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Selective extraction of vanadium from vanadium-titanium magnetite concentrates by non-salt roasting of pellets-H₂SO₄ leaching process

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Abstract: In this work, a novel process of pellet non-salt roasting and H₂SO₄ leaching was proposed for the selective extraction of vanadium from vanadium-titanium magnetite concentrate. Vanadium can be leached but the iron impurity was maintained in the pellets. Moreover, the leached pellets can meet the quality requirements of the iron-making process after secondary roasting, realizing comprehensive utilization. The maximal vanadium leaching efficiency was up to 60.3%, whereas 0.17% of the iron impurity was leached. The optimum conditions of pellet roasting and leaching were obtained by single-factor experiments. The X-ray diffraction and scanning electron microscopy-energy disperse X-ray spectrometry analyses showed that the vanadium iron spinel can be oxidized and decomposed into Fe₂O₃ and vanadate during the roasting process. Given that dilute sulfuric acid can react with vanadate without reacting with Fe₂O₃ in the leaching process, selective vanadium extraction was realized. This work provides new insights into the industrial production of vanadium-titanium magnetite concentrate involving the direct extraction of vanadium.

Keywords: vanadium-titanium magnetite; pellets; selective extraction; sulfuric acid leaching

1. Introduction

Vanadium is an important strategic resource that is widely used in steel, aviation, chemical industry, and vanadium battery due to its special physical and chemical performances (Mirazimi et al., 2013; Xue et al., 2017; Guo et al., 2020; Liu et al., 2021). In addition to vanadium-bearing stone coal, vanadium-titanium magnetite is a main raw material for vanadium extraction (Gilligan and Nikoloski, 2020). At present, the fundamental process for smelting vanadium-titanium magnetite involves iron-making and extraction of vanadium from vanadium slag by the sodium salt roasting-water leaching process (Chen et al., 2013; Li et al., 2015; Ji et al., 2017; Gilligan and Nikoloski, 2020). However, this method has a low total recovery ratio of vanadium and titanium and produces Cl₂, SO₂, and HCl, causing serious environmental pollution. Therefore, how to use a new environment-friendly method to extract valuable elements (V, Ti, and Fe) comprehensively and efficiently from vanadium-titanium magnetite has attracted widespread attention.

The direct extraction of vanadium from vanadium-titanium magnet concentrate has good industrial prospects due to its top recovery rate of vanadium (Chen et al., 2015; Zhu et al., 2016; Luo et al., 2021). The procedure is mainly divided into non-salt roasting-leaching process, alkaline salt roasting-water leaching process, and calcium salt roasting-leaching process. Li et al. (2017) studied a clear method featuring non-salt roasting and ammonium bicarbonate leaching for the extraction of vanadium. Chen et al. (2013) used NaOH molten salt roasting and water leaching process to extract vanadium, and 96.6% of the vanadium was leached out. Liu et al. (2018) achieved 71.37% vanadium leaching efficiency by potassium salt roasting-leaching process under optimal conditions. In addition, Wang et al. (2011) used calcium carbonate roasting-leaching process to extract vanadium. However, almost all of these methods

have the disadvantages of poor selectivity and high cost, and they ignore the leaching of iron impurities. The iron content in vanadium–titanium magnetite concentrate is 50% or higher. The leaching of vanadium is accompanied by the leaching of a large amount of iron ions, reducing the purity of vanadium products (Hu et al., 2018). Iron seriously affects the extraction process of vanadium from leaching liquor. $\text{Fe}(\text{OH})_3$ affects the process of ion exchange and leads to emulsification in the solvent extraction process (Hu et al., 2017). Moreover, Fe^{3+} hinders the reutilization of extractants because stripping of iron from the organic phase is difficult (Liu et al., 2020). Therefore, solving the problem of the separation of vanadium and iron is the key to the comprehensive utilization of vanadium–titanium magnetite concentrate.

Sulfuric acid has a selective separation effect. Ning et al. (2019) realized the preliminary separation of iron and scandium by using sulfuric acid, with leaching rates of 11.32% and 57.41% respectively; Kul et al. (2015) used sulfuric acid to leach zinc from electric arc furnace steelmaking dust. Under optimized conditions, the leaching rates of zinc and iron are 79.09% and 4.08%, respectively, achieving high selectivity. Therefore, this study intended to use sulfuric acid as a selective leaching agent to separate vanadium and iron from the vanadium–titanium magnetite concentrate.

Given the above problems, this paper proposes a pellet non-salt roasting–leaching process for vanadium extraction. First, the preparation of the concentrate as pellets prevents the powder from sintering and sticking to the wall at high temperatures. In addition, the non-salt roasting process avoids the addition of alkali ions and the generation of exhaust gas, which is environmentally friendly. Second, sulfuric acid leaching can selectively separate vanadium and suppress the leaching of impure iron ions. Moreover, the leached pellets meet the quality requirements of the iron-making process after secondary roasting. This condition is conducive to realize comprehensive utilization and industrial production.

In this study, using vanadium–titanium magnetite concentrate as the research object, the optimal roasting and leaching conditions for extracting vanadium were investigated by single-factor experiments. X-ray diffraction (XRD) and scanning electron microscopy (SEM) were used to study the mechanism of selective extraction of vanadium.

2. Experimental methods

2.1. Materials

The vanadium-titanium magnetite concentrate (which was obtained from Panzhihua, Sichuan Province, China) was ground to less than 0.074 mm, accounting for 70% of the total. Calcium-based bentonite was used as the binder in the pelletizing process. Sulfuric acid was used as the leaching agent.

2.2. Experimental procedure

Fig. 1 shows the vanadium leaching process in this experiment, and the specific operating procedures are as follows.

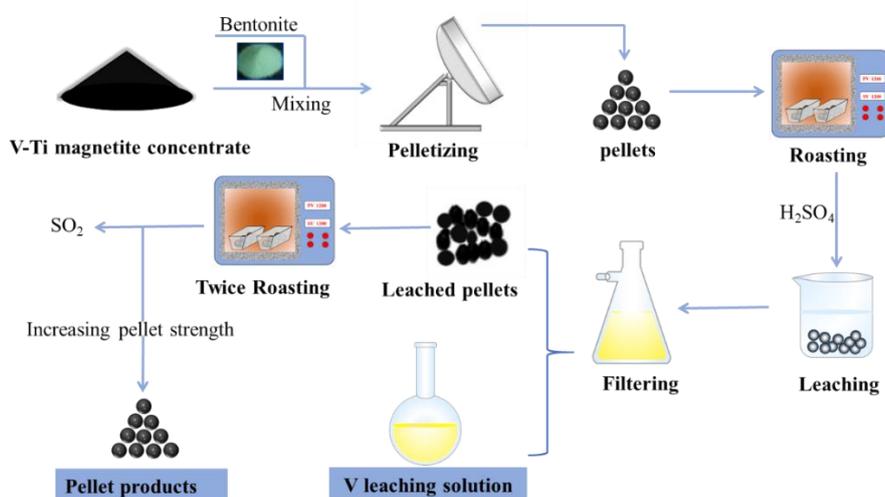


Fig. 1. Process chart of the non-salt roasting-pellet acid leaching

2.2.1. Pelletizing process

For each test, 2.0 kg concentrate and 40 g bentonite (2% of the concentrate) were weighed by electronic balance and mixed in a porcelain plate for 10 min.

The pelletizing process can be divided into three stages, namely, formation, growth, and compaction, and it was carried out in a disc granulator with a diameter of 500 mm and an inclination of 45°. About 100 g mixed ore samples were added to the disc granulator in advance and then sprayed with water to prepare the mother ball with a diameter of 1–2 mm.

Water was sprayed while adding the mineral powder to trigger the adhesion of the mother ball to the mineral powder. The mother ball grew slowly, and wet pellets with a diameter of 8–10 mm were hand-selected. The ball-making time was controlled at 1 h, and the amount of added water was controlled at 10%. Finally, the wet pellets were dried at 70 °C for 12 h to obtain dry pellets.

2.2.2. Non-salt roasting

The dried pellets were roasted in the muffle furnace from room temperature to 1050-1250 °C at the heating rate of 5 °C/min and kept for 1-210 min. After roasting, the roasted pellets were cooled to room temperature for the next leaching experiments.

2.2.3. Sulfuric acid leaching

The roasted pellet sample was leached by 1–20% sulfuric acid at a liquid/solid mass ratio of 1–5 (100 g roasted pellets and 100–500 cm³ sulfuric acid) and leached for 1–10 days at room temperature.

The leaching efficiencies of vanadium and iron were calculated using equations (1) and (2).

$$\beta_V = \frac{C_V \times V}{\omega_V \times m} \times 100\% \quad (1)$$

$$\beta_{Fe} = \frac{C_{Fe} \times V}{\omega_{Fe} \times m} \times 100\% \quad (2)$$

where β , V , C and m refer to the leaching efficiency, volume of the leaching solution (dm³), concentration of vanadium and iron in the leaching solution (g/dm³), the grade of vanadium and iron in the raw sample, and mass of the raw sample (g), respectively.

2.3. Analytical methods

The concentrations of vanadium and iron in the leach solution were determined by inductively coupled plasma optical emission spectrometer (Agilent Technologies 700). The phase compositions of solid samples were identified by XRD (Panalytical Empyrean). The chemical composition of vanadium–titanium magnetite concentrate was identified by X-ray fluorescence spectrometry (XRF). Microscopic observation and elemental analysis were conducted by SEM equipped with energy disperse X-ray spectrometry (SEM-EDS).

3. Results and discussion

3.1. Characterization of ore

The chemical multi-element analysis and vanadium phase analysis of the concentrate were conducted. The results are shown in Tables 1 and 2. The phase of vanadium is determined by the chemical method shown in Fig. 2 (Luo et al., 2021).

Table 1. Main chemical composition of the concentrate

Compound	Fe ₂ O ₃	TiO ₂	SiO ₂	MgO	Al ₂ O ₃	SO ₃	Cr ₂ O ₃	CaO	V ₂ O ₅	Others
wt. %	75.88	10.55	3.81	3.22	2.44	1.23	0.78	0.75	0.61	0.73

Table 2. Phase analysis of vanadium wt. %

Vanadium Phase	Sulfide	Silicate	Ilmenite	Magnetite
Content	1.31	0.81	0.69	97.19

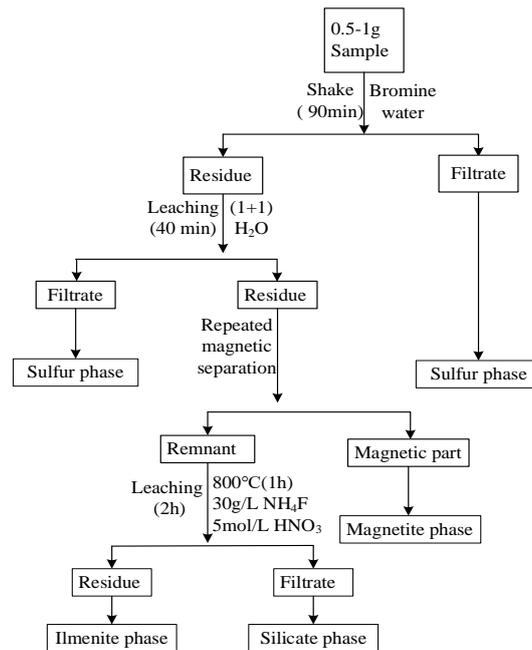


Fig. 2. Phase analysis process of vanadium in vanadium-titanium magnetite

Table 1 shows that the grade of V_2O_5 was 0.61%. Thus, the concentrate was a typical low-quality material for vanadium extraction. Meanwhile, the grades of Fe_2O_3 and TiO_2 were up to 75.88% and 10.55%, respectively. Thus, the separation of V from Fe must be considered in the vanadium extraction process. In addition, other low-grade elements, such as magnesium (Mg), aluminum (Al), sulfur (S), silicon (Si), and calcium (Ca), were present. Table 2 reveals that vanadium mainly existed in the magnetite phase, accounting for 97%, and the remaining phases account for 2.9%. Vanadium exists in magnetite phase in the form of vanadium-iron spinel (FeV_2O_4) because V^{3+} replaces Fe^{3+} in Fe_3O_4 (Zheng et al., 2019).

Combining XRD and SEM-EDS to analyze the main mineral composition of the concentrate, the XRD pattern of the original ore was obtained (Fig. 3). The concentrate was mainly composed of Fe_3O_4 and $FeTiO_3$. Given that the vanadium grade is lower than the XRD detection range, no vanadium-containing minerals have been found. Then, the concentrate was observed by SEM and scanned with EDS (Fig. 4). The results show that V, Fe, and O were highly correlated, indicating that vanadium is present in Fe_3O_4 instead of iron in the concentrate. This result was consistent with that in Table 2. In addition, the relevance of Si, Mg, and Ca was good, suggesting that Mg and Ca existed in the concentrate in the form of silicate.

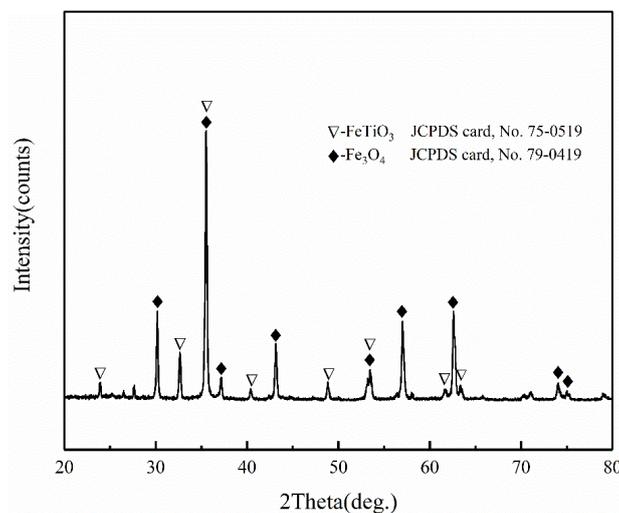


Fig. 3. XRD pattern of vanadium-titanium magnetite concentrate

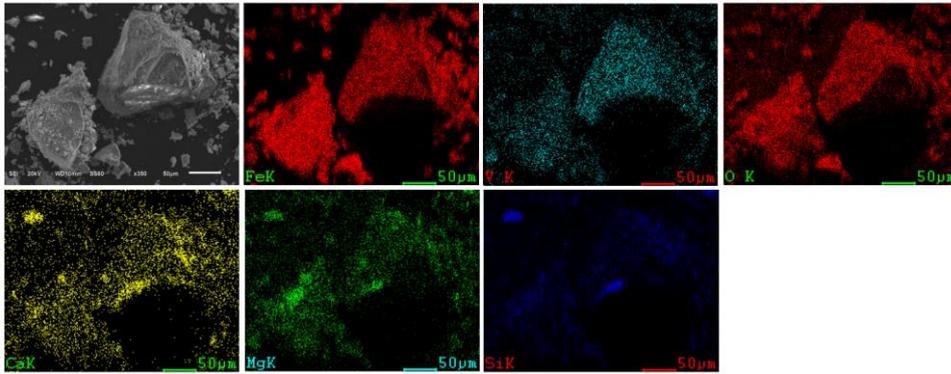


Fig. 4. SEM images with EDS element mapping of the concentrate

3.2. Thermodynamic analysis for roasting process

Vanadium (V^{3+}) is hardly soluble into acid, neutral and alkaline solutions (Zheng et al., 2019). Thus, V^{3+} or V^{4+} must be changed into V^{5+} by roasting given that V^{5+} easily dissolves in acids. Therefore, thermodynamic analysis of the pellet sample during roasting is worthy of discussion.

Based on the phase composition and main element content of the concentrate, the possible reactions during the oxidative roasting of the pellets were determined (Eqs. 3–11). The thermodynamic analysis was calculated based on HSC 5.1 software, and the relationships between the standard Gibbs free energy and temperature (ΔG^θ -T) for each reaction are shown in Fig. 5. The calculations of ΔG^θ were based on a reference value of 1 mol oxygen to ease the comparison (Wang et al., 2020).

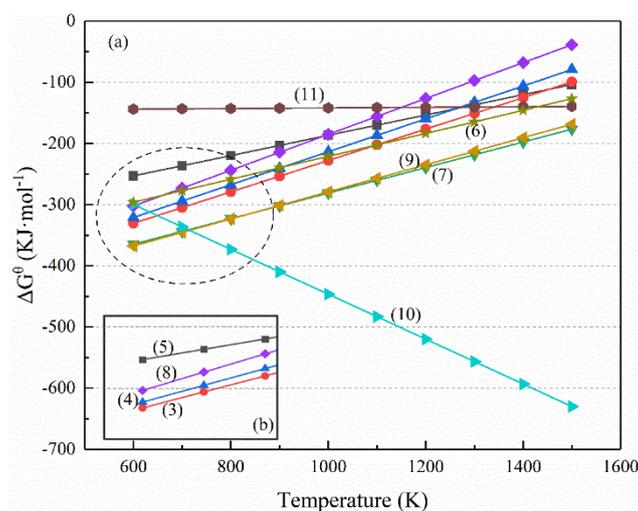
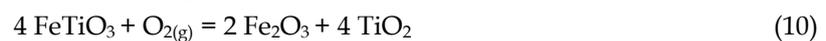
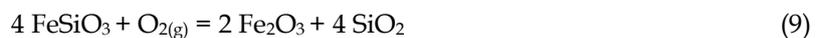
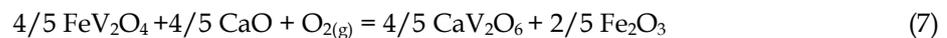
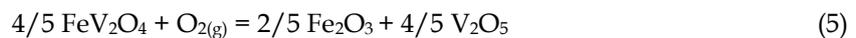
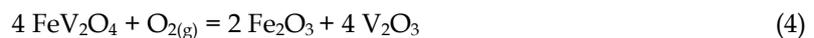
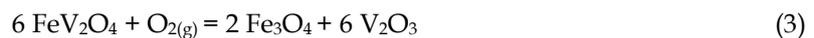


Fig. 5. ΔG^θ -T diagrams for possible chemical reactions (Eqs. 3-11)

As shown in Fig. 5, the ΔG^θ for all the reactions was negative over the temperature range of 600–1500 K. This finding indicates that the reactions were thermodynamically workable under standard

conditions. The smaller the standard Gibbs free energy, the easier the reaction will occur. Given that the ΔG^θ for Eq. (3) was the most negative, as shown in Fig. (5b), the oxidation of Fe^{2+} to Fe^{3+} occurred first. Based on the changes in ΔG^θ , the oxidation and decomposition of FeV_2O_4 into Fe_2O_3 and V_2O_3 occurred next, followed by the oxidation of V^{3+} to V^{5+} . Thus, the oxidation reaction chain of the vanadium-iron spinel is $\text{FeV}_2\text{O}_4 \rightarrow \text{Fe}_3\text{O}_4 \rightarrow \text{Fe}_2\text{O}_3 \rightarrow \text{V}_2\text{O}_5$. Judging from Eq. (11), the complex silicate compound $\text{CaMgSi}_2\text{O}_6$ was generated at high roasting temperatures.

3.3. Results of roasting and leaching experiments

The roasting temperature was varied from 800 to 1300 °C to investigate the effect of roasting temperature on the leaching rate of vanadium and iron. Other conditions were controlled as follows: roasting time of 180 min, leaching time of 6 days, liquid/solid ratio equal to 3:1, and 10% H_2SO_4 . The results are shown in Fig. 6.

The vanadium leaching rate reached the maximum value at 57.9% after roasting at 1200 °C. When the calcination temperature was lower than 1200 °C, the vanadium leaching rate decreased significantly because the reaction was still incomplete. After exceeding the temperature of 1200 °C, the pellets could be roasted to form new silicate compounds, such as molten silicate, preventing the leaching of vanadium. The iron leaching rate decreased as the temperature increased, and the leaching rate of iron was 0.11% at 1200 °C. In summary, the optimal roasting temperature of 1200 °C was selected.

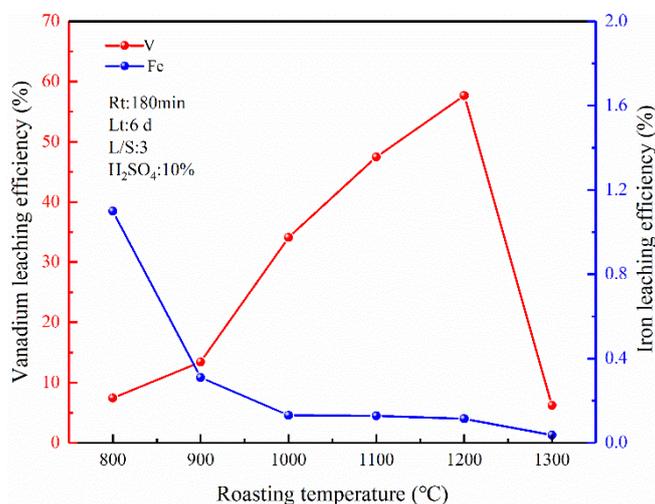


Fig. 6. Effect of roasting temperature on the leaching efficiency of V and Fe

The effect of different roasting times ranging from 1 to 210 min on V and Fe leaching efficiency was studied at a roasting temperature of 1200 °C, leaching time of 6 days, sulfuric acid concentration of 10% (V/V), and liquid-to-solid ratio of 3 cm³/g.

As shown in Fig. 7, the vanadium leaching rate increased rapidly from 40.9 to 60.3% as the roasting time increased from 1 to 180 min. The further increase in the roasting time to 210 min caused no evident change on the vanadium leaching rate. Meanwhile, when vanadium reached the maximum leaching rate, the iron leaching efficiency was 0.17%. Therefore, the optimal roasting time of 180 min was selected.

The effects of different leaching times (2–10 days) on the leaching rate of vanadium and iron were investigated under the conditions of a roasting temperature of 1200 °C, roasting time of 180 min, liquid–solid ratio of 3:1, and 10% H_2SO_4 . The results are shown in Fig. 8.

The experiment showed that the leaching rate of iron vanadium increased slowly as the leaching time increased. After leaching for 6 days, the vanadium leaching rate was up to the maximum value of 60%. Further increasing the leaching time had no remarkable effect on the vanadium leaching rate, but it can increase the leaching rate of iron. Therefore, the optimal leaching time was determined to be 6 days.

The effects of different liquid–solid ratios on vanadium leaching rate were studied under controlled conditions of the other parameters as follows: roasting at 1200 °C for 40 min, sulfuric acid concentration

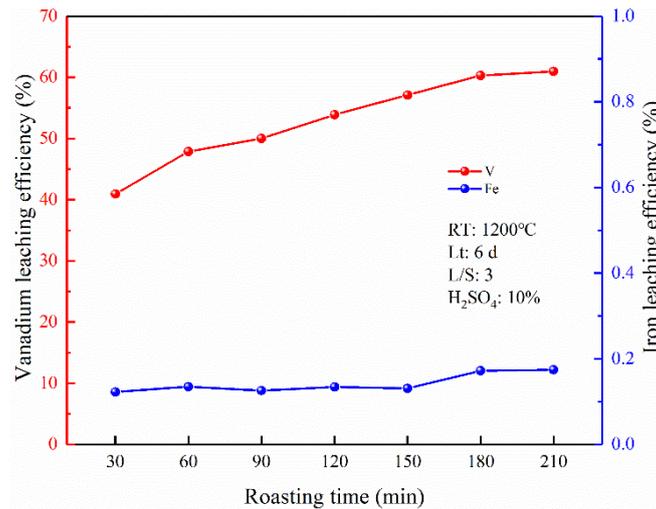


Fig. 7. Effect of roasting time on the leaching efficiency of V and Fe

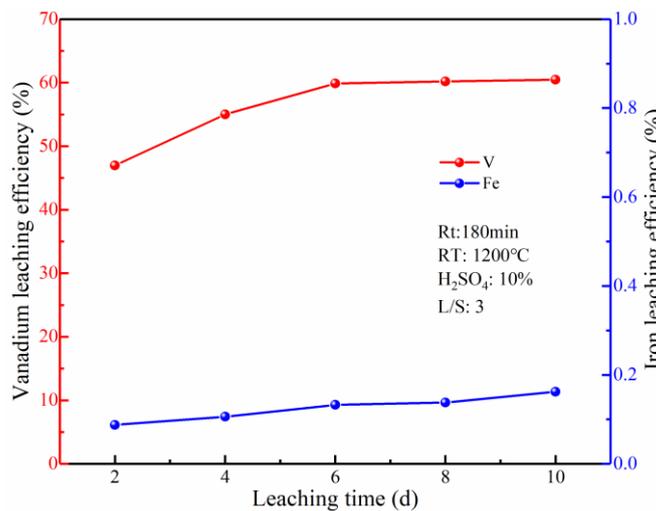


Fig. 8. Effect of leaching time on the leaching efficiency of V and Fe

of 10%, and leaching time of 6 days. The experimental results are shown in Fig. 9.

In the range of 3:1-5:1 liquid–solid ratios, the vanadium leaching rate can reach 60.3%. However, the higher the liquid–solid ratio, the lower the concentration of vanadium and the higher the acid consumption, which were not conducive to the recovery of vanadium in the subsequent processes. Therefore, the best liquid–solid ratio condition was 3:1. In addition, the iron leaching rate was consistently maintained at 0.13%, which proved that this process can suppress iron leaching and result in good selectivity under all liquid–solid ratio conditions.

The effect of different sulfuric acid on the leaching rate of V and Fe was studied under the following conditions: roasting temperature of 1200 °C, roasting time of 180 min, leaching time of 6 days, and liquid/solid ratio of 3:1. Fig. 10 shows the leaching results.

The leaching rate of vanadium showed a rising trend at first and then levelled off with the increase in sulfuric acid concentration. The vanadium leaching rate reached the highest level when the sulfuric acid concentration ranged from 10 to 15%. Meanwhile, the iron leaching rate increased from 0.06 to 0.17% as the sulfuric acid concentration increased from 1 to 20%. For sulfuric acid saving, the optimal condition of sulfuric acid concentration was set at 10%.

3.4. Discussion

The pellet crushing strength in the three stages of the vanadium extraction process was measured, and

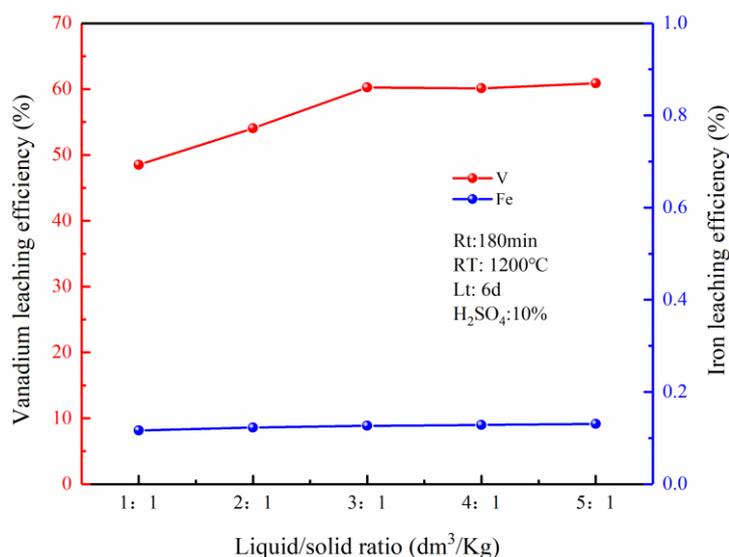


Fig. 9. Effect of liquid/solid ratio on the leaching efficiency of V and Fe

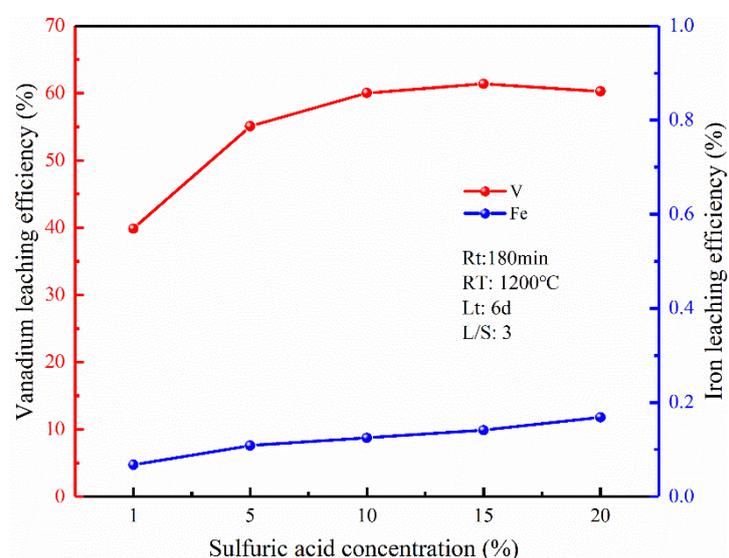


Fig. 10. Effect of sulfuric acid concentration on the leaching efficiency of V and Fe

the results are shown in Fig. 11.

Under the best calcination condition of roasting at 1200 °C for 3 h, pellet crushing strength can reach 2755 N. After leaching with 10% sulfuric acid at room temperature for 6 days, the pellet crushing strength decreased to 677 N. Finally, the leached pellets were dried and roasted at 1200 °C for 10 min, and the secondary roasting pellet crushing strength was up to 2555 N, which can meet the quality requirements of pellets produced by blast furnace production. Thus, this selective pellet roasting-acid leaching process of the concentrate is workable.

Based on the optimal leaching conditions, it revealed the concentration of vanadium and iron in the pregnant solution in Table 3. The results showed that V concentration was up to 559.2 mg/dm³, and Fe concentration was 319.6 mg/dm³. The leaching rate of vanadium was 60.3%, while the loss rate of iron was only 0.17%, showing that vanadium can be effectively separated from iron. Therefore, most of the vanadium in the leaching solution can be extracted after solvent extraction or ion exchange.

Because sulfuric acid is used for leaching, impurity sulfur will be included in the pellets. Therefore, the sulfur in the slag at each stage was tested, and the results are shown in Table 4. It can be seen from the test results that the sulfur content of the concentrate itself is extremely low, accounting for only 0.11%. After leaching, the sulfur content reached 0.58%, indicating that a certain amount of sulfur entered the pellets during sulfuric acid leaching. Finally, after the second roasting, the sulfuric acid

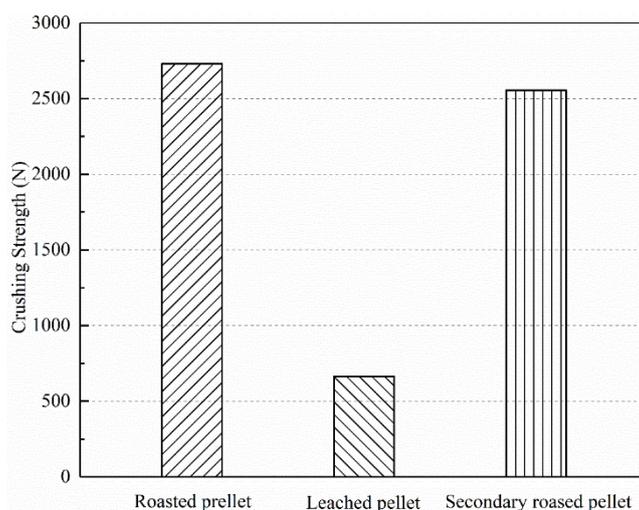


Fig. 11. Crushing strength of roasted pellets, leached pellets and secondary pellets

Table 3. Concentrates of vanadium and iron in the leaching solution (mg/dm³)

Elements	V	Fe
Concentrate	559.2	319.6

Table 4. The S content in the slag of each stage (%)

	concentrate	Leached pellets	Twice roasted pellets
S	0.11	0.58	0.12

content in the pellets dropped to 0.12%, indicating that the second roasting can not only increase the strength of the pellets, but also achieve the purpose of desulfurization.

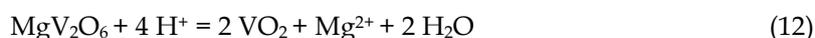
To understand the mechanisms of vanadium and iron roasting and leaching behavior, we analyzed the raw ore, roasted pellets, and leached pellets. XRD analyses were conducted to obtain a primary acquaintance of the phase changes, and the results are presented in Fig. 12.

As shown in Fig. 12, the magnetite (Fe₃O₄) and ilmenite (FeTiO₃) phases in the raw ore had been oxidized, and Fe₂O₃ and Fe₂TiO₅ appeared after blank roasting. Figs. 12(b) and 12(c) show that the diffraction peaks and intensity were not significantly different before and after leaching, which indicated that the Fe₂O₃ and Fe₂TiO₅ generated after high temperature roasting did not react with sulfuric acid in the leaching process. This finding is also in accordance with the result revealing that 0.17% of iron was leached in the experiment. Given its low content, vanadium cannot be shown in the XRD pattern.

The evolution of V phases after roasting and leaching was investigated with SEM and an EDS element mapping of the roasted and leached pellets (Fig. 13).

Fig. 13(a) shows that vanadium is evenly distributed in all regions, which is in accordance with the oxidization vanadium-iron spinel and its decomposition after roasting. Meanwhile, most areas in which V was distributed contained magnesium and calcium. This result, combined with Eqs. 6 and 7, indicates that V, Ca, and Mg transformed into CaV₂O₆ and MgV₂O₆ during the roasting process.

As shown in Fig. 13(b), the appearances of the pellet samples before and after leaching were similar, presenting unevenness, looseness, and porosity. Comparing the distribution of vanadium (Fig. 13a), the concentration of vanadium was reduced, indicating the good sulfuric acid leaching effect. The disappearance of the correlation between V and Ca/Mg demonstrates that vanadate reacted with sulfuric acid, which is also the principle of vanadium leaching. The reaction equations are shown below:



In conclusion, the main reaction chain of the vanadium is FeV₂O₄→V₂O₅→(Ca, Mg)V₂O₆→V₂O²⁺. In addition, vanadium could not be completely leached in this study because several compounds, such as

molten silicate $\text{Ca}_2\text{MgSi}_2\text{O}_7$, FeTi_2O_5 , and CaSO_4 , encapsulated a part of vanadium. Moreover, in the whole leaching process, XRD and SEM-EDS showed that the Fe_2O_3 obtained after high-temperature roasting cannot be leached by sulfuric acid. This result provides conditions for the selective separation of vanadium and iron.

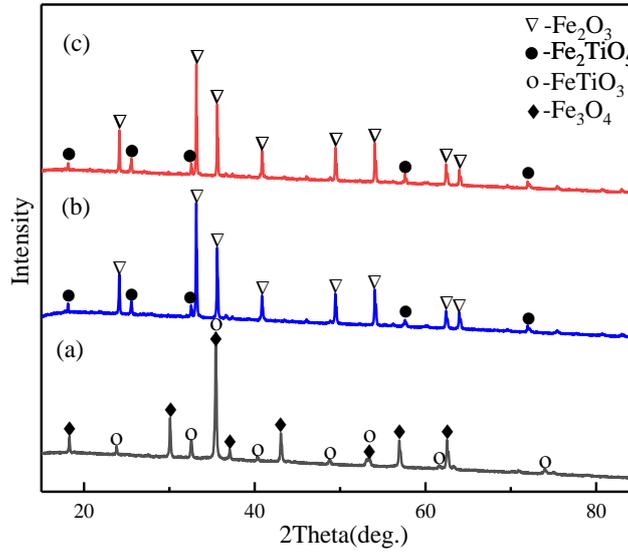
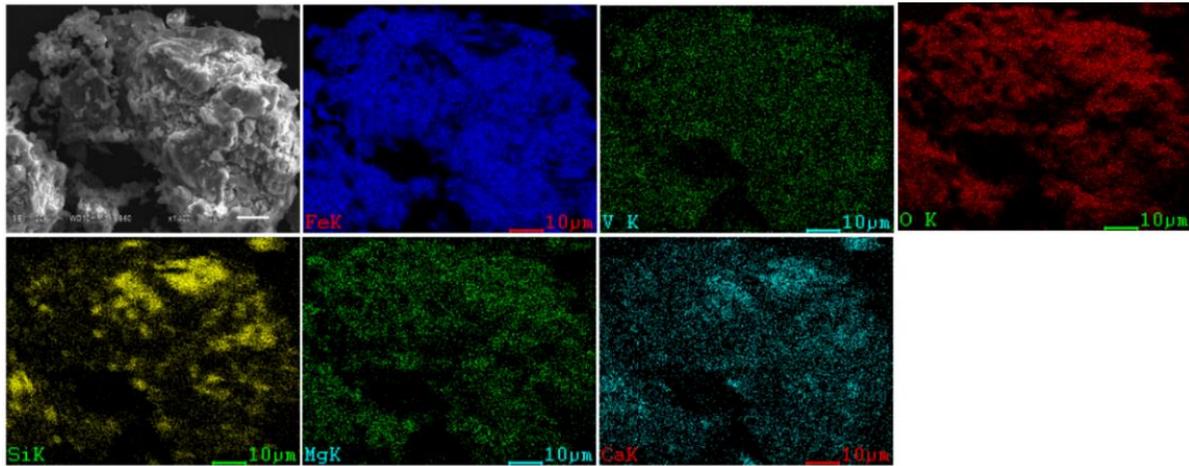
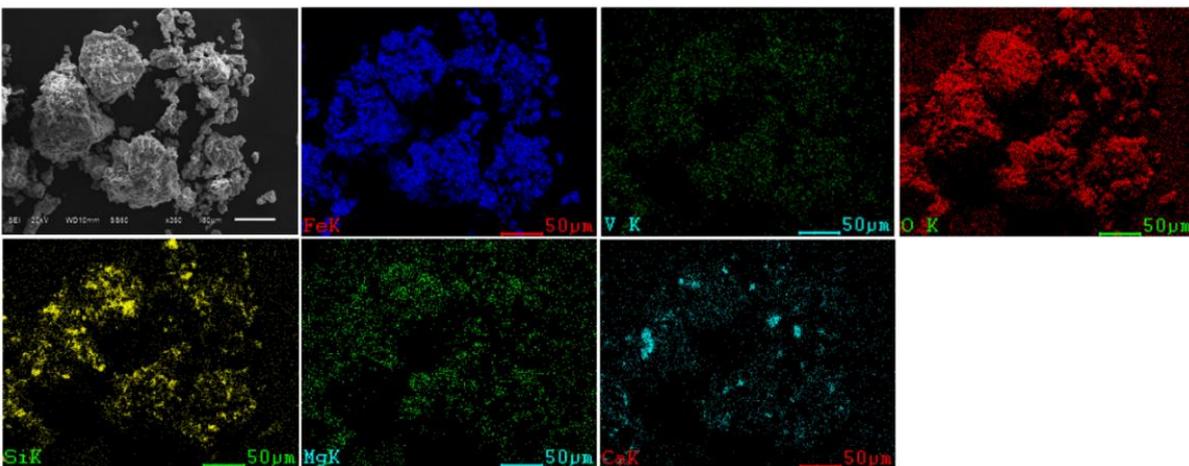


Fig. 12. XRD pattern of pellet samples: (a) concentrate, (b) roasted pellets, and (c) leached pellets



(a) Roasting pellets



(b) Leaching pellets

Fig. 13. SEM images with EDS element mappings of (a) the roasting and (b) leaching pellets

4. Conclusions

In this work, a novel process of pellet non-salt roasting–acid leaching was proposed to selectively extract vanadium from vanadium–titanium magnetite concentrate. The optimal roasting and leaching conditions for vanadium extraction were explored. In addition, the mechanism of selective vanadium extraction was studied in detail. The following conclusions were drawn:

(1) The most suitable roasting and leaching conditions were obtained as follows: roasting temperature of 1200 °C, roasting time of 3 h, leaching time of 6 days, and a leaching liquid–solid ratio of 3 cm³/g with 10% H₂SO₄. The maximal vanadium leaching efficiency was up to 60.3% with 0.17% of the iron impurity leached. Finally, the leached pellets can reach 2555 N after the secondary roasting, meeting the quality requirements for blast furnace production.

(2) The XRD and SEM-EDS analyses of the concentrate, roasted pellet, and leached pellet illustrated that the vanadium iron spinel was oxidized and decomposed into Fe₂O₃ and vanadate during the roasting process. As in the leaching process, the dilute sulfuric acid reacted with vanadate and cannot react with Fe₂O₃, selective vanadium extraction can be achieved.

Acknowledgments

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