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Simultaneous extraction of vanadium and chromium from vanadium slag using low-pressure liquid phase oxidation method

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Abstract: A low-pressure liquid oxidation method was proposed and proven to be effective to extract vanadium and chromium simultaneously from the vanadium slag in concentrated NaOH aqueous solutions. The effect of temperature, NaOH mass concentration, liquid-to-solid mass ratio, stirring speed and pressure on the extraction of vanadium and chromium in NaOH aqueous solutions were systematically investigated. Under the optimal reaction conditions (temperature of 473 K, liquid-to-solid mass ratio of 6:1, stirring speed of 700 rpm, NaOH mass concentration of 50%, pressure of 1 MPa and reaction time of 180 min), the vanadium and chromium recovery reached 95% and 90%, respectively. It was found that the reaction temperature and NaOH concentration were important factors for the extraction of vanadium and chromium. The kinetics of the decomposition of vanadium slag in concentrated NaOH aqueous under low pressure was analyzed using the shrinking core model, and the results indicated that the extraction of vanadium and chromium were both governed by the internal diffusion step, with apparent activation energies calculated to be 26.22 and 32.79 kJ/mol, respectively.

Keywords: vanadium slag; low pressure leaching; liquid phase oxidation; kinetics.

1. Introduction

Vanadium is a scarce resource and a strategic metal. Vanadium and its compounds are widely used in the fields of metallurgical industry, petrochemical industry, defense industry, electronic industry, paint and coatings industry etc. (Yang, 2010), due to their excellent physicochemical properties (Huang, 2000; Boudin et al., 2000).

Vanadium slag, produced by vanadium-titanium magnetite ores, is one of the most widely used resources for vanadium production (Ena, 1982), and about 60% of the vanadium products use vanadium slag as raw material globally (Moskalyk et al., 2010). Currently, sodium salt roasting is the most representative and the most widely used method for extracting of vanadium from the vanadium slag (Wang et al., 2014), and this process mainly consists of the following operation units: salt roasting, water leaching, purification, precipitation and V₂O₅-calcination (Ye, 2006). During the salt roasting operation, the low-valance vanadium oxide in the vanadium slag could be oxidized to sodium metavanadate in the presence of sodium reagents such as NaCl, Na₂CO₃ and Na₂SO₄. Such operation typically proceeds under oxidative environment in a rotary kiln or a multiple hearth furnace at high temperature (1023-1123 K) (Liang, 1975). Although the technological process is simple with good product quality, the vanadium recovery is 60-70% (Wang et al., 2012). Moreover, chrome spinel associated in the vanadium slag is much more stable than the vanadium spinel, thus the chrome spinel cannot be oxidized effectively under these conditions, leading to serious resource waste and potential environmental pollution risks (Liu et al., 2013). Besides, during the salt roasting, large

quantity of aggressive gases, such as Cl_2 , SO_2 , and HCl are generated due to the pyrolysis of sodium reagents and their interactions with the water vapor (Yang and Jin, 2007).

In order to improve the vanadium recovery, minimize the environmental impact, as well as realize comprehensive utilization of associated chromium during vanadium extraction, liquid-phase oxidation methods have been developed recently (Liu et al., 2012; Wang et al., 2012; Liu et al., 2013; Liu et al., 2013; Wang et al., 2014) In these processes, the vanadium slag was treated in either molten NaOH-NaNO_3 medium (Liu et al., 2013) or highly concentrated alkali metal hydroxide solutions (known as the sub-molten salt medium, SMS) under oxidative conditions (Liu et al., 2017). Compared to traditional roasting method, the operation temperatures in these new methods drop from 1023-1123 K to 473-673 K, yet the yields of vanadium and chromium can reach 96% and 90% after reacting for 6 h, respectively (Liu et al., 2011). Furthermore, no hazardous gases or toxic tailings are discharged during the whole processes. However, excessive alkali is added in the leaching process. To realize the recycle of reaction medium, much energy need to be consumed, which hinders the industrial application. At present, several improved sub-molten salt methods have been studied. WANG's (Wang et al., 2012) NaOH sub-molten salt method used activated carbon to intensify the oxidation of vanadium slag to achieve the simultaneous extraction of vanadium and chromium at the alkali concentration of 80%. LIU (Liu et al., 2017) used the micro-bubble generator to reduce the oxygen bubble size to increase the amount of dissolved oxygen in the medium, thereby the vanadium slag oxidation was enhanced. The NaOH concentration effectively reduced from 80% to 60%. However, 60% is still not a satisfactory alkali concentration, and the energy consumption and corrosion of the equipment is still not a negligible issue. Therefore, optimization of the liquid oxidation process to realize high V and Cr recovery in less concentrated alkali solutions is in an urgent need. In this paper, pressurized oxidative leaching of vanadium slag under low alkali concentration conditions has been examined, and the main reaction parameters include alkali concentration, reaction temperature, reaction time, liquid-to-solid mass ratio, and system pressure on the influence of vanadium and chromium leaching rate have been systematically investigated. Further, leaching kinetics and mechanisms have been discussed to provide in-depth information with respect to this new technology.

2. Materials and methods

2.1 Materials

The vanadium slag, supplied by Chengde Iron and Steel Group Co., Ltd., Hebei, China, was dried in an oven overnight, followed by dry-sieving to obtain particles with diameters of less than 200 mesh. The chemical composition is listed in Table 1. The sodium hydroxide used in the experiments was of analytical grade and was obtained from Xilong Chemical Group Co., Ltd. Deionized water (produced by water super-purification Milli-Q, Millipore) was used throughout the experiments. Commercial pure oxygen was obtained from Beijing Qianxi Gas Sales Center.

Table 1. The composition of the vanadium slag (<200 mesh)

Component	V_2O_5	Cr_2O_3	FeO	SiO_2	Al_2O_3	MgO	CaO	MnO_2	TiO_2
Content, wt%	10.2	4.15	49.01	20.21	2.42	1.6	1.22	5.22	11.03

2.2 Experimental apparatus and procedure

All experiments were carried out in a 1000 cm^3 nickel reactor with a spherical bottom (GSHA-1000 cm^3). The reactor was placed in an electric hood and was equipped with a temperature controller (CKW-III) with a precision of ± 1 K, a stirrer with double paddles and a cooling coil. Oxygen was passed through the reaction system via an aeration pipe installed at the bottom of the reactor and the oxygen flow was controlled by an oxygen flowmeter (D07-7B). The oxygen inlet pressure of the autoclave was controlled by a pressure relief valve at the rear of the cylinder, and the back-pressure valve after the reactor was used to maintain the pressure inside the reactor. A schematic diagram of the experimental apparatus is shown in Fig. 1.

For each run, certain amount of vanadium slag with particle size of less than 200 mesh and certain amount of NaOH were prepared. Subsequently, the autoclave was sealed and stirred to keep the

slurry suspended during the experiment. The autoclave was placed in the cavity of a heating furnace, then heated to the preset reaction temperature and oxygen was injected. The oxygen pressure was quickly raised to the desired value and simultaneously, timing of the reaction was initiated. At selected time intervals, about 2-3 g of the reacting slurry sample was taken out and diluted using 80 cm³ of deionized water, followed by filtration and washing to obtain the residues for further analysis. To ensure the accuracy of the sample composition, all washing and filtering water was also deionized water and all the experimental equipment used was also washed with deionized water. After the reaction, the slurry was diluted with deionized water, and then filtered and washed to obtain a leached cake for further analysis. The vanadium and chromium extraction efficiency from vanadium slag was calculated using the following equation:

$$X = \left(1 - \frac{[C]_r}{[C]_0}\right) \times 100\% \quad (1)$$

where $[C]_0$ and $[C]_r$ are the contents of chromium or vanadium in the original vanadium slag and the residue sample that was taken at a certain reaction time, respectively.

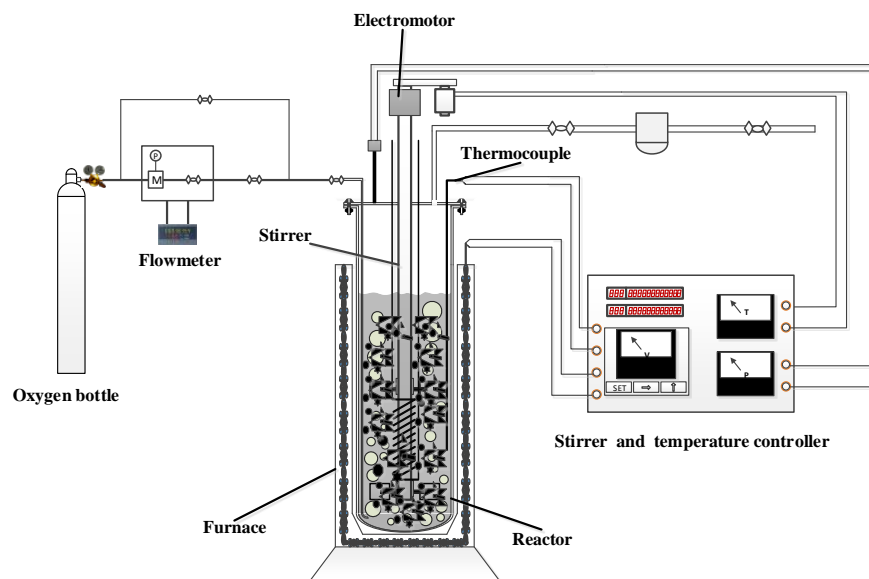


Fig. 1. Diagram of the experiment set-up

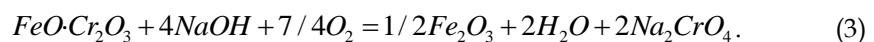
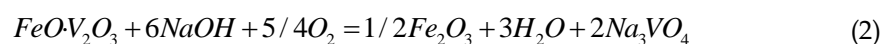
2.3 Analysis instruments

The composition, phase structure and the morphology of the original vanadium slag and the residues were analyzed using ICP-OES (PE Optima 7300V, PerkinElmer), X-Ray Diffraction (XRD, Phillips PW223/30), and the SEM (JSM-7001F+INCA, JEOL) equipment, respectively.

3. Results and discussion

3.1 Reaction principles

In a vanadium slag particle, V, Cr, Ti, Mn and Fe primarily exist in the central area as spinel (FeV_2O_4 , FeCr_2O_4), and Si primarily exists in the surrounding area as quartz (SiO_2) or fayalite (Fe_2SiO_4) (Jin et al., 2010). In the alkali solutions, the silicates can react with NaOH, exposing the spinel to the reaction medium (Wang, 2012). In the presence of oxygen, the spinel could be oxidized to water-soluble vanadate and chromate, as described below:



According to the thermodynamic data (Ye and Che, 1981; Liang, 1993), the $\Delta_r G^\theta$ of vanadium and chromium spinels oxidation reaction was calculated and the plots is shown in Fig. 2. It is clear that the oxidation reactions in NaOH alkaline medium are thermodynamics feasible.

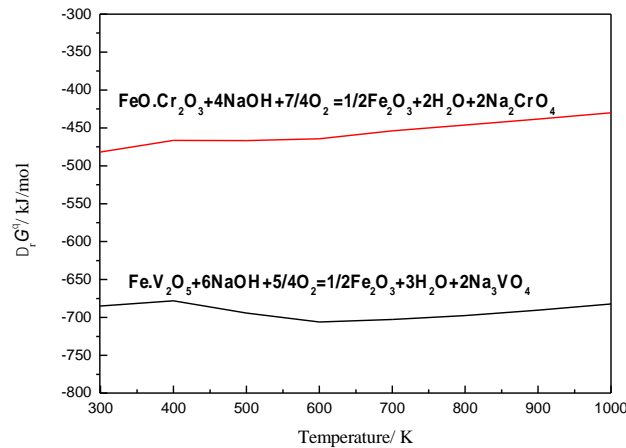


Fig. 2. Standard Gibbs free energy change versus temperature for vanadium and chromium spinels oxidation in the NaOH-H₂O-O₂ system

3.2 The effects of alkali mass concentration

The effect of alkali mass concentrations on extraction of vanadium and chromium was investigated, with other conditions fixed to be: pressure of 1 MPa, temperature of 473 K, liquid-to-solid mass ratio of 6:1, and stirring speed of 700 rpm. The alkali mass concentrations are in the range of 35% to 55%, and the results are shown in Fig. 3.

As can be seen from the results, the leaching rates of vanadium and chromium increased with the NaOH mass concentration. When the mass concentration of NaOH was 50%, the leaching rates of vanadium and chromium reached the maximum values of 94.67% and 89.65% at 180 min, respectively. Further increase the NaOH mass concentration to 55% did not show significant influence on the vanadium and chromium leaching rates. This may be due to the fact that the oxidation of vanadium and chromium in vanadium slag is greatly related with the amount of dissolved oxygen in the medium. As the literature suggested (Beijing Petrochemical Engineering Company, 1988), the solubility of oxygen decreases with increasing of the alkali mass concentration, which is unfavorable for the oxidation reactions. Therefore, 50% was chosen as the optimal alkali concentration.

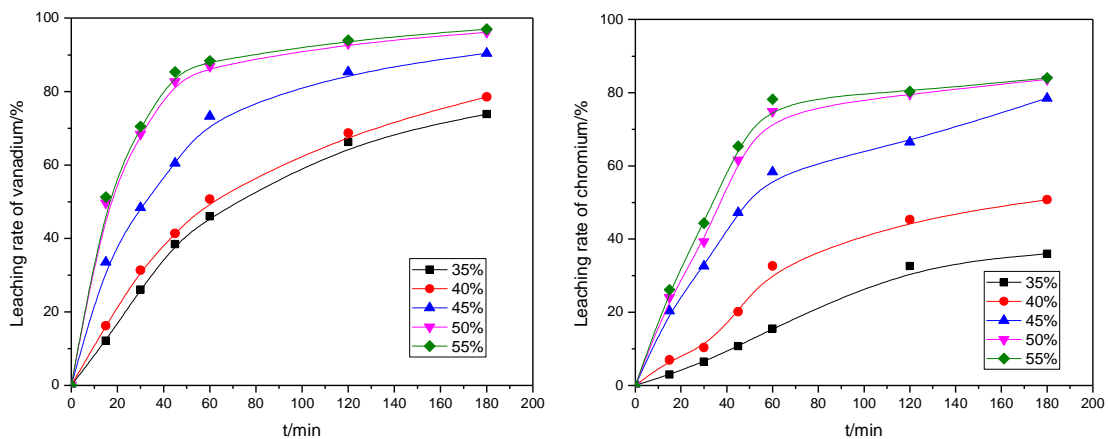


Fig. 3. Effect of NaOH concentration on the extraction of vanadium and chromium

3.3 Effect of agitation

Agitation affects the mass transfer of the medium, especially the diffusion of oxygen, which is closely related with the oxidation of vanadium and chromium. The effect of agitation speed was therefore investigated under the conditions of pressure 1 MPa, temperature 473 K, alkali mass concentration 50%, and liquid-to-solid mass ratio 6:1. The agitation speed was set from 100 rpm to 900 rpm. The results are shown in Fig. 4.

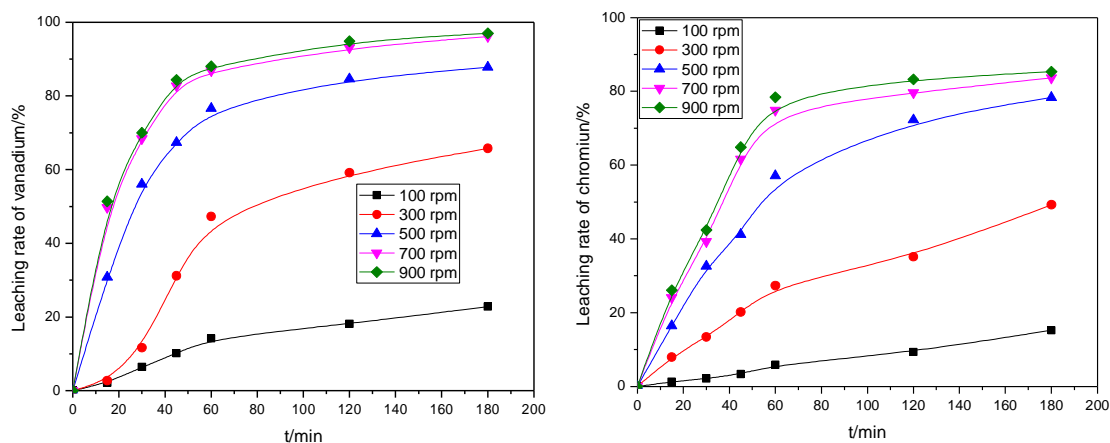


Fig. 4. Effect of agitation speed on the extraction of vanadium and chromium

It was observed that the vanadium and chromium conversion obviously increased with agitation speed. When the agitation speed was elevated from 100 rpm to 700 rpm, the leaching rate of vanadium increased from 22.8% to 97.0%, while that of chromium increased from 15.2% to 83.6%. It means the vanadium slag particles were effectively reacted with alkali medium and oxygen, as the agitation speed became higher. However, when the agitation speed was further increased to 900 rpm, the increase of vanadium and chromium leaching rates was negligible. Therefore, the agitation speed of 700 rpm was enough to ensure the mass transfer effect. Based on this, the agitation speed will be set at 700 rpm in the following study.

3.4 Effects of pressure

In order to investigate the effect of pressure on the conversion of vanadium and chromium, the pressure was set to be 0.5, 0.75, 1 and 1.25 MPa with other parameters fixed as: alkali mass concentration of 50%, temperature of 473 K and liquid-to-solid mass ratio of 6:1. The relationship of pressure and conversion is shown in Fig. 5.

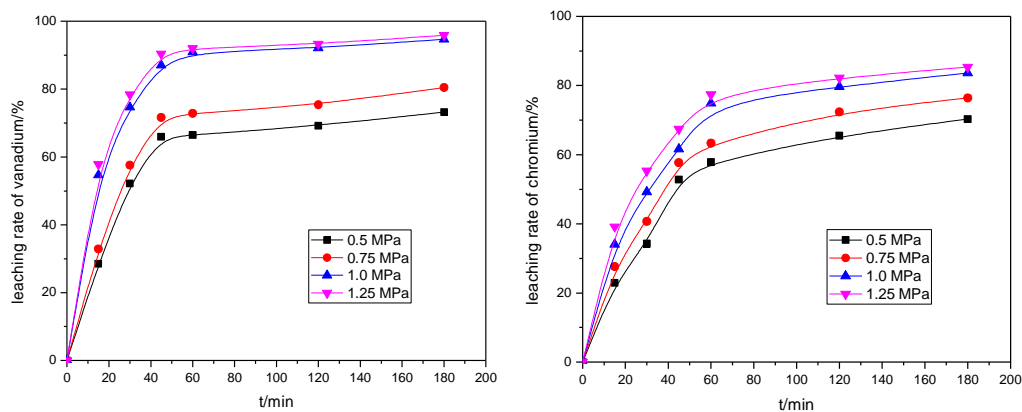


Fig. 5. Effect of system pressure on the extraction of vanadium (left) and chromium (right)

It can be seen that as the pressure increased, the leaching rates of vanadium and chromium gradually increased. According to Henry's Law, the solubility of oxygen in the NaOH solution is proportional to the oxygen partial pressure above the solution. As a result, the leaching rates of vanadium and chromium at 1.0 MPa are obviously higher than those obtained at 0.5 MPa and 0.75 MPa. Because high pressure is very challenging for industrial application, 1 MPa was chosen for the following experiments rather than 1.25 MPa.

3.5 Effect of liquid-to-solid mass ratio

High liquid-to-solid mass ratio is always beneficial for the reaction to due to the improvement of the mass transfer. The effect of liquid-to-solid mass ratios was investigated under the conditions:

temperature 473 K NaOH mass concentration 50%, stirring speed 700 rpm and pressure 1 MPa. The liquid-to-solid mass ratios of 2:1, 4:1, 6:1 and 8:1 were examined and the results are presented in Fig. 6.

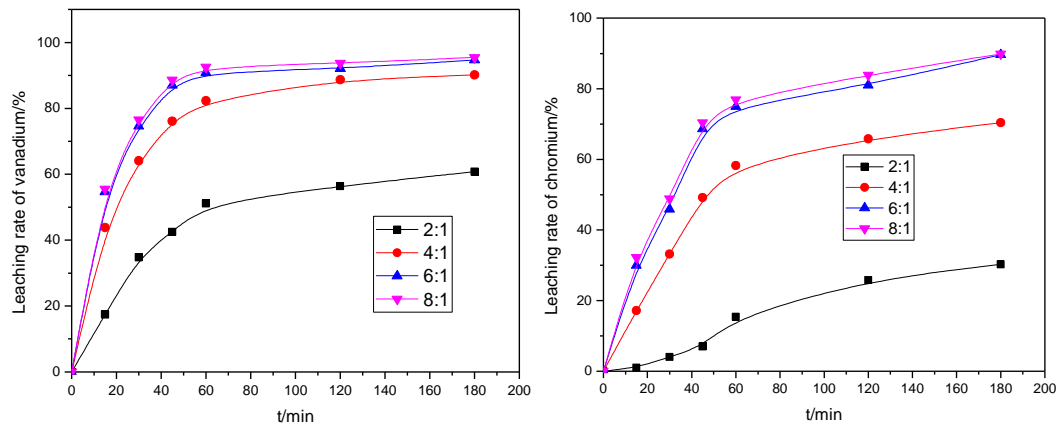


Fig. 6. Effect of liquid-to-solid mass ratio on the extraction of vanadium and chromium

It can be seen that the vanadium and chromium conversions increased with the liquid-to-solid mass ratio. The leaching rates of vanadium and chromium reached 97.0% and 86.3% respectively at the mass ratio of 6:1. But when the mass ratio further increased from 6:1 to 8:1, the conversions of vanadium and chromium showed almost no further increase. This is because when the mass ratio of liquid-to-solid was high enough, the reaction agent and vanadium slag particles can be fully contacted under the strong agitation. Thus, a liquid-to-solid mass ratio of 6:1 was chosen for further experiment.

3.6 Effect of temperature

The effect of temperatures on the extraction of vanadium and chromium was investigated in 50% NaOH solutions at the pressure of 1 MPa, with a mass ratio of 6:1 and agitation speed of 700 rpm. The results are shown in Fig. 7.

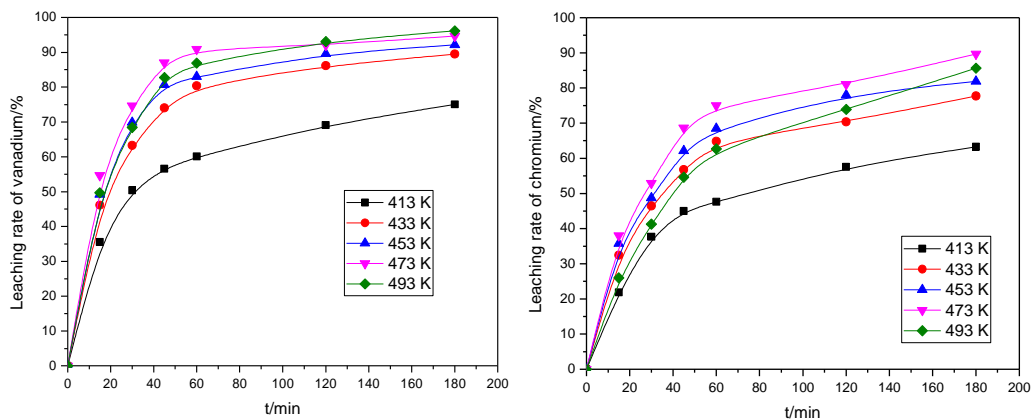


Fig. 7. Effect of temperature on the extraction of vanadium (left) and chromium (right)

As we know, high temperature can decrease the medium viscosity and increase the reaction activity, and thus the leaching rates of vanadium and chromium normally increase with temperature. It can be seen that when the temperature increased from 413 K to 473 K, the leaching rate of vanadium increased from 75.04% to 94.67%, while the leaching rate of chromium increased from 63.27% to 89.65%. However, when the temperature was elevated to 493 K, the leaching rates of vanadium and chromium did not show further increase. This is due to the fact that the water vapor pressure would increase as the temperature was elevated, which will lead to the decrease of oxygen partial pressure, inhibiting the oxidation of vanadium and chromium. Thus, the optimal temperature was chosen to be 473 K.

3.7 XRD and SEM analysis of reacted residues

During the leaching of vanadium slag, the phase change of vanadium slag with time were investigated, and the results are shown in Fig. 8. The original vanadium slag mainly contained spinel, fayalite and quartz. The diffraction patterns of fayalite and quartz quickly disappeared after reacting for 15 min, suggesting that the decomposition of silicon-containing phases in NaOH solutions were very fast. However, the decomposition of vanadium and chromium spinels proceeded progressively. Until reaction 180 min, the spinels was completely oxidized, as indicated by the vanishing of spinel diffraction peaks.

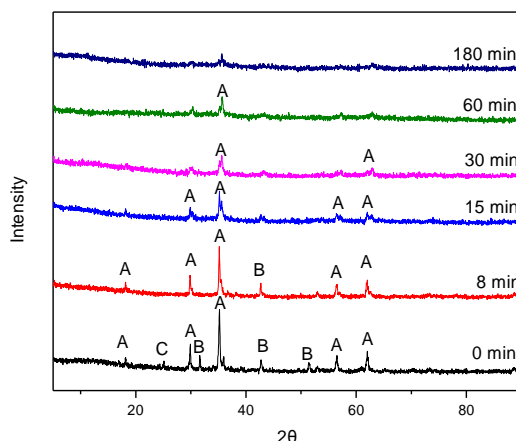


Fig. 8. XRD patterns of leaching residue at different conversion time (A: $(\text{Mn, Fe})(\text{V, Cr})_2\text{O}_4$; B: Fe_2SiO_4 ; C: SiO_2)

The morphology change of vanadium slag at different reaction time is shown in Fig. 9. It can be seen that the untreated vanadium slag particles are featured as compact solid with smooth surface. However, after reaction 15 minutes, the vanadium slag particles became coarse and porous, indicating that the vanadium slag particles were strongly attacked by NaOH solutions, forming fine iron oxide particles.

3.8 Kinetics analysis

The shrinking core model has been proven to be suitable for studying the kinetics of the decomposition of vanadium slag in concentrated alkali solutions (Wang et al., 2012; Liu et al., 2013; Liu et al., 2013; Liu et al., 2017). In order to confirm the rate determine step of the whole decomposition process of the vanadium slag, three established kinetic models (Liu et al., 2013; Zhang et al., 2010) were usually used and expressed as the following equations (Liu et al., 2013):

- liquid boundary layer diffusion control:

$$X = kt, \quad (4)$$

- solid product layer diffusion:

$$1 - 2(1 - X) - 3(1 - X)^{2/3} = kt, \quad (5)$$

- surface reaction control:

$$1 - (1 - X)^{1/3} = kt, \quad (6)$$

where X is the conversion of vanadium or chromium, k is the overall rate constant.

To reveal the controlling step of the vanadium extraction, the conversion data of vanadium at 433 K in Fig. 7 were used for fitting according to Eqs. (4), (5), and (6), and the results are shown in Fig. 10.

The result shows that Eq. (5) fits the experimental data perfectly with R^2 of 0.997. Therefore, it is clear that the internal diffusion step is the controlling step for the vanadium oxidation process.

The leaching rate of vanadium at various temperatures was fitted with time using Eq. (5), and the results are shown in Fig. 11.

From Fig. 11 (left), the reaction rate constant, which is the slopes of the straight lines, can be obtained. Then the apparent activation energy can be calculated by Arrhenius equation, as shown in Fig. 11 (right).

$$\left(\ln k = \ln A - \frac{E_a}{R} \cdot \frac{1}{T} \right), \quad (7)$$

where k is the kinetics constant, E_a is the apparent activation energy, A is the pre-exponential factor, R is the universal gas constant and T is the Kelvin temperature.

It is known from Fig. 11, the apparent activation energy for vanadium extraction was calculated to be 26.22 kJ/mol, and the vanadium extraction kinetics equation could be expressed by Eq. (8)

$$1 - 3(1 - X)^{2/3} + 2(1 - X) = 2.99e^{-\frac{26220}{RT}t}. \quad (8)$$

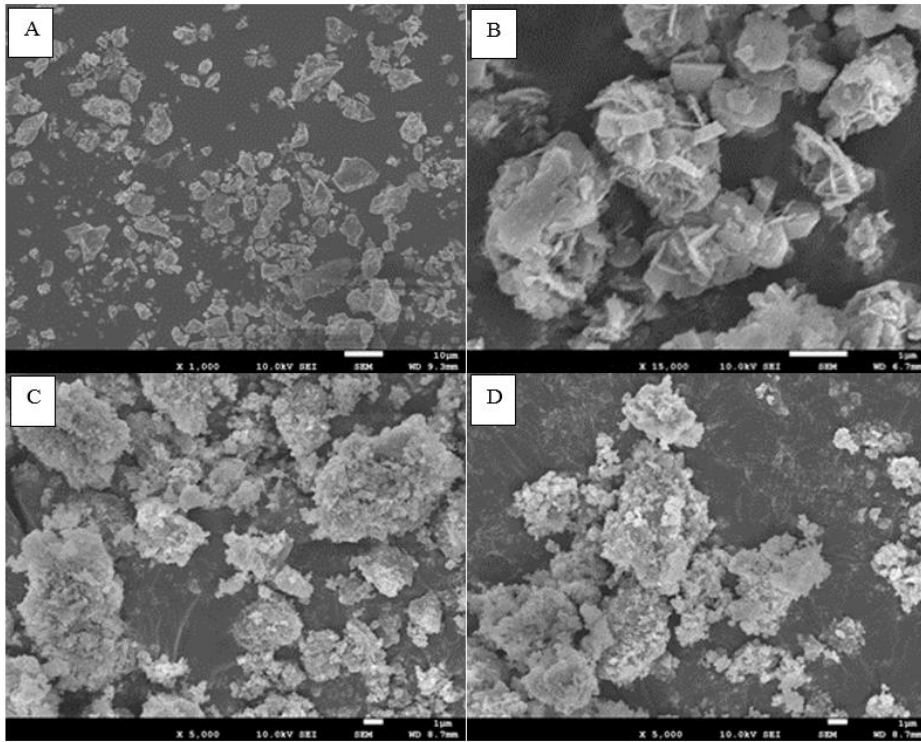


Fig. 9. SEM images of leaching residue at different conversion time ((A) 0 min; (B) 7 min; (C) 15 min; (D) 180 min)

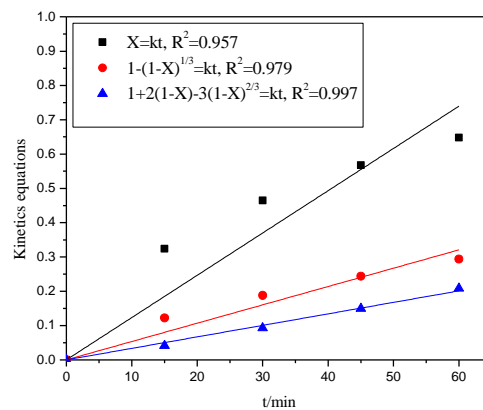


Fig. 10. Vanadium extraction ratio versus time at 433 K fitted by three kinds of kinetics equations

Similarly, to reveal the controlling step of the chromium extraction, the conversion data of chromium at 433 K were also fitted according to Eqs. (4), (5) and (6), as shown in Fig. 12.

The results show that Eq. (5) also gave very good fitting result, with a correlation coefficient of 0.999. The internal diffusion step is also the controlling step for the chromium extraction process.

The relationships between the conversion and the time for chromium at different temperatures were fitted by Eqs. (4), (5) and (6), and the results are shown in Fig.13 (left). Based on the calculated rate constant k at different temperatures, the relationship of $\ln k$ and $1000/T$ was plotted in Fig. 13 (right). The apparent activation energy was calculated to be 32.79 kJ/mol. The kinetic equation for chromium extraction can be expressed as:

$$1 - 3(1-X)^{2/3} + 2(1-X) = 43.42e^{-\frac{32790}{RT}} \quad (9)$$

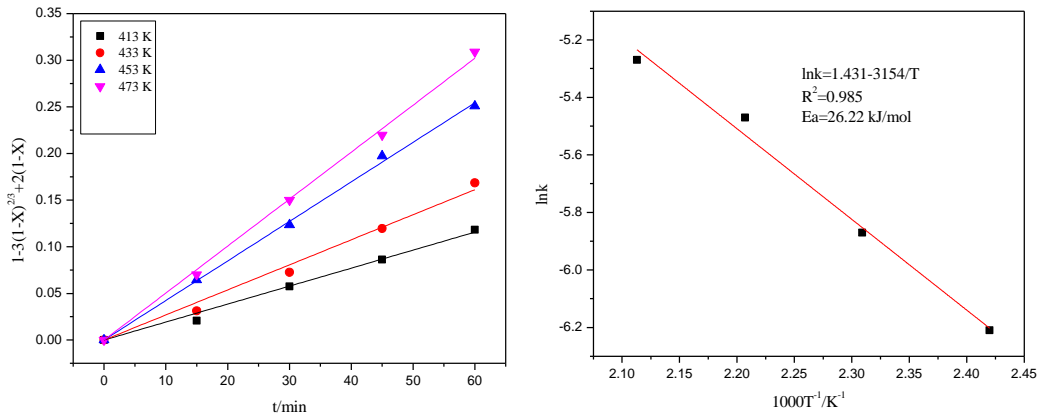


Fig. 11. Plot of leaching kinetics of vanadium under various reaction temperature (left) and natural logarithm of reaction rate constant versus reciprocal temperature of vanadium (right)

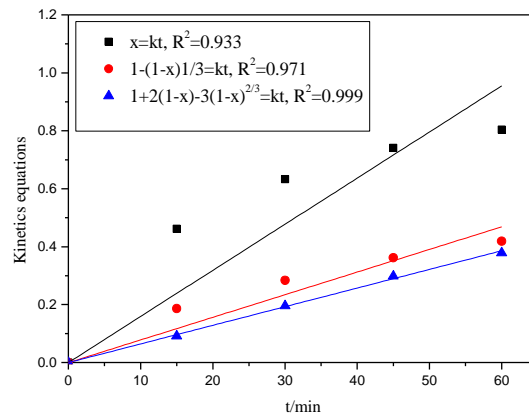


Fig. 12. Chromium extraction ratio versus time at 433 K fitted by three kinds of kinetics equations

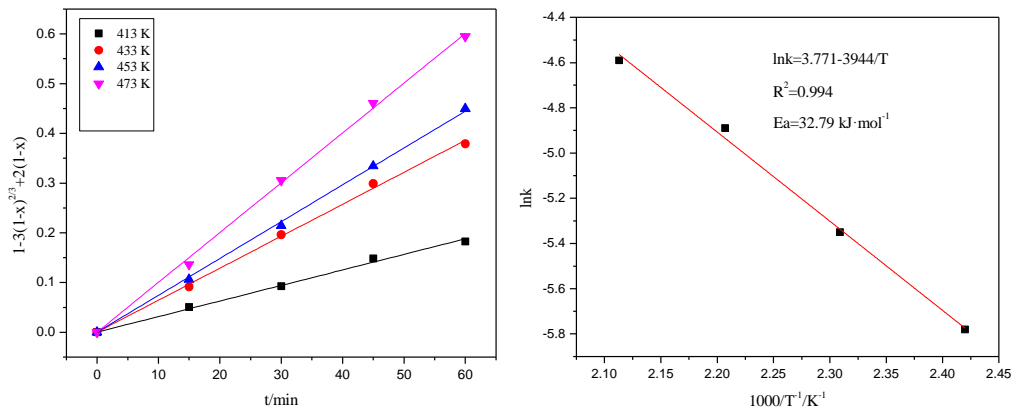


Fig. 13. Plot of leaching kinetics of chromium under various reaction temperature (left) and natural logarithm of reaction rate constant versus reciprocal temperature of chromium (right)

4. Conclusions

- 1) Under the optimal conditions of temperature 473 K, liquid-to-solid mass ratio 6:1, stirring speed 700 rpm, NaOH mass concentration 50%, reaction time 180 min and pressure 1 MPa, vanadium and chromium conversions could reach 95% and 90%, respectively.
- 2) Increasing temperature, stirring speed, pressure and liquid-to-solid mass ratio was beneficial for the extraction of vanadium and chromium.
- 3) XRD analysis showed that vanadium and chromium spinels were difficult to be decomposed and oxidized after the decomposition of fayalite and quartz.
- 4) The kinetics of the decomposition of vanadium slag in concentrated NaOH solutions with low pressure can be described using the shrinking core model. The rate controlling steps for vanadium and chromium extraction are both the internal diffusion step, with apparent activation energies calculated to be 26.22 and 32.79 kJ/mol, respectively.

Acknowledgements

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