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Determining the Concentration of Trace Vanadium in Natural Saline Lake Brines

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Abstract: To determine the concentration of trace vanadium in saline lake brines, a vanadium pre-purification process is established to reduce the matrix effect of the huge coexisting ions. Two steps, extraction and stripping, are included in the process. The factors affecting the vanadium purification efficiencies are investigated in detail and the optimum conditions are determined to be: vanadium in the solutions was extracted by the organic phase containing 30% D2EHPA (*v*/*v*), 20% TBP (*v*/*v*) in n-hexane for 30 min at pH 3.0, and then stripped with 3 mol/L H_2SO_4 for 10 min. Trace vanadium in two natural brine samples are pre-purified using this process and their concentrations are determined by inductively coupled plasma-mass spectrometry (ICP-MS) with sensitivity and limit of detection (LOD) for ^{51}V are 53 171 cps/(μg/L) and 1.88 ng/L, respectively. The standard addition recoveries of the brine samples are ~100% but with small relative standard deviations (RSD<0.6%), indicating that the method can be used to measure the concentration of trace vanadium in natural complicated waters, such as seawater and saline lake brines.

Key words: vanadium; determination; extraction; saline lake brine; purification

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1 Introduction

With the progress of world economy, the energy requirement increased significantly. Comparing with traditional fossil fuels, nuclear power can generate million times higher energy but with much lower greenhouse gas emissions[1]. Uranium is the basis of nuclear energy, but the terrestrial reserves of uranium is only ~7.6 million tons and will be exhausted within one century[2−3]. There are ~4.5 billion tons of uranium in seawater and saline lake brines, which are 1 000 times larger than the terrestrial supply[4]. As an endless uranium resource, the recovery of uranium from these natural complicated systems have received worldwide attentions.

Amidoximated sorbents are the current state-of-the-art materials for collecting uranium (U) from seawater and saline lake brines, experiments showed that vanadium (V) was preferentially extracted over U and many other cations[5−7]. To recover uranium from natural complicated waters by amidoximated sorbents, it is necessary to quantify the influences of vanadium to uranium sorption. However, till now rarely effort has been put on the vanadium concentration determination in natural water samples, especially saline lake brines.

Saline lake brines can be regarded as concentrated seawater, the concentration of vanadium in seawater is about 1.83 μg/L[3, 8]. Although vanadium in saline lake brines may be higher than that in seawater, determining the concentration of vanadium in saline lake brine samples is difficult, because vanadium concentrations may still in be trace level. Atomic absorption spectrometry (AAS)[9−10], fluorescence spectrometry[11], electrochemical analytical method[12], inductively coupled plasma mass spectrometry (ICP-MS)[13], *etc.* had been used to determine the concentration of trace vanadium in solutions, ICP-MS with relative low detection limits is more suitable for trace vanadium determ-

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ination. But since the large number of Na^+, K^+, Ca^{2+}, Mg^{2+}, *etc*. in saline lake water, whose total concentration is at least 10^7 higher than that of vanadium[14], the huge concentration difference makes direct dilution impossible to eliminate the matrix effect of coexisting ions to vanadium concentration determination by ICP-MS.

To reduce the influence of the matrix effect from the coexisting ions and obtain more accurate vanadium concentrations in brine samples, a vanadium pre-purification process is necessary. Many methods, such as extraction, co-precipitation and ion exchange[15−16], were adopted to separate vanadium from complicated sample matrices. Solvent extraction is more widely used due to the easy operation with high selectivity and extraction efficiency[17−18]. Di-(2-ethylhexyl) phosphoric acid (D2EHPA) is an excellent extractant for V(IV). Many researches had observed that more than 80% of vanadium were extracted by D2EHPA[19−22]. Moreover, in most of these studies, tributyl phosphate (TBP) was used as an interface modifier to improve the phase separation and the extraction efficiency[19, 21−22].

In present work, to determine vanadium concentrations in saline lake brine samples, a vanadium pre-purification process was established. Two steps including extraction and stripping were involved in the process, D2EHPA and H_2SO_4 were separately used as the extraction and stripping reagents. Considering the high salinity of the brine samples, TBP was also added to avoid the formation of the third phase in the extraction step. The parameters influencing the vanadium purification efficiencies were systematically investigated and vanadium concentrations in two natural saline lake brine samples were determined. Our study provides a simple analytical method for vanadium concentration determination in natural water samples, and makes it easier to quantify the influence of vanadium to uranium recovery from seawater and saline lake brines.

2 Materials and methods

2.1 Materials

$Na_3VO_4 \cdot 12H_2O$ and Na_2SO_3 were products of Aladdin and Kelong reagent companies, respectively. D2EHPA was purchased from Energy Chemical Trading Co., Ltd. (Shanghai); Tributyl phosphate (TBP) was acquired from Tianjin Kemiou Chemical Reagent Co., Ltd., and other reagents such as n-hexane, carbon tetrachloride, benzene and trichloromethane were bought from the companies of China. All reagents were of analytical reagent grade and used without purification.

Two natural brine samples named as S1 (discharged water of a saline Potash Fertilizer Plant) and S2 (saline field brine) were collected from different places of Qinghai Province, China.

2.2 Methods

2.2.1 Optimizing the vanadium pre-purification process

Two steps were included in the vanadium pre-purification process: extracting vanadium with D2EHPA and stripping it with H_2SO_4 solution. In present work, both steps were optimized to ensure the maximum recovery of vanadium ions. The complicacy of brine samples makes them impossible to be directly used in optimizing the process, pure vanadium solutions with concentration of 5 mg/L (dissolved in 0.5 mol/L H_2SO_4) were adopted. The concentrations of vanadium in the solutions were determined by inductively coupled plasma-atomic emission spectrometry (ICP-AES) (Agilent 5100, Agilent technologies, USA).

Before extraction, sufficient amount of Na_2SO_3 was added to reduce vanadium (V) to vanadium (IV), since D2EHPA had better extraction property toward vanadium (IV) [23].

In the extraction step, organic phase, containing D2EHPA (extraction reagent), TBP (interface modifier) and diluent, with total volume of 3 mL was contacted with the same volume of vanadium solution at an oscillation speed of 300 rpm. To optimize the extraction conditions, the effects of D2EHPA and TBP concentrations, diluents, pH of vanadium solution and time on extraction efficiency (E) were investigated. After sufficient contacting, the mixed solution was further centrifuged at 5 000 rpm for 5 min to ensure the complete separation of inorganic and organic phase. The extraction efficiency (E) was calculated by

$$E = \frac{C_t - C_I}{C_t} \times 100\%, \tag{1}$$

where C_t and C_I are the vanadium concentrations in inorganic phase before and after extraction, respectively. $C_t - C_I$ is the vanadium concentration in organic phase.

In the stripping step, 2 mL organic phase loaded with vanadium was stripped by the same volume of H_2SO_4 on an oscillator shaking at 300 rpm for 10 min. To optimize the stripping conditions, H_2SO_4 with different concentrations were applied as the stripping reagents. After centrifuging the mixed solution at 5 000 rpm for 5 min, vanadium concentration in the stripped inorganic phase was determined. The stripping ratio (E_b) was calculated by

$$E_b = \frac{C_{bI}}{C_t - C_I} \times 100\%, \tag{2}$$

where C_{bI} is the vanadium concentration in the stripped inorganic phase.

The parameters which can acquire the highest vanadium extraction and stripping efficiencies were selected as the optimum conditions for pre-purifying vanadium.

2.2.2 Reliability test of the pre-purification process

Since trace level of vanadium existed in brine water, ICP-MS (iCAP-QC+ICS5000+, Thermo Fisher Scientific, USA) was used to get the vanadium concentration in these samples. To ensure the optimum vanadium purification conditions determined by ICP-AES are reliable in trace vanadium concentration measurement by ICP-MS, experiments were carried out with standard vanadium solutions of 20 μg/L, but concentrations were acquired by ICP-MS. The operation parameters of ICP-MS were listed in Table 1.

Table 1 The operational parameters of ICP-MS.

Parameter	Value
Plasma power/W	1 550
Cool flow/(L·min^{-1})	14
Nebulizer flow/(L·min^{-1})	0.97
Auxilliary flow/(L·min^{-1})	0.80
Sample depth/mm	5.00
Torch horizontal position/mm	−0.48
Torch vertical position/mm	1.07
Spray chamber temperature/℃	2.7
Extraction lens 2/V	−90.67
CCT focus lens	−0.96

2.2.3 Determination of vanadium concentrations in natural brine samples

Brine samples S1 and S2 are saturated salt solutions with obvious crystal structures. They were two times diluted so as to make the pH adjustment easier.

Vanadium concentrations in these brines samples were measured by the standard recovery technique. Furthermore, to reduce the matrix effect of the huge coexisting ions, 30 mL diluted brine water was extracted by 6 mL organic phase, but the phase ratio of the stripping step is still 1:1. The vanadium in diluted brine and standard spiked diluted brine samples were purified and then their concentrations were measured by ICP-MS. The standard addition recovery (E_r) of each sample was acquired by[24−25]

$$E_r = \frac{C_{aB} - C_{mB}}{C_a} \times 100\%, \tag{3}$$

where, C_{mB} and C_{aB} are the measured vanadium concentrations of the diluted brine and standard spiked diluted brine samples, respectively. C_a is the spiked standard vanadium concentration.

Hence, the actual vanadium concentration of the brine samples (C_B) were:

$$C_B = C_{mB} \times 2 / E_r, \tag{4}$$

because the brine samples were two times diluted.

3 Results and discussion

3.1 Optimizing the vanadium pre-purification process

The effect of D2EHPA concentration [varied from 10% to 30% (v/v)] on vanadium extraction is shown in Fig.1(a). Although vanadium extraction efficiency increases slowly with increasing D2EHPA concentrations, it achieves 76% when D2EHPA concentration is only 10%.

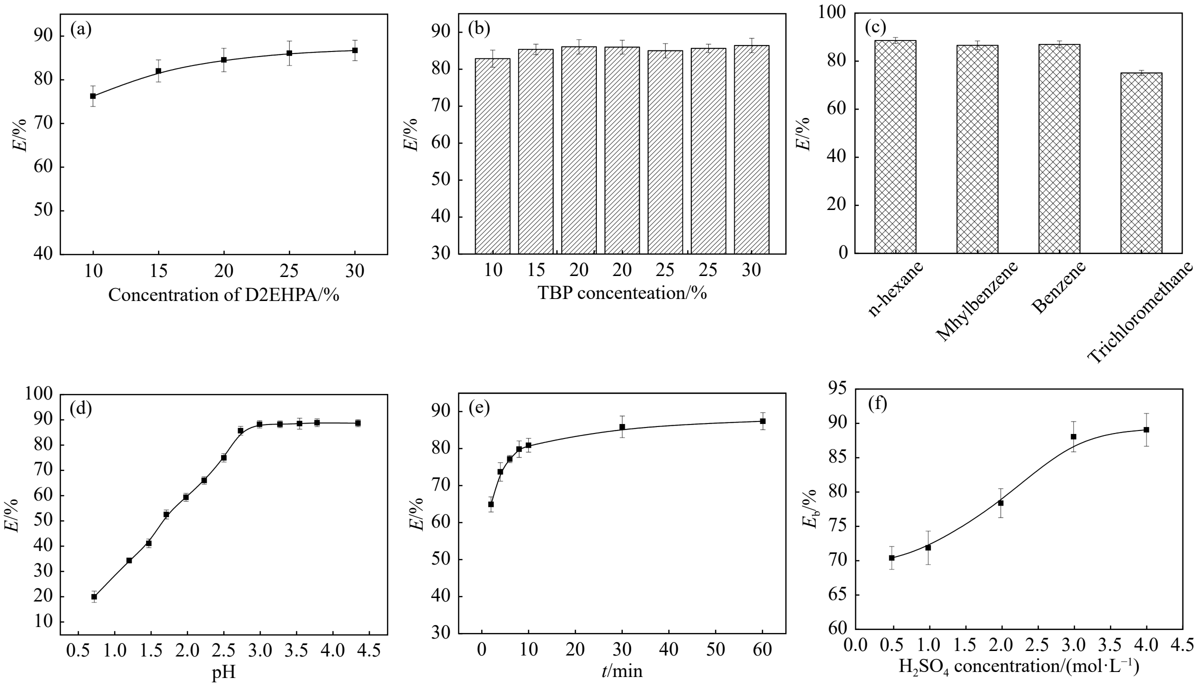

Fig. 1 Optimizing the vanadium pre-purification process.

The effects of D2EHPA concentration (a), TBP concentration (b), diluents (c), solution pH (d) and time (e) on vanadium extraction efficiency. The effect of H2SO4 concentration on vanadium stripping efficiency (f).

The highest vanadium extraction efficiency is 87% when D2EHPA concentration is 30%. Hence, D2EHPA concentration of 30% is sufficient to achieve relative high vanadium extraction efficiency.

To avoid the third phase formation in the extraction process, TBP is used as the interface modifier. The volume concentration of TBP was changed from 5% to 25% (*v*/*v*). As shown in Fig.1(b), the extraction efficiencies of V(IV) are independent of TBP concentrations and keep nearly constant. This is different from the results of Zhang *et al*.[23] where V(IV) extraction was enhanced by TBP, but consistent with the finding of Cherafhi *et al*.[26]. Although TBP has negligible effect on vanadium extraction from pure solutions, small floccule appeared in the saline lake brine samples at TBP concentrations less than 10% (data not shown for concise). Hence, TBP concentration was chosen to be 20%.

In addition, n-hexane, methylbenzene, benzene and trichloromethane were respectively employed as diluent to evaluate their effects on vanadium extraction, the acquired results are illustrated in Fig.1(c). The highest and lowest extraction efficiencies are obtained with n-hexane and trichloromethane as the diluents, respectively. While the extraction efficiencies are close to each other when methylbenzene and benzene are the diluents. This phenomenon is correlated with the stability of D2EHPA-vanadium ion complex and influenced by diluents. More stable D2EHPA-vanadium ion complex can be formed when D2EHPA is polymerized[26], and it is much easier for D2EHPA to form dipolymer in the diluent with lower polarity. The polarity of n-hexane, methylbenzene, benzene and trichloromethane are 0, 2.4, 2.7 and 4.1, respectively. Therefore, the most effective extraction of vanadium is observed when n-hexane was selected as the diluent.

The forms of vanadium(IV) in water solutions are very complicated and vary with solution pH[27]. Hence, solution pH is an important factor that affects the extraction ratio. At pH lower than 4.5, vanadium(IV) is mainly existed as VO^{2+} and convert to $HV_2O_5^-$ at higher pH. To avoid the influences caused by vanadium(IV) form, vanadium solutions with pH of 0.75 to 4.5 were selected. As shown in Fig.1(d), vanadium extraction efficiencies rise linearly within solution pH of 0.75 to 3.00, then keep almost constant with further increase of pH, which is in line with the references[22, 28]. D2EHPA is an acid extractant, the effective extraction of vanadium ions is caused by exchanging H^+ ions in D2EHPA with VO^{2+} ions through the reaction $VO^{2+}+2H_2A_2=VOA_2\cdot 2HA+2H^+$[29]. Here, D2EHPA was represented by HA. Therefore, increasing the solution pH will facilitate the extraction process. Moreover, the forms of vanadium ions are also influenced by vanadium concentration, higher vanadium concentration leads to a lower pH range for VO^{2+} to be stable[16]. The investigated vanadium concentration here was only 5 mg/L, two orders of magnitude lower than the vanadium concentrations in references (500 mg/L[26] or 1 810 mg/L[28], optimum extraction pH of ~1.8). Therefore, the maximum vanadium extraction efficiency is obtained at pH of 3.00 and remains constant in pH from 3.00 to 4.50. To ensure high extraction efficiency, solution pH of 3.0 was selected.

Except for the above conditions, the effect of time on extraction efficiency was also investigated and data are illustrated in Fig.1(e). The extraction efficiency can reach 65% within 2 minutes and 10 minutes is enough to achieve extraction equilibrium. In fact, the relatively low initial vanadium concentration (5 mg/L) may be another reason for the fast extraction kinetics. To guarantee the stable vanadium extraction efficiency, extraction time of 30 min was employed.

The extracted vanadium must be stripped and further used for vanadium concentration determination. H_2SO_4 with concentrations of 0.5 to 4 mol/L were applied as the stripping reagents. From Fig.1(f), it can be found that the stripping efficiencies display positive correlation with the increased H_2SO_4 concentrations and (88.7±2.5)% of the loaded vanadium can be stripped by $H_2SO_4 \geqslant 3.0$ mol/L. On the contrary to the extraction procedure, more acid environment will benefit the stripping efficiency. Hence, 3.0 mol/L H_2SO_4 was used as the stripping solution.

Based on the above investigations, the vanadium pre-purification process can be determined (Fig.2). After adjusting the valence of vanadium and solution pH, 3 mL vanadium solution is extracted by the organic phase composed of 30% D2EHPA (*v*/*v*), 20% TBP (*v*/*v*) in n-hexane for 30 min at pH 3.0; and then vanadium in the organic phase is stripped by 3 mol/L H_2SO_4 of 2 mL for 10 min. The phase ratio of each step was 1 : 1.

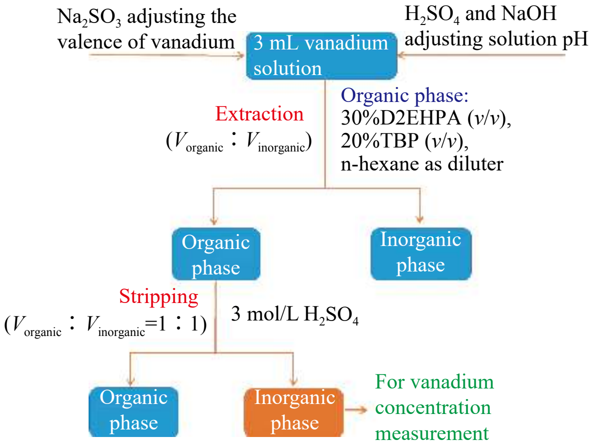

Fig. 2　(color online) Scheme of the vanadium pre-purification process.

3.2　Reliability test of the pre-purification process

The calibration curve of ^{51}V measure by ICP-MS within 0 to 20 μg/L is: A = 53 171 C_V (μg/L) – 4 168.9

(R^2=0.99). Here, A is the measured intensity of ^{51}V and C_V is the vanadium concentration (μg/L). Thus, the sensitivity of ICP-MS for vanadium is 53 171 cps/(μg/L). The limits of detection (LOD) and quantification (LOQ) can be further estimated to be 1.88 ng/L (3 S_b/s) and 6.27 ng/L (10 S_b/s), respectively. Where S_b is the standard deviation of the blank and s is the slope of the analytical curve analytical curve [9].

To ensure whether the vanadium pre-purification process is reliable in trace vanadium measurement by ICP-MS, three standard vanadium solution with concentration of 20 μg/L are undergone the vanadium pre-purification process (Fig.2). Vanadium concentrations in the inorganic phases of each step were quantified by ICP-MS. The extraction and stripping efficiencies as well as their corresponding relative standard deviations (RSDs) are calculated and listed in Table 2. All the extraction and stripping efficiencies are higher than 93% with small RSDs. Hence, the vanadium pre-purification process is reliable to be used in trace vanadium concentration measurement by ICP-MS.

Table 2 The extraction and stripping efficiencies of the standard samples.

Measured number	Extraction efficiency/%			Stripping efficiency/%		
	Calculated value	Average	RSD/%	Calculated value	Average	RSD/%
1	94.50	93.84	0.37	95.14	96.34	1.41
2	93.68			97.82		
3	93.32			96.05		

3.3 Determination of vanadium concentrations in natural brine samples

S1 and S2 are natural brine samples collected from different places of Qinhai Province, China. The concentrations of the main co-existing ions in these two samples were listed in Table 3. It is clear that the concentrations of Na^+, K^+, Ca^{2+}, Mg^{2+} and Cl^- are extremely high, so their matrix effect to vanadium concentration determination must be reduced.

The standard addition technique was used for vanadium concentration determination in natural brine samples. The measured vanadium concentrations in diluted brine and standard spiked diluted brine samples are listed in Table 4. The average standard addition recoveries (E_r) are found to be (104.4±0.3)% for brine sample S1 and (101.2±0.5)% for brine sample S2 (Table 4). The admitted standard addition recovery ranges are 80%~120%, 90%~110% and 95%~105%, if the concentrations are ⩽1 mg/L, 1~100 mg/L and ⩾100 mg/L, respectively[30]. The actual vanadium concentrations (C_B) of S1 and S2 are (6.58±0.08) μg/L and (6.96±0.15) μg/L, respectively (Table 4). These values are much lower than 1 mg/L, so the obtained standard addition recovery of sample S1 and S2 are acceptable. Moreover, the RSD values for both brine samples are less than 2.5% (Table 4). With the high recovery and small RSDs, the method obtained in present work can be used for determining the concentration of trace vanadium in natural complicated waters, such as seawater and saline lake brines.

Table 3 The concentrations(mg/L) of the main co-existing ions in saline sample S1 and S2.

Sample name	Na^+	K^+	Mg^{2+}	Ca^{2+}	Cl^-	SO_4^{2-}
S1	5 595	24 702	101 413	19 215	207 616	51 376
S2	66 221	35 228	15 828	18 610	105 856	48 725

Table 4 Measured vanadium concentration in the diluted and standard spiked diluted brine samples and the actual vanadium concentration of brine samples.

Sample	Measured number	C_{mB}/(μg·L^{-1})	C_a/(μg·L^{-1})	C_{aB}/(μg·L^{-1})	E_r/%	Average/%	RSD/%	C_B/(μg·L^{-1})	Average/(μg·L^{-1})	RDS/%
S1	1	3.47	10.67	14.45	104.1	104.4	0.30	6.66	6.58	1.3
	2	3.44	10.67	14.67	104.4			6.59		
	3	3.40	10.67	14.62	104.8			6.49		
S2	1	3.58	10.67	13.97	100.7	101.2	0.54	7.11	6.96	2.2
	2	3.46	10.67	14.28	101.7			6.80		
	3	3.52	10.67	14.71	101.2			6.96		

4 Conclusion

To determine the concentration of trace vanadium in saline lake brine samples in the presence of macro amount of the co-existing ions, a vanadium pre-purification process is established. Factors affect the vanadium purification efficiencies are systematically investigated and the optimum conditions are determined. By using the acquired pre-purification process, vanadium concentrations of two natural brines samples are identified through the standard addition technique. The obtained high addition recoveries and small RSDs of both brine samples indicate that the method can be used to determine the concentration of trace vanadium in natural complicated waters.

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盐湖卤水中微量钒的浓度测定

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摘要: 为了测定盐湖卤水中微量钒的浓度，开发了一个钒的分离纯化流程来降低大量的共存离子的基质效应。该分离纯化流程包括萃取和反萃两个步骤。详细地研究了影响钒纯化效率的各种因素，得到钒的最佳分离纯化条件为：以正己烷为稀释剂，有机相中D2EHPA和TBP的体积百分比分别为30%及20%，在pH为3.0时萃取30 min；然后用3 mol/L H_2SO_4反萃10 min。基于此分离纯化流程，将两个实际盐湖卤水样品中的微量钒纯化后，再用电感耦合等离子体质谱仪(ICP-MS)测定其浓度，该ICP-MS对^{51}V的检测灵敏度和检测限别为53 171 cps/(μg/L)和1.88 ng/L。所得实际盐湖卤水钒测定结果的加标回收率接近100%而相对标准偏差低于0.6%，表明该方法可用于实际复杂体系中微量钒的浓度测定，例如海水和盐湖卤水。

关键词: 钒；测定；萃取；盐湖卤水；纯化

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