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Structures of sodium vanadate solution under different conditions

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Abstract Combined with Fourier transform infrared spectroscopy (FTIR) analysis of sodium vanadate solution, the relationship between conductivity and structure was investigated by measuring the electric conductivity of the solution under different alkali concentrations and molar ratios of NaOH to V_2O_5. Results suggest that the polymerization vanadium acid radical ions gradually transform into monomer with the solution diluting. When the solution is diluted to a certain extent, only the vanadium acid radical ion with V–OH chemical bond exists in the solution. At NaOH concentration of below 105.21 $g\cdot L^{-1}$, the vanadate anions mainly exist in the form of vanadium acid radical ion with V–OH chemical bond and the ion transference number is approximately from 0.58 to 0.82. In the medium NaOH concentration range of 105.21–117.03 $g\cdot L^{-1}$, the vanadate anions mostly exist in the form of vanadium acid radical ion with V–OH and V–O–V chemical bonds and the ion transference number is approximately 3.29. At NaOH concentration of above 117.03 $g\cdot L^{-1}$, vanadate anions exist in the form of vanadium acid radical ion with V–OH and V–O–V chemical bonds.

Keywords Sodium vanadate solution; Electric conductivity; Migration number; Infrared spectroscopy

1 Introduction

Vanadium is an important strategical metal and is widely used in national defense, energy, metallurgical industry, etc. Many researches on vanadium extraction were carried out and many achievements were obtained. Vanadium residue is the main source of vanadium resource [1–4], and there are some ways to recover vanadium, such as oxidizing roasting–alkaline leaching [6], acid leaching [5, 7], sodium roasting–water leaching [8], calcium roasting–production leaching [8, 9], fluidized bed acid leaching [10], sodium roasting–acid leaching–solvent extraction [11]. However, except alkaline leaching, these techniques have considerable disadvantages, including special requirements for expensive corrosion-resistant equipment and high energy requirements. Alkaline leaching is widely used in iron and steel enterprise, especially in carbon enterprise. Yet alkaline leaching rate of vanadium is lower than 75 %.

Vanadium mainly exists in the form of vanadium oxide in nature. Only V_2O_5 in all the vanadium oxide is dissolved in alkali solution. In alkali solutions, the form of vanadate mainly exists in the following complexes: VO_4^{3-}, VO_3^-, HVO_4^{2-}, $V_3O_9^{3-}$, $V_4O_{12}^{4-}$, $V_{10}O_{28}^{6-}$ and $V_2O_7^{4-}$ [12], which depends on the vanadium concentrations, the pH value of solutions and other leaching conditions.

The structure of sodium vanadate solution is an important basic problem in alkali leaching process. The form of vanadate is closely related to the leaching rate. However, there are few relevant literatures that clarify the evolution of vanadium structure in the leaching. This brings great confusion to improve vanadium leaching rate. There are mainly two types in the structure of vanadate: one is conjecturing the ion structure of the solutions which depends on its chemical and physical properties; the other one is judging ion forms of vanadate combined with Fourier transform infrared (FTIR) and theoretic calculations. In this paper, the electric conductivity and FTIR were investigated, and the ion transference number of ions was also

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calculated in order to explain the evolution of vanadate structure under different leaching conditions.

2 Experimental

Vanadium oxide reacts with sodium hydroxide and forms sodium vanadate solution. The conductivity was tested at different NaOH concentrations and different ratios of Na to vanadium in sodium vanadate solution, and then the migration number of ions was calculated [13]. And the infrared spectra in the same condition were also tested. The structure transformation of sodium vanadate solution under different conditions was figured out.

DDSJ-308A conductivity meter was used to measure the electrical conductivity of sodium vanadate solution. FTIR analysis was carried out using FTIR spectrometer spectrum RX1 (Perkin-Elmer) in the wavenumber region of 4000–400 cm^{-1}. The resolution of the spectra was 4 cm^{-1} at room temperature. FTIR data were recorded in the transmittance mode [14].

3 Results and discussion

3.1 Electrical conductivity studies

Electrical conductivity is one of the most important physical and chemical properties in sodium metavanadate solution, and it closely contacts with the ionic structures and compositions in vanadium ion solutions.

3.1.1 Influences of Na concentration on conductivity of sodium metavanadate solutions

Influences of Na concentration on the conductivity (η) of sodium metavanadate solutions were studied. Three solutions with different mass ratios of vanadium to sodium ($m_t = m(\mathrm{Na})/m(\mathrm{V})$) were diluted gradually. The solution conductivity under different concentrations was measured, and the results are shown in Fig. 1.

From Fig. 1, it can be easily found that when m_t value is certain, the electrical conductivity increases with V concentration increasing, and a maximum η value between 100 and 120 $mol \cdot L^{-1}$ is reached and then η value decreases. The probable reason is that with the concentration increasing, more and more conducting particles are generated. When ion concentration reaches a certain value, there is an obvious increase in the interactions between ions, which has a significant obstruction to the ion transference rate [15]. Thereby, the electrical conductivity increases slowly with the increase in V concentration [15].

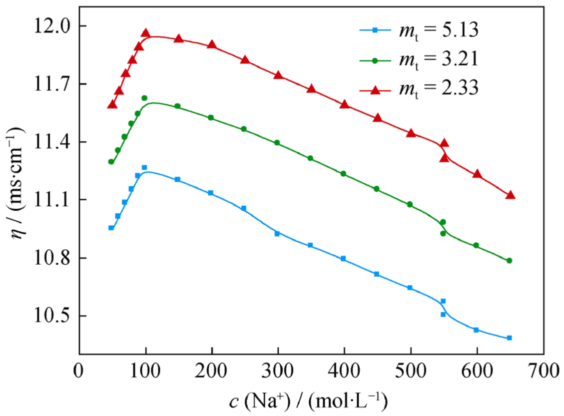

Fig. 1 Effect of Na concentration on conductivity (η) of sodium vanadate solution

3.1.2 Influences of m_t on conductivity of sodium metavanadate solutions

In sodium metavanadate solution, different m_t values significantly affect ion structure and its physicochemical property. Influences of m_t values on conductivity were investigated. Results are presented in Fig. 2.

Figure 2 shows the changing trend of conductivity at different m_t values. When sodium hydroxide concentration is 100 $g \cdot L^{-1}$, the conductivity increases with an increase in m_t value. In all ions, the ionic structure and the concentration of free base can easily influence the conductivity of sodium metavanadate solutions. The conductivities of H^- and OH^- are greater than those of other ions. The concentration of V_2O_5 decreases with the increase in m_t value. If the sodium hydroxide concentration is fixed, the concentration of free hydroxyl ions increases with a decrease in the amount of V_2O_5; so the conductivity of the solution increases.

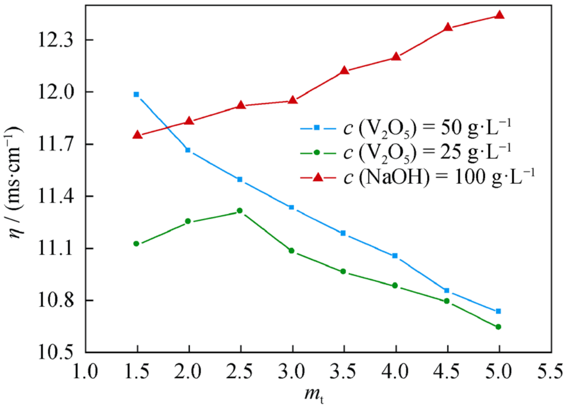

Fig. 2 Effect of ratio of Na to V on sodium vanadate solution conductivity

When vanadic oxide concentration is 50 g·L^{-1}, the sodium hydroxide concentration increases with the increase in m_t value, and the solution viscosity increases; so the ion transference rate and thus electrical conductivity decrease. When vanadic oxide concentration is 25 g·L^{-1}, sodium hydroxide concentration increases with m_t value increasing, and electrical conductivity increases. If sodium hydroxide concentration increases to some extent, the solution viscosity increases and the electrical conductivity decreases. So at m_t value of below 2.5, the increase in m_t value is found to have a significant effect on the conductivity of solution. However, the conductivity decreases at m_t value of above 2.5.

3.2 Number of ion transference in sodium metavanadate solution

The ion transference rate is not only related to the nature of ions (including ionic radius, ion hydration, charge number, etc.) and the solvent, but also related to the electric potential gradient—d*E*/d*l*. The ion transference rate can be written as [16]:

$$r_+ = u_+ \cdot \frac{\mathrm{d}E}{\mathrm{d}l} \tag{1}$$

$$r_- = u_- \cdot \frac{\mathrm{d}E}{\mathrm{d}l} \tag{2}$$

where *r* is ionic mobility (m^2·S^{-1}·V^{-1}), *u* can be equivalent to the ion transference rate under unit potential gradient (1 V·m^{-1}), *E* is potential, *l* is distance, and subscripts + and − represent positive and negative ions, respectively. The magnitude of ionic mobility is related to temperature, concentration and other factors. The value can be measured by moving interface test.

Because of the difference in transference rate of positive and negative ions with the electric charge, the share is also different when they transfer electric charge [17]. This is called transference number, which is expressed by the equation as below [18].

$$t_\mathrm{B} = \frac{I_\mathrm{B}}{I} \tag{3}$$

where t_B is the score of transfer electric charge of B ions, I_B is current that is carried by B ions, and *I* is total current. In another way, $\sum t_\mathrm{B} = 1$, which can be expressed as [17]:

$$\sum t = \sum t_+ + \sum t_- \tag{4}$$

where *t* is the total score of transfer electric charge, t_+ is the score of transfer electric charge of positive ion, and t_- is the score of transfer electric charge of negative ion. Molar conductivity of total ions is the sum of the molar conductivity of positive and negative ions. The ion transference number can be seen as a fraction of molar conductivity of some irons occupying the molar conductivity of electrolyte. For the strong electrolyte solution, when the concentration is low, it can be concluded by the equations as below [19]:

$$\varLambda_m^\infty = \varLambda_{m,+}^\infty + \varLambda_{m,-}^\infty \tag{5}$$

$$t_+ = \varLambda_{\mathrm{m},+} \cdot \varLambda_m \tag{6}$$

$$t_- = \varLambda_{\mathrm{m},-} \cdot \varLambda_\mathrm{m} \tag{7}$$

where $\varLambda_\mathrm{m}^\infty$ is limit molar conductivity of total ions, $\varLambda_{\mathrm{m},+}^\infty$ and $\varLambda_{\mathrm{m},-}^\infty$ are limit molar conductivities of positive ions and negative ions, respectively; $\varLambda_{\mathrm{m},+}$ and $\varLambda_{\mathrm{m},-}$ are molar conductivities of positive ions and negative ions, respectively; and $\varLambda_\mathrm{m}$ is molar conductivity of total ions. t_+, t_- and $\varLambda_\mathrm{m}$ can be measured by experiment, and then the molar conductivity of sodium vanadate solution at 25 °C under different concentrations was calculated. Results are presented in Fig. 3.

Figure 3 shows that the molar conductivity ($\varLambda_\mathrm{m}$) of sodium vanadate solution linearly decreases with the increase in $c^{1/3}$(Na). Turning point appears at $c^{1/3}$(Na) of 1.66 (mol·L^{-1})$^{1/3}$ (the equivalent of sodium ion concentration of 105.21 g·L^{-1}) and 1.72 (mol·L^{-1})$^{1/3}$ (the equivalent of sodium ion concentration of 117.03 g·L^{-1}). Ion transference numbers at low, middle and high concentrations were calculated, respectively. The following formulae were used [19]:

$$t(\mathrm{Na}^+ + \mathrm{OH}^-) = \left[f(\mathrm{Na}^+) \cdot \varLambda_\mathrm{m}^\infty(\mathrm{Na}^+) + f(\mathrm{OH}^-) \cdot \varLambda_\mathrm{m}^\infty(\mathrm{OH}^-)\right] / \varLambda_\mathrm{m}^\infty(\mathrm{aq}) \tag{8}$$

$$t(\mathrm{V}) + t(\mathrm{Na}^+) + t(\mathrm{OH}^-) = 1 \tag{9}$$

where $f(\mathrm{Na}^+)$ and $f(\mathrm{OH}^-)$ are molar fractions of Na$^+$ and OH$^-$ under different NaOH concentrations, respectively. Values of $\varLambda_\mathrm{m}^\infty(\mathrm{Na}^+)$ and $\varLambda_m^\infty(\mathrm{OH}^+)$ can refer to Ref [15]. Calculation results are shown in Table 1.

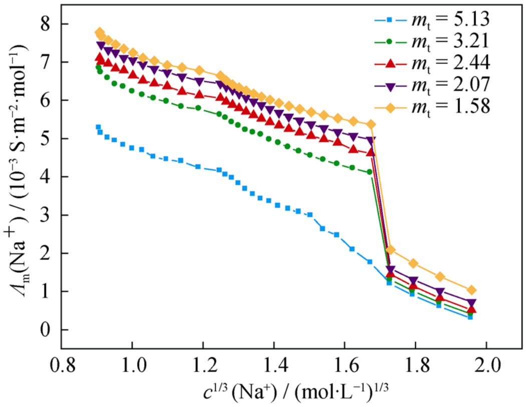

Fig. 3 Effect of Na$^+$ concentration on molar electric conductivity of vanadate solution with different m_t at 25 °C

Table 1 Molar electric conductivity and transference number of vanadate ions in vanadate solution

m_t	$c^{1/3}(\text{Na}^+)/(\text{mol}\cdot\text{L}^{-1})^{1/3}$	$c(\text{NaOH})/(\text{g}\cdot\text{L}^{-1})$	$\Lambda_m^\infty(\text{aq})/(\text{S}\cdot\text{m}^{-2}\cdot\text{mol}^{-1})$	$[t(\text{Na}^+) + t(\text{OH}^-)]/t(\text{V})$
1.58	0.91–1.66	<105.21	0.1340	0.71
	1.67–1.72	105.21–117.03	0.0054	3.53
2.07	0.92–1.65	<105.21	0.1880	0.63
	1.65–1.72	105.21–117.03	0.0068	3.72
2.33	0.91–1.68	<105.21	0.2150	0.58
	1.68–1.74	105.21–117.03	0.0083	4.15
3.21	0.90–1.69	<105.21	0.2430	0.74
	1.69–1.75	105.21–117.03	0.0102	3.83
5.13	0.88–1.66	<105.21	0.3020	0.82
	1.66–1.73	105.21–117.03	0.0114	3.29

When m_t values change from 5.13 to 1.58 and $c(\text{NaOH})$ is lower than 105.21 g·L^{-1}, the value of $[t(\text{Na}^+) + t(\text{OH}^-)]/t(\text{V})$ changes from 0.58 to 0.82, which means that the transference number of vanadium acid radical ion is greatly independent of m_t values. When $c(\text{NaOH})$ increases from 105.21 to 117.03 g·L^{-1}, the value of $[t(\text{Na}^+) + t(\text{OH}^-)]/t(\text{V})$ increases firstly and then decreases with the increase in m_t value. The reason could be that the change in vanadium acid radical ion form is ignored in the calculating process of the transference number with the concentration increasing. With $c(\text{NaOH})$ increasing, the vanadium acid radical ion monomer is aggregated to form the vanadium dimer acid radical ion and the high degree of vanadium polymer acid radical ion because of thermal motion. The polymer ion dimension is bigger. The structure of polymerization ion might be 4-coordinate complexes, 5-coordinate complexes and 6-coordinate complexes. The ability of its transference decreases with functional groups increasing. Then the ion transference number decreases while the conductivity decreases.

3.3 Infrared spectrum of sodium vanadate solution

3.3.1 *Influence of concentration on structure of sodium vanadate solution*

Different sodium vanadate solutions have different ion structures. It can be studied by the change of infrared spectral in the diluting process of sodium solution. Concentration changes in the process of dilution at $m_t = 5.13$, 3.21 and 2.33 are shown in Tables 2, 3 and 4, respectively. The infrared spectra in the process of dilution are presented in Fig. 4.

As shown in Fig. 4, there are four obvious peaks in the infrared spectra and their wavenumbers are 2200, 1650, 1400 and 800 cm^{-1}, respectively. The vanadate ions in the solution mainly exist the following rules:

$$[\text{VO}_4]^{3-} \rightarrow [\text{VO}_3 \cdot \text{OH}]^{2-} \rightarrow [\text{V}_2\text{O}_6 \cdot \text{OH}]^{3-} \quad (10)$$

And the infrared absorption peak of OH$^-$ ions mainly exist in the wavenumber range of 2500–3300cm^{-1}, according to the number of metals combined with OH$^-$ ions, the absorption peak changes. It can be rationally judged that the peak at 800 cm^{-1} is the vibrating belt of V–O–V. The peaks at 1650 and 1400 cm^{-1} are the symmetric stretching vibrating band and the antisymmetric stretching vibrating band of V–OH, respectively. And at the peak number of 2200cm^{-1}, the vanadate ion structures are types of V–O. From above can be known that, there are three ions (including Na$^+$, OH$^-$, vanadium acid radical ion with

Table 2 Concentration of vanadate solution at $m_t = 5.13$ with five measurements

Nos.	1	2	3	4	5
$c(\text{NaOH})/(\text{g}\cdot\text{L}^{-1})$	250	125	62.5	31.25	15.625
$c(\text{V}_2\text{O}_5)/(\text{g}\cdot\text{L}^{-1})$	50	25	12.5	6.25	3.125

Table 3 Concentration of vanadate solution at $m_t = 3.21$ with four measurements

Nos.	1	2	3	4
$c(\text{NaOH})/(\text{g}\cdot\text{L}^{-1})$	250	125	62.5	31.25
$c(\text{V}_2\text{O}_5)/(\text{g}\cdot\text{L}^{-1})$	80	40	20.0	10.00

Table 4 Concentration of vanadate solution at $m_t = 2.33$ with four measurements

Nos.	1	2	3	4
$c(\text{NaOH})/(\text{g}\cdot\text{L}^{-1})$	250	125	62.5	31.25
$c(\text{V}_2\text{O}_5)/(\text{g}\cdot\text{L}^{-1})$	110	55	27.5	13.75

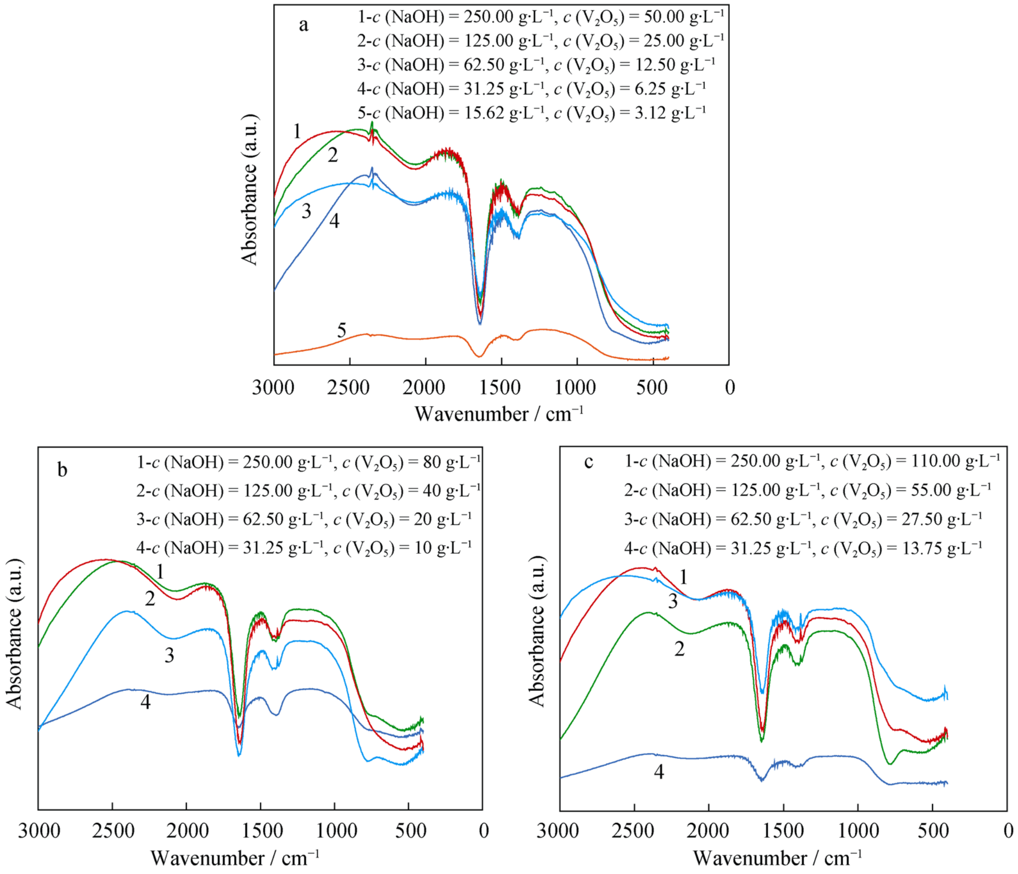

Fig. 4 FTIR absorption spectra of vanadate solution at different concentrations: **a** $m_t = 5.13$, **b** $m_t = 3.21$, and **c** $m_t = 2.33$

V–OH, V–O and V–O–V chemical bonds) and their ion pairs exsiting in the sodium vanadate solution:

With the decrease in concentration of sodium vanadate solution, the characteristic peak intensity corresponding to the bending vibration of V–O at 2200 cm^{-1} in the infrared spectrum is gradually weakened at m_t values of 5.13, 3.21 and 2.33. It indicates that the ion corresponding to the peak is gradually reduced in the diluting process of sodium vanadate solution. The peak strength at the wavenumber of 1650 cm^{-1} increases with the decrease in concentration. The half-width decreases, and the peak symmetry increases. This means the increase in content of vanadium acid radical ion with V–OH. The peak strength at the wavenumber of 800 cm^{-1} changes little in the infrared spectrum of sodium vanadate solution at m_t values of 2.33 and 3.21. It means that the vanadium acid radical ion concentration corresponding to the vibration of V–O–V changes little in these conditions. The infrared spectrum peak intensity at 800 cm^{-1} becomes weakened gradually at m_t value of 5.13. This shows that the concentration of vanadium acid radical ion corresponding to the vibration of V–O–V is kept at a high level under high concentrations of sodium vanadate solution. With the reduction in the concentration of sodium vanadate solution, the concentration of vanadium acid radical ion with V–O–V chemical bond decreases gradually. The vanadium dimer acid radical ion may be converted to V–OH.

In addition, with the solution diluting, the absorption peak of the wavenumber at 1650 cm^{-1} is deviated to high wavenumber gradually. Thus it can be seen that the same group of characteristic absorption frequency is not fixed. This is because the chemical bond vibration frequencies are not only related to nature of ions but also affected by external factors.

3.3.2 *Influence of m_t on structure of sodium vanadate solution*

In order to study the influence of m_t value on the structure of sodium vanadate solution individually, the concentrations of NaOH and V_2O_5 in sodium vanadate solution were fixed at 100 and 50 $g \cdot L^{-1}$, respectively. The structure of sodium vanadate solution was determined by infrared spectrum at different m_t values. The experimental results are shown in Fig. 5.

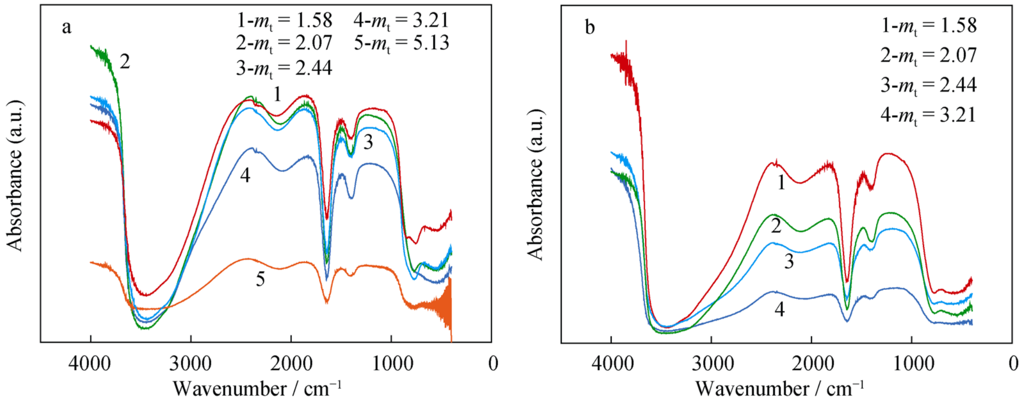

Fig. 5 IR absorption spectra of sodium vanadate solution at different m_t: **a** c(NaOH) = 100 g·L^{-1} and **b** c(V_2O_5) = 50 g·L^{-1}

As shown in Fig. 5a, when NaOH concentration is 100 g·L^{-1}, the peak intensity and area at 1650 cm^{-1} slightly increase with the increase in m_t value, which shows that the concentration of vanadium acid radical ion with V–OH in solution slightly increases. Under the condition of NaOH concentration of 100 g·L^{-1}, m_t values increase with the conductivity increasing. This is because of not only the increase in free hydroxyl ions concentration, but also the change in the structure of vanadium acid radical ion.

When V_2O_5 concentration is fixed at 50 g·L^{-1}, as shown in Fig. 5b, the peak intensity and area at 1650 cm^{-1} increase slightly with m_t value going higher. It shows that that the concentration of vanadium acid radical ion with V–OH in solution decreases rapidly, and then the peak gradually decreases to low band. It is probably because of the higher atomic radius and the decrease in the force constant and the chemical bond vibration frequency. On the other hand, it could be due to the influence of the enhanced conjugate and hydrogen bonding between the aggregation ions and the concentration of the V–OH vanadate ions. So the absorption peak moves to the low band. Simultaneously, the vanadium acid radical ion concentration of the V–O–V aggregation at the peak of 800 cm^{-1} increases. From above, it can be concluded that when the V_2O_5 concentration is fixed at a certain value, the conductivity decreases as m_t goes higher. This is because the concentration of vanadium acid radical ion with V–OH becomes less, and more vanadium acid radical ions with V–O–V form, which means the conductivity of vanadium acid radical ion with V–OH is better than that with V–O–V. The results are in accordance with the calculation results in Table 1 completely.

3.4 Relationships between structure of sodium vanadate solution and its conductivity

In order to study relationships between the structure of the sodium vanadate solution and its conductivity, the infrared spectra under different concentrations of the sodium vanadate solution were tested, respectively. Conditions of the experiment are exactly the same as those given in Table 1, and the infrared spectrum can be presented in Fig. 6.

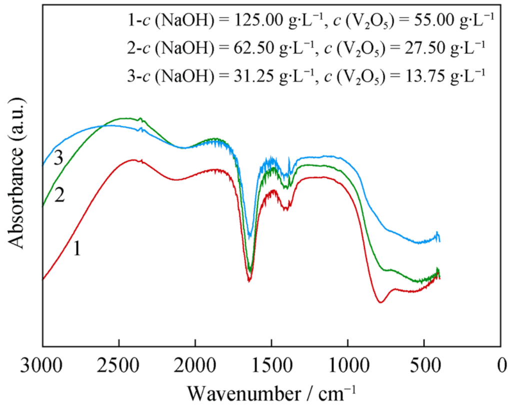

Fig. 6 Changes of IR absorption spectral in diluting process of vanadate solution

As shown in Fig. 6, in the range of the low and middle concentrations, the main vibration peak of the sodium vanadate solution is located at 1650 cm^{-1}. At this point, the value of $[t(\mathrm{Na}^+)+t(\mathrm{OH}^-)]/t(\mathrm{V})$ is from 0.58 to 0.82, which means that the transference number of vanadium acid radical ion is greatly independent of m_t values. It indicates that there exist a large number of vanadium acid radical ions with V–OH in the solution. When NaOH concentration increases to 62.5 g·L^{-1}, there is a characteristic peak at the wavenumber of 800 cm^{-1}. At this point, the value of $[t(\mathrm{Na}^+)+t(\mathrm{OH}^-)]/t(\mathrm{V})$ increases initially and then decreases with the increase in m_t value. This suggests that the solution component concentration affects the number of ion transference obviously. With the concentration increasing, the IR spectrum curves change upward gradually. This means that the polymerization of the vanadium acid radical ions in the solution increases. At this time, the sodium ions have high hydration ability despoil water molecules from vanadium acid radical ion with V–OH. Dehydration of vanadium acid radical ion with V–OH forms vanadium acid radical ion with V–O–V.

So when NaOH concentration increases, the degree of vanadium acid ion polymerization increases. The conclusion is consistent with that in Table 1.

4 Conclusion

The electrical conductivity of sodium vanadate solution increases, and then decreases with the increase of NaOH concentration. In the sodium vanadate solution with high concentration, vanadium acid radical ion mainly exists in the form of aggregation. As the solution dilutes, the polymerization of ions in sodium vanadate solution reduces gradually. Monomer of vanadium acid radical ion with V–OH increases gradually. When diluted to a certain extent, there is only vanadium acid radical ion with V–OH in the sodium vanadate solution.

Under low NaOH concentration, the transference number of vanadium acid radical anion is about 0.58–0.82. The ion structure in IR is vanadium acid radical ion with V–OH mostly in this concentration range. Under medium amount NaOH condition, the transference number of vanadium acid radical anion is about 3.29 and IR result suggests that the ion structure is vanadium acid radical ion with V–OH and V–O–V. Under high concentration, the transference number is about 4.15 and IR result suggests that the ion structure is vanadium acid radical ion with V–O–V chemical bond. In addition, there are also a small number of vanadium acid radical ions with V–OH.

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