and solvents were commercially obtained from Merck, Aldrich or Fluka. Spectrograde solvents were used for spectral measurements.

Physical measurements

UV–Vis measurements were carried out in Perkin–Elmer Lambda 25 UV–Vis spectrophotometer. The NMR spectra were recorded by a Bruker Avance DPX-500 spectrometer in [D_6]-DMSO and $CDCl_3$ solvent using TMS as an internal standard at 500 MHz. IR spectra were measured from 4000 to 400 cm^{-1} as KBr pellets on a Shimadzu FTIR-8300 spectrophotometer. Mass spectra were obtained with AGILENT 5973 instrument and thermogravimetry were carried out by a PL-1500. The measurements were performed in air atmosphere and the heating rate was held at 10 °C min^{-1}.

Synthesis of vanadyl Schiff base complexes

All of the ligands were prepared by the reaction of 3,4-diaminobenzoic acid and salicylaldehyde derivatives [13,14] (Scheme 1-sup). The complexes were prepared by the reaction of Schiff base ligands with vanadyl acetylacetonate in methanol. To a methanolic solution (50 ml) of vanadyl acetylacetonate (1 mmol, 0.267 gr) was added to the ligand (1 mmol in 30 ml methanol) and the mixture was stirred for 3 h at 50 °C. The resulting precipitate was collected by filtration, washed with H_2O and dried in vacuum (Scheme 2-sup).

3,4-bis((E)-2-hydroxybenzylideneamino)benzoic acid oxovanadium(IV), [VOL^1]

Color: black green; yield: 60%, m.p.: >250 °C. FT-IR (KBr, cm^{-1}): 2400–3700($\nu_{\text{O—H(acid)}}$), 1607($\nu_{C=O}$), 1580($\nu_{C=N}$), 1440, 1465($\nu_{C=C}$), 1200($\nu_{\text{C—O}}$), 975($\nu_{V=O}$), 555($\nu_{\text{C—N—M}}$), 450($\nu_{\text{C—O—M}}$). UV–Vis: (λ_{max}, nm, MeOH): 273, 404. Mass spectra (ESI): *m*/*z*(%) = 425 $[M]^+$, 381, 303, and 99.

3,4-bis((E)-2-hydroxy-3-methoxybenzylideneamino)benzoic acid oxovanadium(IV), [VOL^2]

Color: green; yield: 65%, m.p.: >250 °C. FT-IR (KBr, cm^{-1}): 2400–3700($\nu_{\text{O—H(acid)}}$), 1680($\nu_{\text{C—O}}$), 1600($\nu_{\text{C—N}}$), 1480, 1540($\nu_{\text{C—C}}$), 1250($\nu_{\text{C—O}}$), 978($\nu_{\text{V—O}}$), 550($\nu_{\text{C—N—M}}$), 450($\nu_{\text{C—O—M}}$). UV–Vis: (λ_{max}, nm, MeOH): 305, 374. Mass spectra (ESI): *m*/*z*(%) = 485 $[M]^+$, 425, 381, 303, and 99.

3,4-bis((E)-2,4-dihydroxybenzylideneamino)benzoic acid)oxovanadium(IV), [VOL^3]

Color: green; yield: 60%, m.p.: >250 °C. FT-IR (KBr, cm^{-1}): 2400–3700($\nu_{\text{O—H(acid)}}$), 1610($\nu_{\text{C—O}}$), 1600($\nu_{\text{C—N}}$), 1460, 1490($\nu_{\text{C—C}}$), 1250($\nu_{\text{C—O}}$), 990 ($\nu_{\text{V—O}}$), 530($\nu_{\text{C—N—M}}$), 460($\nu_{\text{C—O—M}}$). UV–Vis: (λ_{max}, nm, MeOH): 262, 345. Mass spectra (ESI): *m*/*z*(%) = 457 $[M]^+$, 413, 335, and 99.

Results and discussion

IR spectra

In the FT-IR spectral data of complexes, a band at 2400–3700 cm^{-1} related to O—H(acid) group. The strong bands around 1600 cm^{-1} is assigned to azomethine (C=N) groups [15]. The ring skeletal vibrations (C=C) were in the region of 1400–1600 cm^{-1} [16], and the bands at 1220–1250 cm^{-1} range can be related to the phenolic (C—O) group vibrations [15]. IR spectra of vanadyl Schiff base complexes show two kinds of V=O stretching bands around (960–990 cm^{-1}) for the monomeric forms and (850–880 cm^{-1}) for the polymeric forms [17,18]. In this study, the strong bands at 970–990 cm^{-1} can be related to the (V=O) group [19]. Weak bands in the 530–555 cm^{-1} and 450–460 cm^{-1} ranges can be attributed to $\nu_{\text{C—N—M}}$ and $\nu_{\text{C—O—M}}$, respectively [20]. IR spectrum of [VOL^1] is shown in Fig. 1.

Mass spectra

The mass spectrum of [VOL^1] gives isotropic peak of *m*/*z* at 425 [M^+] which is corresponding to the composition [VOL^1] (molecular mass = 425) (see Fig. 1-sup). Moreover, another observed peaks are in 381($C_{20}H_{14}N_2VO_3$), 303($C_{14}H_8VO_3$), and 99(VO_3). The mass spectrum of [VOL^2] gives isotropic peak of *m*/*z* at 485 [M^+] which is corresponding to the composition [VOL^2] (molecular mass = 485). Moreover, another observed peaks are in 425($C_{21}H_{14}N_2VO_5$), 381($C_{20}H_{14}N_2VO_3$), 303($C_{14}H_8VO_3$), and 99(VO_3). The mass spectrum of [VOL^3] gives isotropic peak of *m*/*z* at 457 [M^+] which is corresponding to the composition [VOL^3] (molecular mass = 457). Moreover, another observed peaks are in 413($C_{20}H_{14}N_2VO_5$), 335($C_{14}H_8N_2VO_5$), and 99(VO_3).

The electronic spectra

Electronic absorption spectral data of the complexes are very similar to each other because of their structural identity. The strong or a shoulder absorption band observed below 300 nm is assigned to intraligand $\pi \rightarrow \pi^*$ transitions of the phenolic chromophores. The absorption band observed in all ligand spectra within the range of 260–340 nm is most probably due to the transition of $n \rightarrow \pi^*$ of imine group [21].

Charge-transfer bands are expected for vanadium complexes, which can be seen in the region of 345–405 nm [22].

Thermal gravimetry

The thermal decomposition of the studied complexes presented pathways. All TG and DTA figures of the compounds had the same

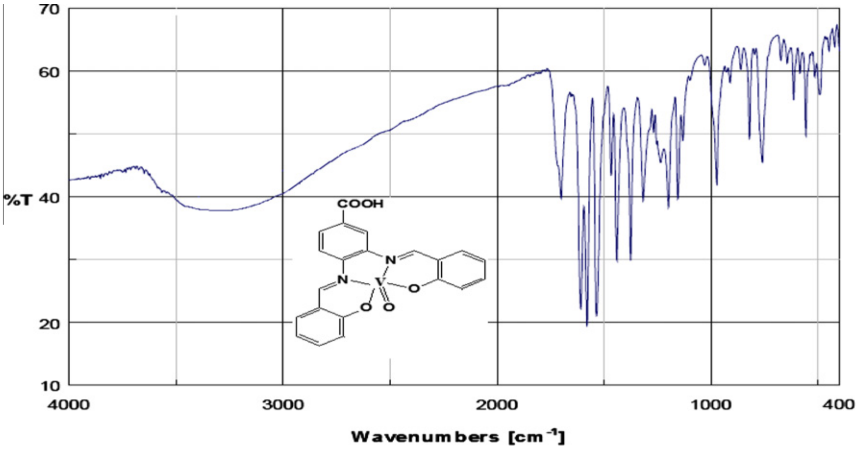

Fig. 1. IR spectrum of [VOL^1].

trend. All the complexes were decomposed in two or three steps [23,24]. The TG and DTA thermograms of [VOL^1] complex are shown in Fig. 2. At 700 °C, the stable metal oxide V_2O_5 was formed [25].

In the thermogram of [VOL^1], two steps were observed, the first mass loss occurs between 310 and 340 °C that corresponding to the decomposition of 17% (calculated: 18.4%) in TG. The second mass loss occurs between 390 and 420 °C that corresponding to the decomposition of 57% (calculated: 60.2%). At 650–700 °C, the stable metal oxide, V_2O_5 was formed.

$$C_{21}H_{14}N_2O_5V \xrightarrow[-C_6H_6]{310-340\ ^\circ C} C_{15}H_8N_2O_5V \xrightarrow[-C_{15}H_8N_2O_{5/2}]{390-420\ ^\circ C} V_2O_5$$

In the thermogram of [VOL^2], the lattice water molecule decomposed between 90 and 110 °C [19,26]. Then three consecutive steps were observed during the thermal decomposition. The first mass loss (TG = 10%, calculated: 9.1%) occurs between 150 and 200 °C. The second mass loss (TG = 15%, calculated: 15.6%) occurs between 270 and 300 °C. The last mass loss (TG = 53%, calculated: 56.5%) occurs between 390 and 450 °C with formation V_2O_5 (TG = 22%, calculated: 18.8%).

$$C_{23}H_{18}N_2O_7V \xrightarrow[-CO_2]{150-200\ ^\circ C} C_{22}H_{18}N_2O_5V \xrightarrow[-C_6H_4]{270-300\ ^\circ C} C_{16}H_{14}N_2O_5V$$

$$C_{16}H_{14}N_2O_5V \xrightarrow[-C_{16}H_{14}N_2O_{5/2}]{330-350\ ^\circ C} V_2O_5$$

In the thermogram of [VOL^3], the lattice water molecule decomposed between 90 and 110 °C. The first mass loss occurs between 330 and 350 °C that corresponding to the decomposition of 25% (calculated: 26.3%) and the second step occurs between 390 and 430 °C that corresponding to the decomposition of 54% (calculated: 53.8%) in TG and finally the stable metal oxide V_2O_5 was formed.

$$C_{21}H_{14}N_2O_7V \xrightarrow[-C_7H_4O_2]{330-350\ ^\circ C} C_{14}H_{10}N_2O_5V \xrightarrow[-C_{14}H_{10}N_2O_{5/2}]{390-430\ ^\circ C} V_2O_5$$

Kinetic studies

Broido has suggested a simple and sensitive graphical method for the treatment of TGA data [27]. According to this method, the weight at any time, $t(W_t)$ is related to the fraction of initial molecules not yet decomposed (y) by the equation:

$$y = \frac{N}{N_0} = \frac{(W_t - W_\infty)}{(W_0 - W_\infty)} \tag{1}$$

where W_0 is the initial weight of the materials and W_∞ is the weight of residue at the end of decomposition.

If the pyrolysis is carried out isothermally, the rate of the reaction is given by:

$$\frac{dy}{dt} = ky^n \tag{2}$$

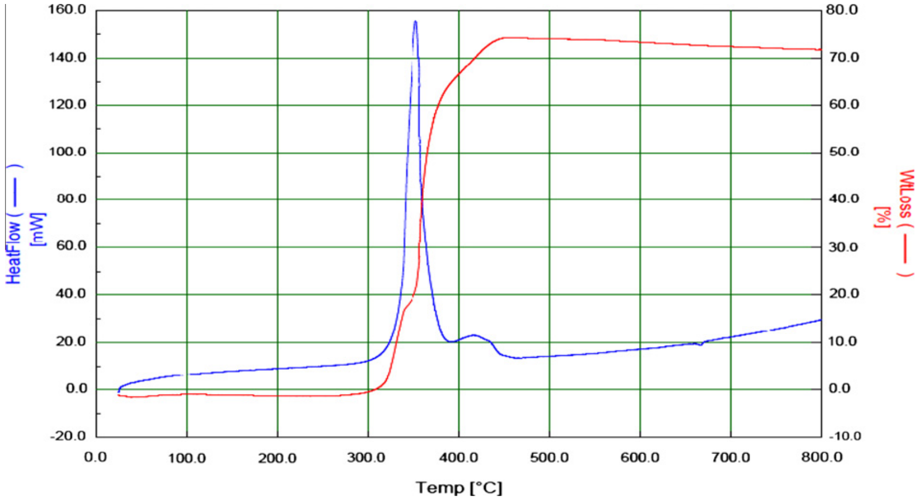

Fig. 2. The TG and DTA thermograms of VOL^1 complex.

where n is the reaction order. The rate constant, k, changes with absolute temperature according to the equation of Arrhenius.

$$k = A \cdot e^{-E/RT} \tag{3}$$

and if T is linear function of time, t, therefore:

$$T = T_0 + u \cdot t \tag{4}$$

u is the heating rate. The first derivative of this equation is:

$$dT = u \cdot dt \tag{5}$$

This rearranges to:

$$\frac{dy}{y^n} = -\left(\frac{A}{u}\right) \cdot e^{-\frac{E}{RT}} dt \tag{6}$$

The thermogravimetric analysis curve for this reaction represents the last equation integrated from a temperature T_0, where $y = 1$, to a temperature for another value of y.

$$\int_y^1 \frac{dy}{y^n} = -\frac{A}{u}\int_{T_0}^{T} e^{-\frac{E}{RT}} dT = \frac{A}{u}\int_{T}^{T_0} e^{-\frac{E}{RT}} dT \tag{7}$$

The main consideration of this method is that the reaction is of the first order. With this supposition, the left side of the reaction can be resolved.

$$\int_y^1 \frac{dy}{y^n} = \int_y^1 \frac{dy}{y} = -\ln y = \ln\left(\frac{1}{y}\right) \tag{8}$$

There are various methods for resolving the right side of the equation. Broido's method is based on approximations done by other authors. We have utilized the approximations introduced by Horowitz and Metsger [28].

$$e^{-\frac{E}{RT}} \approx \left(\frac{T_m}{T}\right)^2 e^{-\frac{E}{RT_m}} \tag{9}$$

T_m is the temperature at which the maximum reaction rate occurs. Introducing this approximation has

$$\ln\frac{1}{y} = \frac{A}{u}\int\left(\frac{T_m}{T}\right)^2 e^{-\frac{E}{RT}} dT \tag{10}$$

changing variable has

$$x = \frac{1}{T} \Rightarrow dx = \frac{1}{T^2} dT \Rightarrow dT = -\left(\frac{1}{x^2}\right) dx \tag{11}$$

$$\ln\frac{1}{y} = \frac{A}{u} T_m^2 \int x^2 e^{-\frac{E}{R}x}\left(-\frac{1}{x^2}\right) dx \tag{12}$$

$$\ln\frac{1}{y} = \frac{A \cdot R \cdot T_m^2}{E \cdot u} \cdot e^{-\frac{E}{R}X} \tag{13}$$

$$\ln\frac{1}{y} = \frac{A \cdot R \cdot T_m^2}{E \cdot u} \cdot e^{-\frac{E}{R}\left(\frac{1}{T}\right)} \tag{14}$$

This equation can be represented by:

$$\ln\left(\ln\frac{1}{y}\right) = -\frac{E}{R}\left(\frac{1}{T}\right) + \text{const.} \tag{15}$$

Eq. (15) is a straight line. The gradient of the graph $\ln[\ln(1/y)]$ *vs.* $(1/T)$ is the activation energy $E_a^{\#}$ and the intercept is frequency factor, A.

$$-E_a^{\#} = \text{slope} \times 2.303 \times R \tag{16}$$

where $E_a^{\#}$ is activation energy and R is the gas constant [29]. Application of this method used to determine the kinetic parameters for complexes.

The entropy of activation $\Delta S^{\#}$ was calculated using the equation:

$$A = \frac{kT_m}{h} e^{\frac{\Delta S^{\#}}{R}} \tag{17}$$

where k, h and T_m are the Boltzmann's constant, the Planck's constant and the peak temperature, respectively.

Fig. 3 shows Broido plots of [VOL^1] complex.

The enthalpy and free energy of activation were calculated using equations:

$$\Delta H^{\#} = E_a^{\#} - RT \tag{18}$$

$$\Delta G^{\#} = \Delta H^{\#} - T\Delta S^{\#} \tag{19}$$

The results were reported in Table 1.

The positive values of $\Delta H^{\#}$ mean that the decomposition processes are endothermic and high values of the activation energies reflect the thermal stability of the complexes.

Thermodynamic interpretation

The thermodynamic parameters are useful tools for studying the interactions between the donor and the acceptor species. The first step in this study is the determination of formation constants

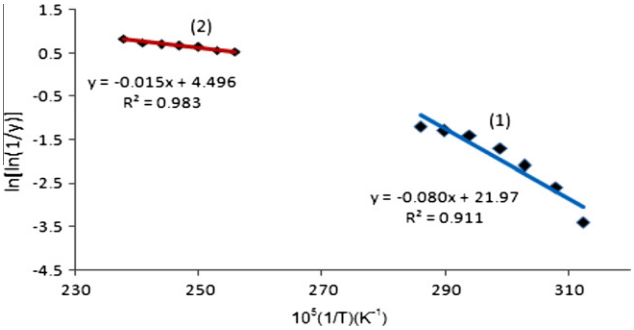

Fig. 3. Broido plots of [VOL^1] complex.

Table 1
Thermal study of oxovanadium(IV) complexes.

Compounds	Step	ΔT (°C)	$E_a^{\#}$ (kJ mol^{-1})	$\Delta H^{\#}$ (kJ mol^{-1})	$\Delta S^{\#}$(JK^{-1} mol^{-1})	$\Delta G^{\#}$ (kJ mol^{-1})
VOL^1	I	310–340	497.8	492.6	60.6	454.8
	II	390–420	80.4	74.6	−241.8	242.2
VOL^2	I	150–200	17.2	13.3	−300.9	155.6
	II	270–300	42.1	37.3	−277.7	196.4
	III	390–450	76.6	70.6	−256.9	256.3
VOL^3	I	330–350	605.1	599.1	125.5	520.9
	II	390–430	176.2	170.4	−183.9	299.7

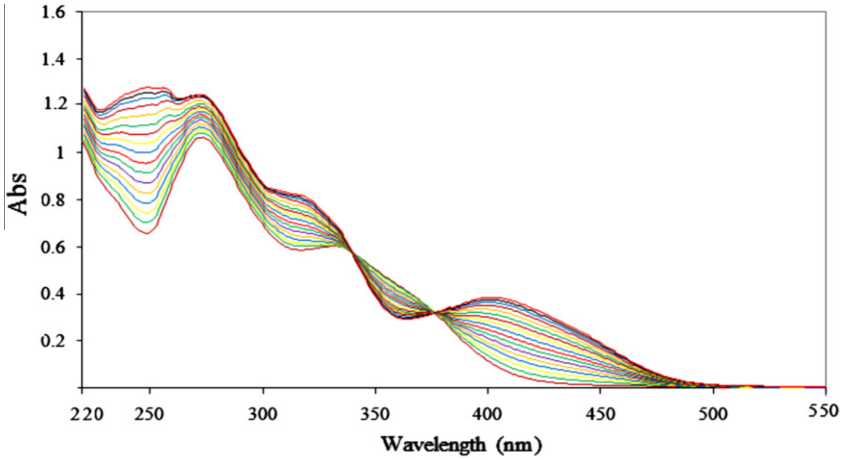

Fig. 4. The variation of the electronic spectra of L^1 titrated with various concentrations of VO^{2+} in constant ionic strength (I = 0.10 M $NaClO_4$) and at 25 °C in MeOH.

Table 2
The log K_f for vanadyl ion with the tetradentate Schiff base ligands in MeOH at 15, 25, 35 and 45 °C at I = 0.10 M $NaClO_4$.

Compounds	Log K_f			
	15 °C	25 °C	35 °C	45 °C
L^1(H)	2.90 (±0.05)	3.61 (±0.09)	3.79 (±0.01)	4.08 (±0.01)
L^2(OMe)	3.34 (±0.02)	4.00 (±0.09)	4.12 (±0.03)	4.65 (±0.03)
L^3(OH)	3.96 (±0.01)	4.29 (±0.03)	4.37 (±0.04)	5.22 (±0.03)

Table 3
The thermodynamic parameter values, Δ*H*°, Δ*S*° and Δ*G*°, for the complex formation in MeOH.

Compounds	$\Delta H°$ (kJ mol^{-1})	$\Delta S°$ (JK^{-1} mol^{-1})	$\Delta G°$ (kJ mol^{-1})[a]
L^1	−28.1	122.8	−2.6
L^2	−30.6	134.6	−2.9
L^3	−29.2	133.8	−3.4

[a] $\Delta G° = -RT\ln K$ and T = 298 K.

in various temperatures, which make help a better understanding of relative stability of adducts.

The formation constants measurements were carried out by spectrophotometric titration at constant ionic strength 0.1 M ($NaClO_4$) at 15, 25, 35 and 45 °C (±0.1 °C). In a typical measurement, 2.5 ml solution of the ligands (5×10^{-5} M) in MeOH was titrated with various concentrations of the vanadyl acetylacetonate (6×10^{-3}–18×10^{-2}) in MeOH. UV–Vis spectra were recorded in the range 200–700 nm about 5 min after each addition. The formed product showed different absorption from the free ligand, while the metal ion solution shows no absorption at those wavelengths. As an example, the variation of the electronic spectra for L^1 titrated with various concentrations of VO^{2+} at 25 °C in MeOH is shown in Fig. 4. The same changes are valid for other systems.

$$VO^{2+} + H_2L^X \leftrightarrows VOL^X + 2H^+ \quad K_f \tag{20}$$

Depending on the method used for solution treatment (potentiometric, spectrophotometric, calorimetric, and so on) there are different computer programs for determining the formation constant. For determination of formation constant, we chose SQUAD program [30]. SQUAD program is designed to calculate the best values for the formation constants of the proposed equilibrium model by employing a nonlinear, least-squares approach. Other thermodynamic parameters were calculated by using the well-known van't Hoff equation. The equilibrium constants and the thermodynamic parameters data are collected in Tables 2 and 3.

The trends in the formation constants of the given metal with these Schiff base ligands are as follow:

$$L^3(OH) > L^2(OMe) > L^1(H)$$

which is according to electron-donor properties of functional groups of the Schiff base ligands [13].

This is in contrast to the electronic spectra that the charge donation from VO^{2+} ion to Schiff base's π^* molecular orbital is increased by electron-withdrawing groups on the Schiff base ligands [31]. OH in L^3 is a richer donor than OMe and H. Therefore, the ligand with the H group has the smallest formation constant while the ligand with OH has the highest [32]. Also, the formation constants are increased by increasing the temperature:

$$45\,°C > 35\,°C > 25\,°C > 15\,°C$$

Conclusions

In this study, the oxovanadium(IV) complexes have been synthesized and characterized by several spectroscopic methods. These complexes form a green monomeric structure with square-pyramidal coordination geometry around the metal center. According to the results discussed from TG curves, the order of thermal stability for the complexes is $VOL^3 > VOL^1 > VOL^2$. The number of decomposition steps of the ligands depends on the substituent group leading to differences in thermal process for the complexes. By considering the results of thermodynamic study, the formation constants (of the complexes for the given metal with different ligands) which is according to the donor properties of the substituent:

$$L^3(OH) > L^2(OMe) > L^1(H)$$

Acknowledgement

The authors thank Persian Gulf University of Bushehr Research Councils for financial support of this work.

Appendix A. Supplementary material

Supplementary data associated with this article can be found, in the online version, at http://dx.doi.org/10.1016/j.saa.2013.01.035.

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