



Source separation of vanadium over iron from roasted vanadium-bearing shale during acid leaching via ferric fluoride surface coating

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ABSTRACT

The vanadium in acid leachates of vanadium-bearing (V-bearing) shale is usually contaminated by massive impurities during leaching, especially iron impurity. Therefore, the separation of vanadium from iron impurity at source for the production of high purity vanadium products is of great necessity because of increasing demand for vanadium products in the fields of vanadium redox flow battery, specialty steel alloy and catalyst. In this paper, the source separation of vanadium over iron from roasted vanadium-bearing shale during acid leaching was achieved via FeF₃ surface coating. Under the conditions of a H₂SO₄ concentration of 20 vol%, a leaching temperature of 95 °C, a leaching time of 4 h and a liquid–solid ratio of 1.5 mL/g, 89.8% of the vanadium leaching efficiency was obtained with only 5.2% of the iron leached with a CaF₂ addition of 5 wt%; however, 20.9% of the iron and only 66.2% of the vanadium were leached without CaF₂ addition. The enhancement of vanadium leaching efficiency was mainly attributed to the muscovite disintegration in HF generated from the reaction between CaF₂ and H₂SO₄. The suppression of iron leaching behavior was due to the surface passivation of hematite coated with FeF₃ which was generated from the reactions of Fe₂O₃ and FeSO₄ with HF. The FeF₃ coating can prevent the attack of H⁺ and SO₄²⁻ on the hematite; therefore, the source separation of vanadium over iron during acid leaching was realized.

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1. Introduction

Vanadium plays an important role in various industries (such as vanadium redox flow battery, specialty steel alloy and catalyst) because of its good properties (Li et al., 2017; Choi et al., 2017). Vanadium-bearing (V-bearing) shale (also called stone coal), accounting for more than 87% of the domestic reserves of vanadium, is a type of V-bearing resource that is widespread in China (Xue et al., 2017; Zhao et al., 2013). Therefore, vanadium extraction from V-bearing shale has been given substantial attention.

The vanadium in V-bearing shale mainly exists as an isomorphism in the crystal lattice of mica group minerals where vanadium can readily replace Al(III), making the release of vanadium from V-bearing shale difficult (Xue et al., 2017; Zeng et al., 2015). At present, high temperature blank roasting together with sulfuric acid leaching is widely adopted to break the structures of mica group minerals owing to the advantages of high efficiency and good environmental compatibility (Zeng et al., 2015; Zhang et al., 2011). However, many impurity ions, particularly iron impurity, are also leached into the leachates because of the poor selectivity for vanadium extraction during leaching (Hu et al., 2017). The iron impurity seriously affects the vanadium enrichment process from the leachates and the purity of vanadium-bearing products. Solvent extraction and ion exchange are the most common adopted processes to separate and concentrate vanadium from the leachates (Zhu et al., 2017; Li et al., 2013). Generally, the ion exchange process is used for leachates with a low

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impurity concentration and a low acidity. However, when the pH value is adjusted to greater than 2.0, Fe(III) will easily form $\text{Fe}(\text{OH})_3$ precipitation which hinders normal industrial production of the ion exchange and solvent extraction processes due to the formation of a third phase (Wu et al., 2013). The separation of vanadium over iron from the acid leachates using mixed reagent D2EHPA/TBP has been widely applied, and a high separation coefficient can be obtained when Fe(III) is reduced to Fe(II) (Chen et al., 2015; Cheraghi et al., 2015; Ma et al., 2015). However, Fe(II) will be gradually oxidized to Fe(III) in air during the extraction, and the iron impurity can be partially co-extracted with vanadium (Hu et al., 2017; Ma et al., 2015), decreasing the purity of vanadium-bearing products. Based on the above, it can be concluded that the sulfuric acid leaching process is of poor selectivity for vanadium extraction over iron. Therefore, it is a better choice to adopt some at-source prevention methods inhibiting the iron leaching behavior as much as possible to eliminate the adverse effects.

Recently, many researchers have studied the coating technology in varied fields, such as the control of acid mine drainage (AMD), the corrosion protection of metals and the materials modification. Coating is recognized as a promising technology for the source control of AMD because coatings, with poor solubility formed on the surface of pyrite, can inhibit pyrite oxidation by preventing the contact of FeS_2 with O_2 in the air (Kang et al., 2016; Liu et al., 2017). For the steel industry, the corrosion of steels troubles industrial applications, and the coating technology is widely used to have the steels coated with some materials which can provide a barrier by preventing the diffusion of O_2 and H_2O (Bandeira et al., 2017; Pourhashem et al., 2017). Meanwhile, the coating technology is a hot topic in the field of extending the properties of advanced materials, especially electrode materials (Ye et al., 2017; Zuo et al., 2017; Huang and Terentjev, 2011; Ito et al., 2017; Ke et al., 2017).

Generally speaking, the coating technology is mainly a chemical surface conversion treatment which aims at modifying and improving the materials surface properties. As discussed above, the sulfuric acid leaching process for the vanadium extraction from V-bearing shale is troubled by a poor selectivity for vanadium over iron. Therefore, it may be a good idea to purposefully modify the surface chemical composition of iron-bearing mineral (mainly Fe_2O_3) in roasted V-bearing shale with a coating which inhibits the Fe_2O_3 dissolution in sulfuric acid to some extent. FeF_3 is usually precipitated on the surface of cathode as a coating through hydrothermal method, enhancing the electrochemical properties by protecting the cathode from being contacted with the electrolyte solution (Zhao et al., 2015; Kim et al., 2014). Extensive studies have been performed to recover or remove the iron as an iron fluoride hydrate precipitation from spent mixed acid pickling solutions (Forsberg and Rasmuson, 2007, 2015). For the sulfuric acid leaching process of V-bearing shale, some fluorides are usually applied to enhance the vanadium leaching efficiency by facilitating disintegration of silicate minerals (Wang et al., 2015); therefore, there exists a possibility that fluorides can enhance the selectivity for vanadium by forming a coating on the surface of Fe_2O_3 . However, much work so far has focused on the vanadium leaching behavior, and there are relatively few studies devoted to the iron leaching behavior with and without fluorides addition, especially the surface conversion of Fe_2O_3 .

The primary purpose of this research is to analyze the surface conversion of iron-bearing mineral in roasted V-bearing shale during the sulfuric acid leaching process, and gives a feasible coating method to enhance the selectivity for vanadium extraction over iron. A roasted V-bearing shale was leached using sulfuric acid with and without CaF_2 addition, respectively. The effects of the leaching conditions on the vanadium and iron efficiencies were investigated. Besides, the mechanisms of the vanadium and iron leaching behaviors were analyzed in detail.

2. Experimental

2.1. Materials

The V-bearing shale used in this work was obtained from Hubei province, China. The V-bearing shale was crushed to a grain size of 0–3 mm by a jaw crusher and a double-roll crusher. The crushed ore was heated to 800 °C at the rate of 10 °C/min for 60 min in a muffle furnace and then the ore was ground to minus 0.074 mm with a vibration mill, accounting for 75% of the total. The obtained ore is referred to as roasted V-bearing shale throughout this study. All reagents were of analytical grade and all water was deionized water in this study.

The ICP-AES and XRD analyses of the roasted V-bearing shale are given in Table 1 and Fig. 1, respectively. It can be seen from Table 1 that the grades of V_2O_5 and Fe_2O_3 are 0.89% and 5.77%, respectively. Fig. 1 indicates that the main mineral phases of the roasted V-bearing shale are quartz, muscovite, hematite and anhydrite. Most of the iron exists as hematite. As mentioned above, the vanadium mostly replaces Al(III) isomorphically in the mica group minerals lattice. The SEM-EDS analysis (Fig. 2) was conducted to obtain a

Table 1
Main chemical composition of the roasted V-bearing shale.

| Element | V_2O_5 | Fe_2O_3 | SiO_2 | Al_2O_3 | CaO | K_2O | MgO | C | S |
|--------------|------------------------|-------------------------|----------------|-------------------------|-------|----------------------|------|------|------|
| Content, wt% | 0.89 | 5.77 | 58.32 | 12.46 | 10.06 | 2.96 | 2.16 | 0.62 | 2.19 |

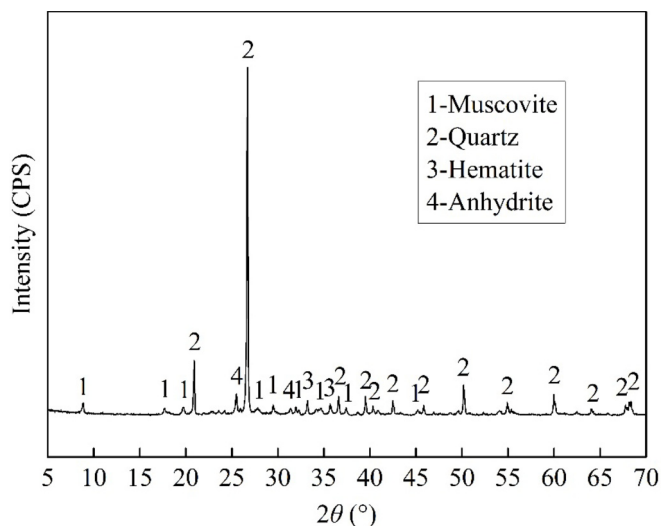


Fig. 1. XRD pattern of the roasted V-bearing shale.

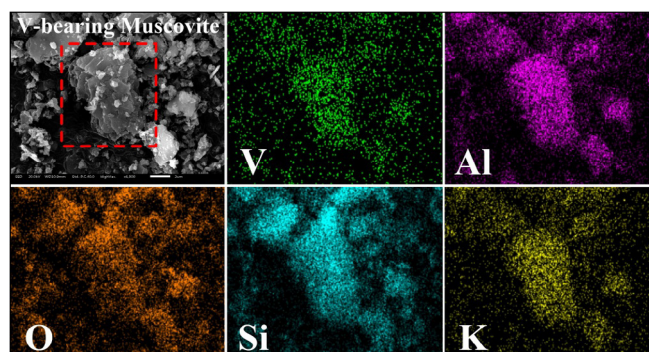


Fig. 2. SEM images with EDS element mapping of the roasted V-bearing shale.

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