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Source separation of V and Fe by two-stage selective leaching during V extraction from stone coal

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A method of two stage selective leaching for separation of V and Fe during V extraction from stone coal was developed. In the primary procedure stone coal was roasted in an inert atmosphere at 900 °C for 1 h, and then the roasted product was leached for Fe removal in 7% (vol%) H_2SO_4 at 30 °C for 30 min when the liquid-to-solid ratio (L/S) was 3 : 1 mL g^{-1}. The Fe removal residue was then leached under the conditions of 20% (vol%) H_2SO_4, L/S = 2 : 1 mL g^{-1}, at 95 °C for 4 h. In the first stage, 98.1% Fe could be removed from the roasted product with only 2.0% V loss. In the second stage, a 97.8% V leaching efficiency was obtained, which was 10.4% higher than the leaching efficiency of the roasted product using one-stage leaching. Based on the different solubilities in acid of muscovite and FeS, the decomposition product of pyrite at high temperature in an inert atmosphere, this method admirably prevented Fe from entering the V leaching liquor, realizing source separation of V and Fe during V extraction from stone coal.

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1 Introduction

Stone coal, a type of V-bearing black shale, is an important vanadium resource in China due to its vast deposit amounts.[1–3] Most of the V in stone coal exists as V(III) because of the reducing environment where stone coal forms, which readily replaces Al(III) as an isomorphism in structures of mica group minerals.[1,4–6] Hence, the V in stone coal is stable and difficult to extract. At present, sulfuric acid leaching after high temperature roasting is widely used due to its good adaptability to raw materials, high efficiency and environmental characteristics.[7–10] However, in the sulfuric acid leaching process, many other impurities such as Fe, Al, Mg, and P are always leached into the acid leaching liquor together with V. These impurities are detrimental to the recovery of vanadium from the acid leaching liquor.[11,12] Particularly detrimental is Fe, a type of valence variable ion that mostly comes from the pyrite in stone coal, as it will seriously affect the enrichment process of V from the V leaching liquor and the final V-bearing products.[8,13,14]

In most cases, V and Fe are separated from the acid leaching liquor by ion exchange, solvent extraction and chemical precipitation.[11,15–19] The ion exchange process is usually used for V leaching liquors with a low impurity concentration and a low acidity.[20–22] Solvent extraction has higher selectivity and adaptability for high acidity leaching liquor with a high impurity concentration. D2EHPA/TBP is the most popular extraction agent in V extraction from acid solution. It can effectively separate Fe and V when Fe is reduced into Fe(II).[23,24] However, even with the use of an extraction agent with a high separation coefficient for V and Fe such as D2EHPA/TBP, Fe will still be co-extracted with V from the leaching liquor to some extent, thus affecting the quality of V-bearing products.[23,25] Ye *et al.* developed another method for the separation of V and Fe, which primarily includes V co-precipitation with Fe in the liquor, V extraction by alkaline leaching from the precipitate, and V pentoxide preparation from the alkaline leaching liquor.[13] High purity V-bearing product can be obtained, but it is a relatively long process with considerable V loss.

Based on the present research, it is clear that the separation efficiency of V and Fe is affected by the amount of Fe leached into the V leaching liquor. A lower amount of Fe in the V leaching liquor is clearly better for separating and purifying the V leaching liquor. Therefore, in this paper, a method detailing a two-stage selective leaching process for the separation of V and Fe was developed. This method realized source control of the Fe impurity during V extraction from stone coal.

2 Experimental section

2.1 Materials and thermodynamic analysis

The V-bearing stone coal used in this study was obtained from Tongshan, Hubei province, China. The raw sample was

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crushed to 0–3 mm size using a jaw crusher (XPC-60 × 100) and a double-roll crusher (HLXPS-φ 250 × 150). To avoid the interference caused by sintering of fine particles, 1 to 3 mm stone coal particles were screened as the raw sample. The chemical composition analysis performed with ICP-AES showed that there was 0.37% V, 3.15% Fe and 3.40% S. Fig. 1 shows the XRD analysis of the raw sample conducted using a D/MAX-RB X-ray diffractometer. The main S-bearing mineral was pyrite, and the Fe in pyrite accounted for more than 95% in raw sample, as calculated from the S content.

The electron probe analysis (EPMA) of the raw sample was performed with a JXA-8230 electron probe micro-analyzer, and the results are shown in Table 1. Most of the V in the raw sample was in mica group minerals.

As analyzed above, most of the Fe in the raw sample was from pyrite and most of the V was in mica group minerals. Hence, the main reactions of pyrite and mica group minerals during the roasting process and the acid leaching process should be considered. When pyrite is roasted in an oxidizing atmosphere and in an inert atmosphere, it transforms into Fe_2O_3 (eqn (1)) and Fe monosulfide (FeS) (eqn (2)), respectively.

$$4FeS_2 + 11O_2 \rightarrow 2Fe_2O_3 + 8SO_2\uparrow \quad (1)$$

$$2FeS_2 \rightarrow 2FeS + S_2 \quad (2)$$

During the acid leaching process, the main reactions of acid with Fe-bearing minerals (Fe_2O_3 or FeS) and mica (in the form of muscovite, $KAl_2(AlSi_3O_{10})(OH)_2$) could be expressed as eqn (3)–(5),[26] respectively.

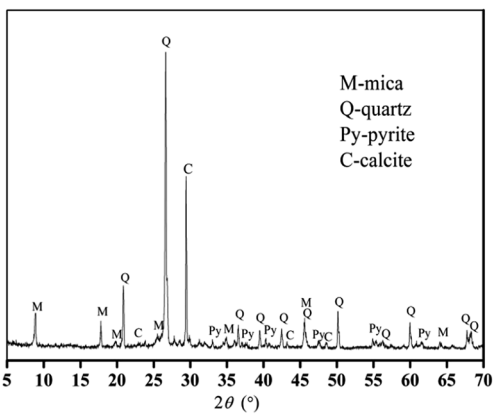

Fig. 1 XRD analysis of raw sample.

Table 2 Relations of pH_T^θ *versus* T of eqn (3)–(5)

Equations	pH_T^θ–T
(3)	$pH_T^\theta = -4.015 \times 10^{-7}T^2 + 1125.3/T - 3.5 \times 10^{-3}$
(4)	$pH_T^\theta = -2.0517 \times 10^{-6}T^2 + 0.1046/T + 2.27 \times 10^{-3}$
(5)	$pH_T^\theta = -2.88 \times 10^{-7}T^2 + 1.4886/T - 3.16 \times 10^{-3}$

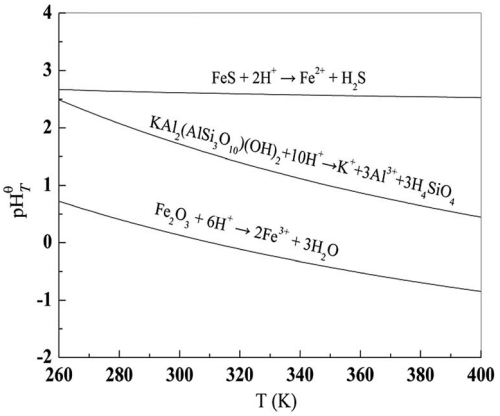

Fig. 2 Plots of pH_T^θ *versus* T of eqn (3)–(5).

$$Fe_2O_3 + 6H^+ \rightarrow 2Fe^{3+} + 3H_2O \quad (3)$$

$$FeS + 2H^+ \rightarrow Fe^{2+} + H_2S\uparrow \quad (4)$$

$$KAl_2(AlSi_3O_{10})(OH)_2 + 10H^+ \rightarrow K^+ + 3Al^{3+} + 3H_4SiO_4 \quad (5)$$

Eqn (3)–(5) can be expressed as the following general formula:[27]

$$aA + nH^+ = bB + cH_2O \quad (6)$$

The Gibbs free energy at a particular T (in kelvin) (ΔG_T) of eqn (6) could be expressed as eqn (7):

$$\Delta G_T = \Delta G_T^\theta + RT\ln[\alpha_A{}^b/\alpha_B{}^a] + 2.303nRTpH_T \quad (7)$$

When the reaction reaches equilibrium, $\Delta G_T = 0$:

$$pH_T = \frac{-\Delta G_T^\theta}{2.303nRT} - \frac{1}{n}\lg\frac{\alpha_A{}^b}{\alpha_B{}^a} \quad (8)$$

Table 1 EPMA result of the raw sample (wt%)

SiO_2	V_2O_5	Al_2O_3	FeO	MgO	CaO	Na_2O	K_2O	Minerals
0.062	0.000	0.000	59.122	0.000	0.000	0.000	0.000	Pyrite
0.000	0.000	0.000	0.000	1.101	65.939	0.000	0.000	Calcite
98.349	0.000	0.000	0.000	0.000	0.000	0.000	0.000	Quartz
51.075	3.475	27.224	0.225	4.531	0.017	0.065	9.521	Muscovite
39.948	4.135	23.250	0.138	2.020	0.148	0.080	8.550	Illite
40.781	1.313	17.632	1.358	21.91	0.017	0.018	8.477	Biotite

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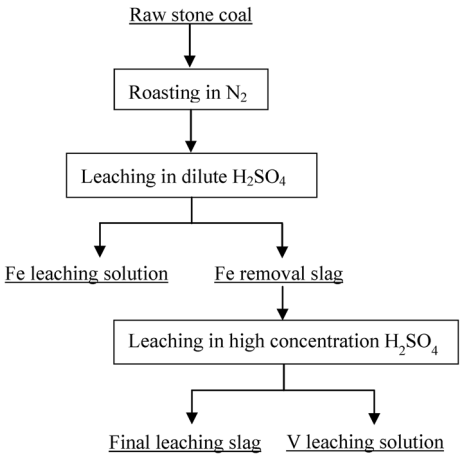

Fig. 3 Flow sheet of two stages selective leaching for separation of V and Fe.

Assuming $\alpha_A = \alpha_B = 1$, then pH_T can be defined as pH_T^θ (the pH value of the reaction system when the reaction reaches equilibrium under standard condition), which can be expressed as eqn (9):

$$pH_T^\theta = \frac{-\Delta G_T^\theta}{2.303nRT} \quad (9)$$

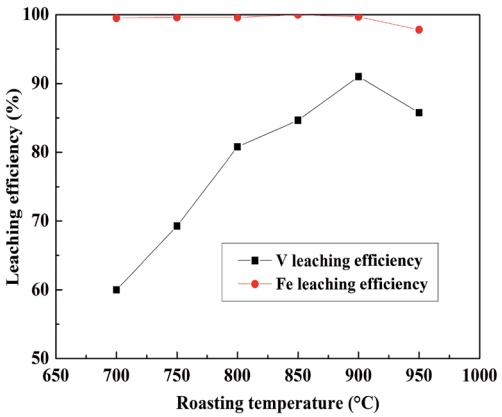

Fig. 4 Effect of roasting temperature on V and Fe leaching efficiencies.

where ΔG_T^θ (J) is the standard Gibbs free energy, R (J mol^{-1} K^{-1}) is the standard molar gas constant (8.314 J mol^{-1} K^{-1}), n is the stoichiometric number of H^+, and T (K) is temperature.

pH_T^θ represents the pH value of the reaction system when the reaction reaches equilibrium. Hence, it can be used to determine the solubility of the reactant; when the reaction has a higher pH_T^θ value, the reactant is easier to dissolve in an acid solution.[28,29] Based on the thermodynamic data,[30] the relation of pH_T^θ *versus* T of these equations is shown in Table 2, and the plot is displayed in Fig. 2. According to the pH_T^θ value, the solubility order of Fe_2O_3, FeS and $KAl_2(AlSi_3O_{10})(OH)_2$ in acid is FeS > $KAl_2(AlSi_3O_{10})(OH)_2$ > Fe_2O_3. Theoretically, both FeS and Fe_2O_3 can undergo stepwise dissociation with muscovite by dilute acid and concentrated acid. However, it is well known that muscovite dissolves quite slowly in dilute acidic solution which results in harsh leaching condition for V leaching from stone coal.[31,32] In highly concentrated acid (with leaching extract ratio), Fe_2O_3 and muscovite can dissolve resulting in massive Fe impurity in the V leaching solution.[8,9] Contrarily, due to the excellent solubility of FeS in acidic solutions, a dilute acid solution is always used to clean the FeS scale on machinery in the oil industry.[33,34] Therefore, because of the quite different solubilities in acid, FeS and muscovite might be efficiently separated using a dilute acidic solution in theory.

2.2 Procedure

Based on the thermodynamic analysis of the raw sample, the procedure of the two-stage selective leaching for separation of vanadium and Fe in stone coal is shown in Fig. 3. First, 200 g of raw stone coal was roasted with an SGL-1700 pipe furnace equipped with an HQZ-IV gas distribution system in N_2. The roasted products were ground in a vibration mill (HLXZM-100) until at least 75% of the particles were −0.074 mm. Then, 20 g roasted product was leached in a dilute sulfuric acid solution to remove the Fe in the roasted product with an SZCL-2A type magnetic and controlling temperature stirrer. The leaching pulp was solid–liquid separated, and the Fe removal residue was then leached in high concentration sulfuric acid solution to leach V.

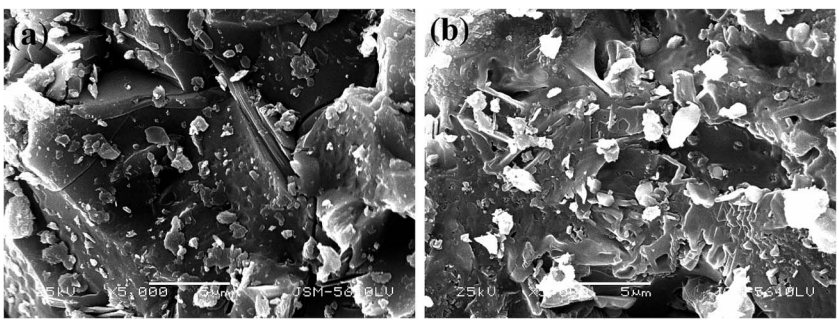

Fig. 5 SEM pictures of the roasted products at 900 °C and 950 °C ((a) 900 °C; (b) 950 °C).

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2.3 Testing method

SEM and SEM-EDS analyses of roasted product were conducted with a PHILIPS XL30 TMP Scanning Electron Microscope equipped with an EDAX PHOENIX energy spectrum analyzer.

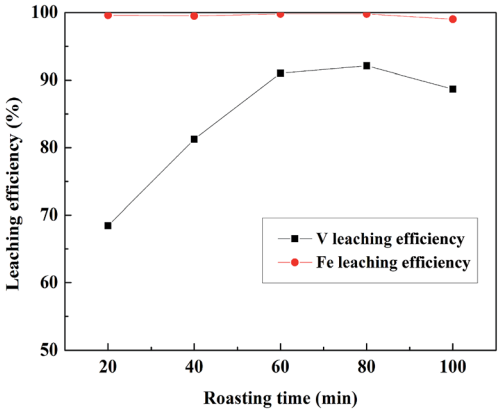


Fig. 6 Effect of roasting time on V and Fe leaching efficiencies.

XRD analysis of the samples was performed with a D/MAX-RB X-ray diffractometer.

The Fe content in the Fe leaching liquor was determined by a phenanthroline spectrophotometric method,[35] and the Fe removal efficiency of the roasted product was calculated using eqn (10):

$$x_{Fe} = \frac{C_{Fel} \times V_l}{C_{Fes} \times M_s} \quad (10)$$

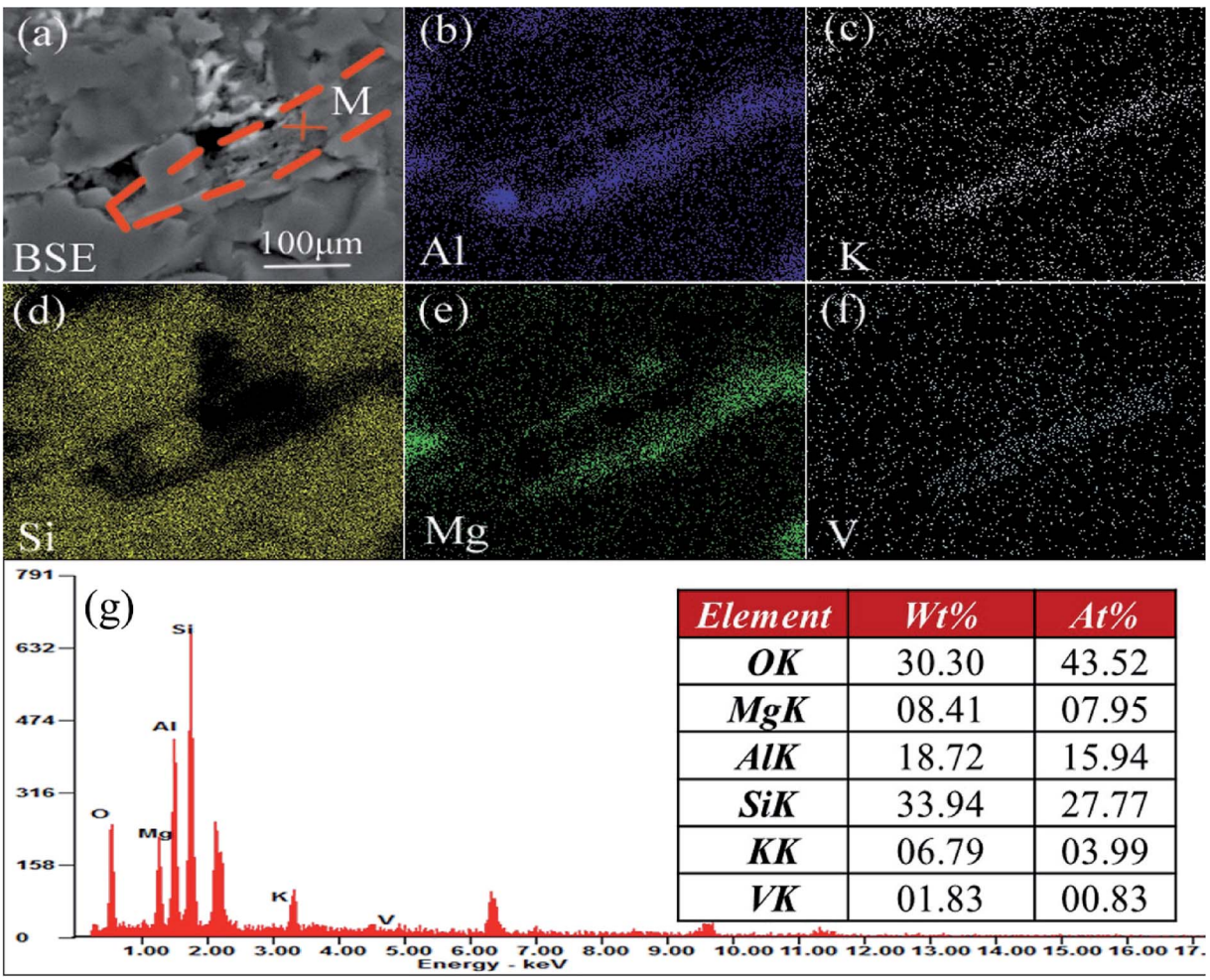
where C_{Fel} is the Fe content of the Fe leaching liquor, V_l is the volume of the Fe leaching liquor, C_{Fes} is the Fe content of the roasted product, M_s is the mass of the roasted product.

The V content in the Fe leaching liquor was determined by ammonium ferrous sulfate method,[36] and the V loss efficiency was calculated using eqn (11):

$$x_V = \frac{C_{Vl} \times V_l}{C_{Vs} \times M_s} \quad (11)$$

where C_{Vl} is the V content of Fe in the leaching liquor, V_l is the volume of Fe in the leaching liquor, C_{Vs} is the V content of the roasted product, and M_s is the mass of the roasted product.

The V leaching efficiency was calculated using eqn (12):



Element	Wt%	At%
OK	30.30	43.52
MgK	08.41	07.95
AlK	18.72	15.94
SiK	33.94	27.77
KK	06.79	03.99
VK	01.83	00.83

Fig. 7 BSE image and EDS analysis of roasted product in N_2 at 900 °C ((a) BSE image of roasted product; (b), (c), (d), (e), and (f) EDS elemental distribution of Al, K, Si, Mg, and V; (g) EDS spectra of point marked as "M" collected form BSE image).

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$$\alpha_V = \frac{C_{Vl'} \times V_{l'}}{C_{Vs'} \times M_{s'}} \quad (12)$$

where $C_{Vl'}$ is the V content of the V leaching liquor, $V_{l'}$ is the volume of the V leaching liquor, $C_{Vs'}$ is the V content of the Fe removal residue, and $M_{s'}$ is the mass of the Fe removal residue.

3 Results and discussion

3.1 Roasting experiment

3.1.1 Effect of roasting temperature. Appropriate roasting condition should satisfy the V leaching efficiency of the roasted product and ensure the Fe in the roasted product exists in the form of FeS. That means under the appropriate roasting condition, the V and Fe leaching efficiencies should be as high as possible. The raw sample was roasted in N_2 for 1 h at 700 °C, 750 °C, 800 °C, 850 °C, 900 °C and 950 °C. The roasted products were leached under the leaching condition of 20% (vol%) H_2SO_4, L/S (liquid/slag) = 2 : 1 mL g^{-1}, 95 °C, 4 h, and the results are shown in Fig. 4. V leaching efficiencies of the roasted products increased with an increase in the roasting temperature, and above 900 °C, the decrease in the V leaching efficiency resulted from a sintering phenomenon.[9,37] The SEM pictures of the roasted samples at 900 °C and 950 °C were showed in Fig. 5. It is obviously that the roasted product at 900 °C still had clear contour but it begin to melting at 950 °C. As for Fe, it had quite high efficiencies when the temperature form 700 °C to 900 °C, there was a slight decline after 900 °C which may also resulted by the sintering phenomenon. Hence, as the V and Fe efficiencies were considered, 900 °C was selected as a suitable roasting temperature.

3.1.2 Effect of roasting time. To investigate the effect of the roasting time on the V and Fe leaching efficiencies, the raw sample was roasted at 900 °C for 20 min, 40 min, 60 min, 80 min, and 100 min, and the roasted products were then leached under the leaching condition of 20% (vol%) H_2SO_4, L/S (liquid/slag) = 2 : 1 mL g^{-1}, 95 °C, 4 h; the results were shown in Fig. 6. Before 60 min, the V leaching efficiency increased with the increase in roasting time. When the roasting time exceeded 60 min, the V leaching efficiency no longer increased, and it decreased after 80 min. There were high Fe leaching efficiencies when the roasting time was form 20 to 80 min and there was a slight decline when the roasting time was 100 min. Therefore, 60 min was a suitable time for roasting based on the V and Fe leaching efficiencies.

3.1.3 Characteristic of roasted sample. Fig. 7 shows the BSE image of the product roasted at 900 °C for 1 h and the EDS analyses of elemental distribution. The EDS spectra of point "M" (g) indicated that the elemental composition of the microscopic area marked with the red dotted line was similar to that of mica group minerals. Then, elemental distribution analysis was conducted. It can be seen clearly that in the marked area, the characteristic elements of mica group minerals, such as Al, K, Si, Mg and V, had a high degree of correlation. High correlation of V with these characteristic elements indicated that vanadium in stone coal still exists in mica group minerals after roasting, and it might not be leached easily by dilute acid solutions. According to the XRD analysis of the raw sample and the roasted sample shown in Fig. 8, the Fe-bearing mineral had totally transformed from FeS_2 to FeS. Therefore, based on the V leaching efficiency and the characteristics of the roasted product, 900 °C for 1 h was selected as the appropriate roasting condition. The roasted product under this condition might be suitable for two-stage selective leaching for separation of V and Fe.

3.2 Fe removal of the roasted product

3.2.1 Effect of H_2SO_4 concentration. To investigate the effect of the H_2SO_4 concentration on Fe removal efficiency and V loss efficiency, the roasted product was leached in 3%, 4%, 5%, 6%, 7% and 8% (vol%) H_2SO_4, with L/S = 3 : 1 mL g^{-1}, a leaching temperature of 30 °C, and a leaching time of 30 min, and the result is shown in Fig. 9. The Fe removal efficiency and V loss efficiency increased with the increase in the H_2SO_4 concentration until 7% (vol%). After 7% (vol%), the Fe removal efficiency tended to be stable, but the V loss efficiency showed an obvious increase. Therefore, 7% (vol%) was selected as the suitable H_2SO_4 concentration.

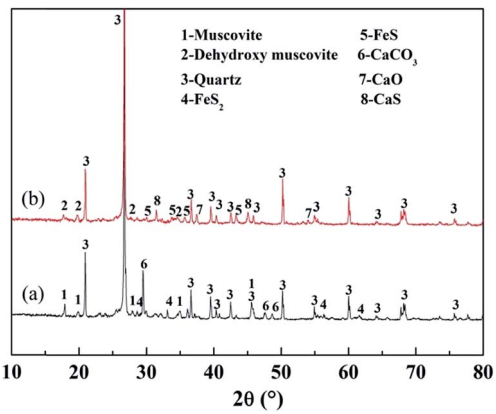

Fig. 8 XRD analysis of raw sample and roasted product ((a) raw sample; (b) roasted product).

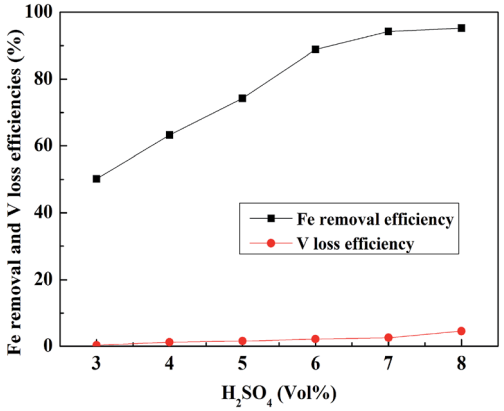

Fig. 9 Effect of H_2SO_4 concentration on Fe removal and V loss efficiencies.

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3.2.2 Effect of leaching temperature. To clarify the effect of temperature on the Fe removal efficiency and the vanadium loss efficiency, the roasted product was leached at 20 °C, 30 °C, 40 °C and 50 °C, H_2SO_4 was 7% (vol%), L/S = 3 : 1 mL g^{-1}, and the leaching time was 30 min and the result is shown in Fig. 10. The Fe removal efficiency increased with an increase in temperature until 30 °C. When the temperature exceeded 30 °C, the Fe removal efficiency was more than 98% and was stable, but the V leaching efficiency increased faster. Hence, 30 °C was selected as the appropriate leaching temperature.

3.2.3 Effect of leaching time. The roasted product was leached for 10 min, 20 min, 30 min, 60 min, 90 min, 120 min and 150 min, the H_2SO_4 concentration was 7% (vol%), L/S = 3 : 1 mL g^{-1}, and the temperature was 30 °C; the results are displayed in Fig. 11. When the leaching time was less than 30 min, the Fe removal efficiency quickly increased with the increase in time. After 30 min, it was more than 98% and remained stable and would hardly increase. However, the loss efficiency of vanadium showed a steady growth with the increase in leaching time. Therefore, the suitable leaching time for Fe removal was selected as 30 min.

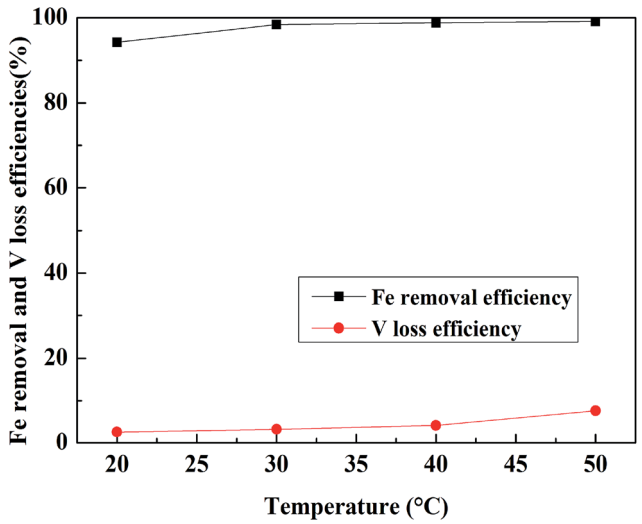

Fig. 10 Effect of temperature on Fe removal and V loss efficiencies.

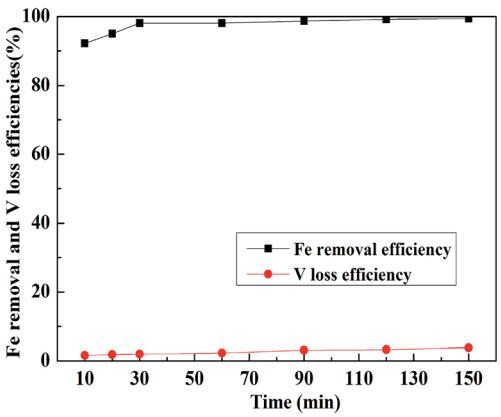

Fig. 11 Effect of time on Fe removal and V loss efficiencies.

According to the results of the Fe removal experiment for the roasted product, when the conditions were 7% (vol%) H_2SO_4, temperature of 30 °C, time of 30 min and L/S = 3 : 1 mL g^{-1}, 98.1% Fe removal efficiency with only 2.0% V loss was achieved.

3.3 V leaching of Fe removal residue

3.3.1 Effect of temperature. To investigate the effect of temperature on the V and Fe leaching efficiencies, the Fe removal residue was leached at 75 °C, 80 °C, 85 °C, 90 °C, 95 °C and 100 °C, H_2SO_4 was 20% (vol%), L/S = 2 : 1 mL g^{-1}, and the leaching time was 4 h, and the result is presented in Fig. 12. The V leaching efficiency increased with an increase in temperature until 95 °C. When the leaching temperature continually increased from 95 °C to 100 °C, the V leaching efficiency increased from 97.8% to 98.6%, which was less than 1%. Because in Fe removal residue, most of the Fe had been removed, the Fe leaching efficiencies of the Fe removal slag were always low. Based on the V leaching efficiency, 95 °C was selected as the suitable leaching temperature.

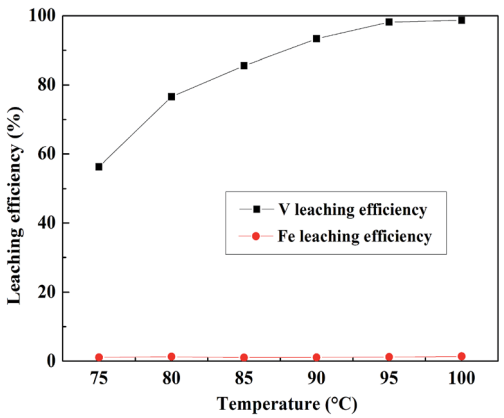

Fig. 12 Effect of temperature on V and Fe leaching efficiency of Fe removal residue.

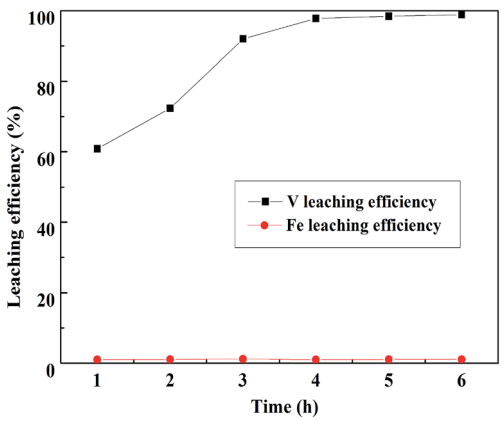

Fig. 13 Effect of time on V and Fe leaching efficiency of Fe removal residue.

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3.3.2 Effect of leaching time. The Fe removal residue was leached for 1 h, 2 h, 3 h, 4 h, 5 h and 6 h, the H_2SO_4 concentration was 720% (vol%), L/S = 2 : 1 mL g^{-1}, and the temperature was 95 °C; the result is displayed in Fig. 13. The V leaching efficiency increased with the increase in leaching time until 4 h, there was no obvious improvement after 4 h. Therefore, 4 h was selected as the optimum leaching time of Fe removal residue.

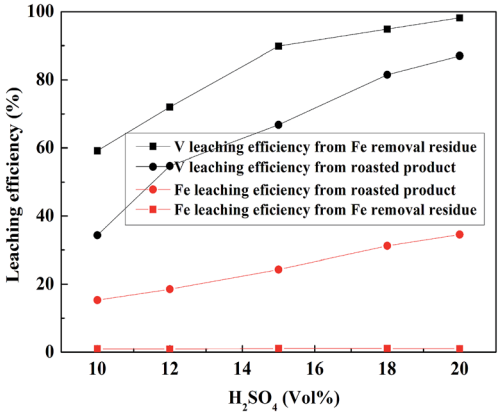

Fig. 14 Effect of H_2SO_4 concentration on V leaching efficiency of Fe removal residue and roasted product.

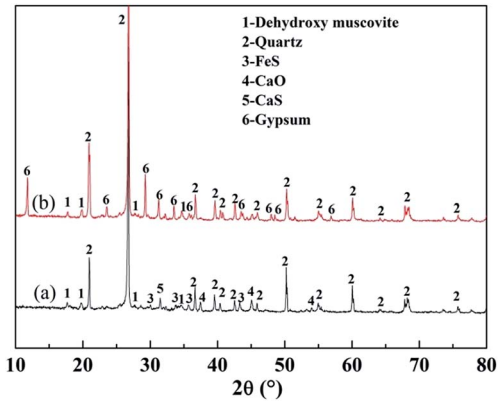

Fig. 15 XRD analyses of roasted product and Fe removal residue ((a) roasted product; (b) Fe removal residue).

3.3.3 Effect of H_2SO_4 concentration. The Fe removal residue and the roasted product were leached under the same conditions to compare the V and Fe leaching characteristic. Thirty grams of Fe removal residue and roasted product was leached under conditions of 10%, 12% 15%, 18% and 20% (vol%) H_2SO_4, L/S = 2 : 1 mL g^{-1}, leaching temperature of 95 °C and leaching time of 4 h; the result is shown in Fig. 14. Under the same leaching conditions, the V leaching efficiency of Fe removal residue was much higher than that of the roasted product. This means that after the first stage of leaching to remove Fe, a considerable amount of sulfuric acid could be saved during the V leaching stage of the Fe removal residue. Thus, 20% (vol%) H_2SO_4 was selected as the appropriate acid concentration to obtain 97.8% V leaching efficiency, and it was 10.4% higher than the leaching efficiency of the roasted product using one-stage leaching method.

Fig. 15 shows the XRD analyses of the roasted product and the Fe removal residue. During the Fe leaching process, FeS and CaO in the roasted product, which are acid-consuming materials, had been mostly removed. Hence, more sulfuric acid could be used to dissolve the V-bearing mica group minerals, thus improving the V leaching efficiency. The SEM images shown in Fig. 16 indicated that there were more pores in the Fe removal residue than in the roasted product. These pores mainly resulted from dissolution of acid-consuming materials in the first stage of leaching, and they enhance the reactions of sulfuric acid with V-bearing minerals.

Based on the Fe removal efficiency and V leaching efficiency under this experimental condition, when the raw sample was roasted at 900 °C for 1 h and a similar V leaching efficiency was obtained (approximately 80%), the Fe and V contents of the final V leaching liquor from four different extraction methods could be compared as shown in Table 3 (the ratio of the final V leaching liquor volume to the solid sample mass was assumed to be 2 : 1 mL g^{-1}).

Compared with one-stage leaching or two-stage leaching after roasting in air, two stages selective leaching after roasting in an inert atmosphere significantly decreased the Fe content in the final V leaching liquor. This method prevented Fe from entering the V leaching liquor which was the most obvious difference from the conventional methods. This method will greatly enhance the purification and enrichment of V leaching liquors.

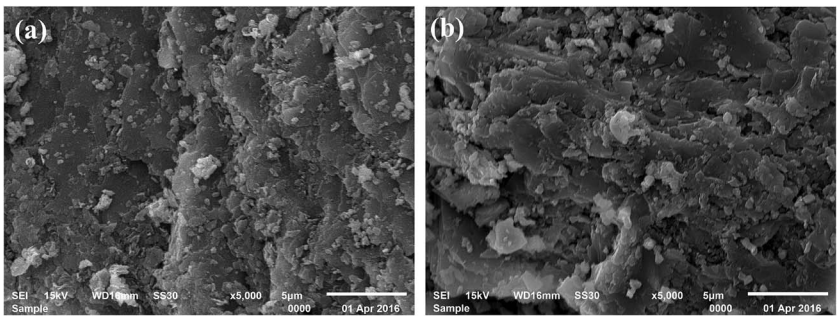

Fig. 16 SEM pictures of Fe removal slag and roasted product ((a) roasted product; (b) Fe removal residue).

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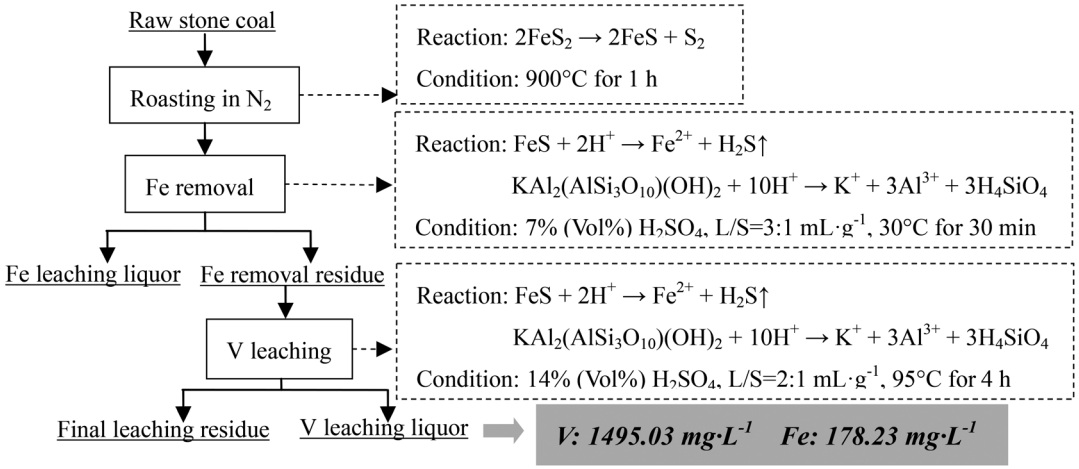
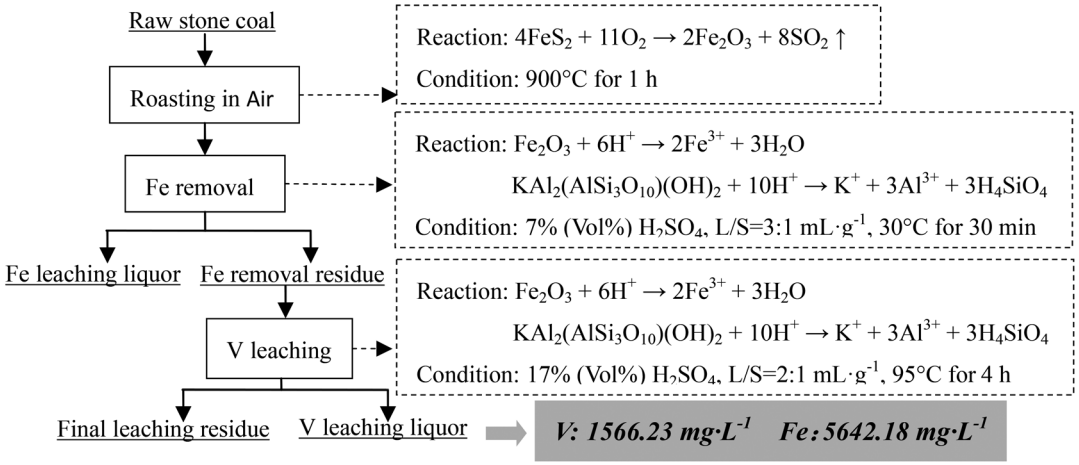
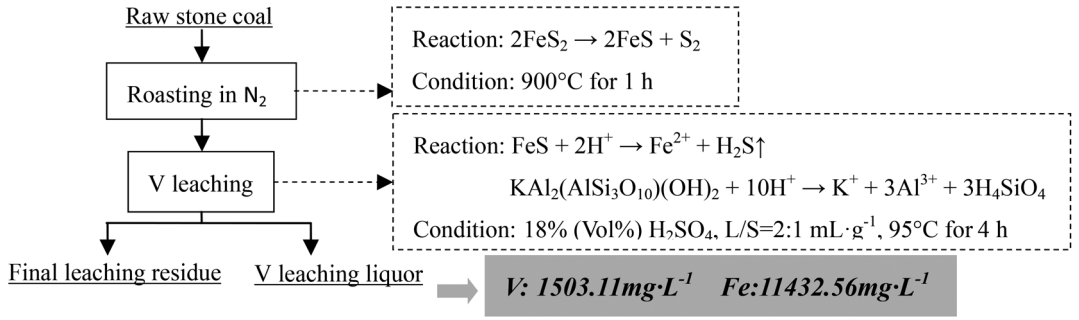
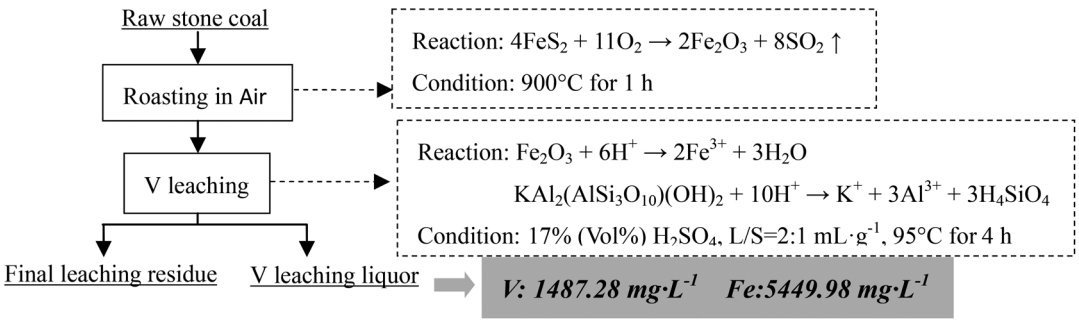
Table 3 Comparison of four different V extraction methods from stone coal

Extraction methods	Principle procedures
Two stages selective leaching after roasting in N_2	
Two stages leaching after roasting in air	
One stage leaching after roasting in N_2	
One stage leaching after roasting in air	

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4 Conclusions

As for the mica-type V-bearing stone coal in which most of the Fe was in the form of pyrite, 98.1% Fe removal efficiency, 2.0% vanadium loss efficiency and 97.8% V leaching efficiency could be achieved by a two-stage selective leaching. The appropriate procedure was that stone coal was roasted in an inert atmosphere at 900 °C for 1 h; the roasted product was leached in the first stage under conditions of 7% (vol%) H_2SO_4, L/S = 3 : 1 mL g^{-1}, 30 °C for 30 min; and then the residue was leached under conditions of 20% (vol%) H_2SO_4, L/S = 2 : 1 mL g^{-1}, 95 °C for 4 h. In the first stage, Fe was efficiently removed from the roasted product with a minimal loss of vanadium. In the second stage, the V leaching efficiency of Fe removal residue was significantly enhanced due to the removal of acid-consuming materials in the previous residue. This method admirably prevented Fe from entering the V leaching liquor realizing source separation of V and Fe during V extraction from stone coal.

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