



# Compound leaching of rare earth from the ion-adsorption type rare earth ore with magnesium sulfate and ascorbic acid



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## ABSTRACT

To reduce the consumption of  $\text{MgSO}_4$  and to leach the colloidal sediment phase rare earth, a  $\text{MgSO}_4$ -ascorbic acid compound leaching agent was introduced to deal with the ion-adsorption type rare earth ore. The ascorbic acid was used as an assistant to enhance the leaching of the ion-exchangeable phase rare earth and to reductively leach the colloidal sediment phase rare earth, making use of its ability to coordinate with rare earth ions and its reductive properties. It was determined that the total rare earth leaching efficiency was 86.2% and the Ce partition was 5.62% at a flow rate of 0.60 mL/min, an initial pH of 2.50, a liquid to ore ratio of 1.30 mL/g, and 0.15 mol/L magnesium sulfate and 1.0 g/L ascorbic acid in the leaching agent. The concentrations of Al and Fe in the leaching liquor were 21.36 mg/L and 30.22 mg/L, respectively. Moreover, the contents of the ion-exchangeable phase and the colloidal sediment phase rare earth in the leaching tailings were 0.03‰ and 0.06‰, respectively. The leaching efficiency of the colloidal sediment phase rare earth was approximately 57.1%. Hence, an alternative process would be provided for the leaching of rare earth from ion-adsorption type rare earth ore. The process could reduce the consumption of  $\text{MgSO}_4$  and would have great significance for environmentally-friendly extraction of ion-adsorption type rare earth ore and the improvement of resource utilization.

## 1. Introduction

The ion-adsorption type rare earth ore, first discovered in 1969, is mainly located in the seven provinces of Southern China (Xu 2002). This kind of ore has many advantages, such as complete rare earth partition, low radioactivity, simple leaching process and rich in middle and heavy rare earth elements, which make it a valuable strategic mineral resource (Xiao et al. 2015a). It is generally accepted that rare earth in the ion-adsorption type rare earth ore presents in four phases (Chi et al. 2005): water soluble phase, ion-exchangeable phase, colloidal sediment phase and mineral phase. The ion-exchangeable phase rare earth accounts for more than 80% of the whole-phase rare earth in general. It can be easily exchanged and released when encountering the cations (such as  $\text{NH}_4^+$ ,  $\text{Mg}^{2+}$ ). According to the ion-exchange property, a series of hydrometallurgy methods for this special ore have been developed along the green chemical leaching approach (Huang et al. 2015). Currently, rare earth (RE) is mainly recovered from the ion-adsorption type rare earth ore by the in-situ leaching process with  $(\text{NH}_4)_2\text{SO}_4$  in the industry. The practice of  $(\text{NH}_4)_2\text{SO}_4$  leaching agent has produced serious ammonia-nitrogen pollution (Xiao et al. 2015b;

Yang et al. 2013). Additionally, the  $(\text{NH}_4)_2\text{SO}_4$  leaching agent can leach most of the rare earth in the ion-exchangeable phase, but it is ineffective to the leaching of the colloidal sediment phase (Chi and Tian 2006). The content of the colloidal sediment phase in the ion-adsorption type rare earth ore varies in different mine environment, ranging from 2% to 40% (Li 2014). Over the past few decades, nearly 300 thousand tons of ion-adsorption type rare earth concentrates have been produced. If the content of the colloidal phase is on average 10%, more than 35 thousand tons of rare earth concentrates have not been recovered (Li 2014), resulting in a serious waste of rare earth resources.

On the one hand, in order to eliminate the ammonia-nitrogen pollution, new non-ammonium leaching agents (Wang et al. 2013; Chi et al. 2013; Huang et al. 2010) were developed to replace  $(\text{NH}_4)_2\text{SO}_4$ , among which the magnesium sulfate leaching agent could achieve an ecologically-friendly leaching of rare earth from the ion-adsorption type rare earth ore (Xiao et al., 2015c; Xiao et al. 2016a). However, the leaching capacity of magnesium sulfate was slightly weaker than that of ammonium sulfate, resulting in a higher consumption of magnesium sulfate (Xiao et al., 2015d). On the other hand, the colloidal sediment phase rare earth was minimally leached even if the initial pH of

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leaching agent was below 1.00 (Xiao et al. 2017). However, it could be reductively leached by a reductive substance. Therefore,  $\text{FeSO}_4$  and  $\text{FeSO}_4\text{-MgSO}_4$  leaching agents were proposed by our team to deal with the ion-adsorption type rare earth ore (Xiao et al. 2016b; Xiao et al. 2017). The results (Xiao et al. 2017) showed that the rare earth leaching efficiency was approximately 85.3% and the Ce partition (The percentage of Ce content in the total rare earth) was 4.85% when the initial pH of the leaching agent was 1.50 and the Mg/Fe proportion was 99:1. Under these conditions, the leaching efficiency of the colloid sediment phase rare earth could be up to 42.9%. However, the concentration of Al in the leaching liquor was 429.44 mg/L and the concentration of Fe was 112.88 mg/L. This concentrations would cause several negative effects, such as low product purity, low rare earth yield, high consumption of precipitant, and even affect the crystallization properties of rare earth carbonate (He et al., 2016a). Ultimately, the leaching performance was unsatisfactory because it was difficult to find a balance between the rare earth leaching efficiency and the influence of impurities in the  $\text{FeSO}_4$  or the  $\text{FeSO}_4\text{-MgSO}_4$  leaching system (Xiao et al. 2017).

An ascorbic acid ( $\text{Vc}$ ,  $\text{C}_6\text{H}_8\text{O}_6$ ) will form a stable complex with rare earth ions (Evtushenko et al. 2002), and therefore it can be used as an assistant to enhance the leaching of rare earth. Moreover, ascorbic acid has reductive properties (Borsook and Keighley 1933), so it is also possible to leach the colloidal sediment phase rare earth. In view of these facts, an  $\text{MgSO}_4\text{-ascorbic acid}$  compound leaching of rare earth from ion-adsorption type rare earth ore was tested, to reduce the consumption of  $\text{MgSO}_4$  and to achieve the simultaneous leaching of the ion-exchangeable phase rare earth and the colloidal sediment phase. A preliminary leaching experiment with 0.20 mol/L  $\text{MgSO}_4$  and 0.5 g/L ascorbic acid was conducted (Xiao et al. 2017), and the results showed that 85.8% rare earth leaching efficiency and 5.33% Ce partition were achieved. The leaching efficiency of the colloidal sediment phase rare earth could also reach to approximately 50%. Moreover, the contents of Fe and Al in the leaching liquor were only 1.35 mg and 43.54 mg. Therefore, an alternative process could be provided for the leaching of the colloidal sediment phase and the reduction of  $\text{MgSO}_4$  consumption. To obtain more detailed leaching characteristics of the  $\text{MgSO}_4\text{-ascorbic acid}$  compound leaching system, analysis of the leaching mechanism was undertaken in this paper, and the leaching behaviors of rare earth and non-rare earth impurities under different conditions were also investigated and discussed. Magnesium-ascorbic acid compound leaching would be a feasible operation for the leaching of rare earth from the ion-adsorption type rare earth ore. It could have great significance for environmentally-friendly leaching of the ion-adsorption type rare earth ore and the improvement of resource utilization.

## 2. Experimental

### 2.1. The characterization and composition of experimental rare earth ore

The ion-adsorption type rare earth ore was collected from the Liutang (LT) rare earth mine area located in Chongzuo City, Guangxi Province, China. The existing state of rare earth in the LT rare earth ore was determined by sequential fractionating extraction methods. The water soluble phase rare earth was the rare earth dissolved in water, which accounted for less than one ten thousandth of total rare earth and was considered negligible. The extraction of other phases was carried out progressively, following the sequence listed below (Chi et al. 2005; Chi and Tian 2006).

F1: 500 g ore was packed in a column of 40 mm in diameter. Then it was leached by 50 g/L  $(\text{NH}_4)_2\text{SO}_4$  solution until there was no rare earth in the effluent. The effluent was collected and analyzed. This fraction should reflect the ion-exchangeable phase rare earth.

F2: The residue from F1 was leached by another solution (0.5 mol/L  $\text{NH}_2\text{OH}\cdot\text{HCl}$  + 2.0 mol/L  $\text{HCl}$ ) until there was no rare earth in the effluent. The effluent was collected and analyzed as well. This fraction

**Table 1**

The content of Rare earth in different phases for LT ore sample (wt.%).

Phases	whole-phase	Water soluble	Ion-exchangeable	Colloidal sediment	Minerals
Content	1.80	negligible	1.50	0.14	0.16

should reflect the colloidal sediment phase rare earth.

F3: The residue from F2 was roasted with sodium peroxide and sodium hydroxide at 900 °C, followed by leaching with  $\text{HCl}$ . The leaching liquor was collected and analyzed. This fraction should reflect the mineral phase rare earth.

F4: The raw ores were roasted with sodium peroxide and sodium hydroxide at 900 °C, followed by leaching with  $\text{HCl}$ . The leaching liquor was collected and analyzed. This fraction should reflect the whole-phase rare earth.

The results are shown in Table 1. The content of total rare earth was 1.80 wt.%. The content of the ion-exchangeable phase rare earth was 1.50 wt.%, accounting for 83.3% of the whole-phase rare earth. Therefore, the rare earth leaching efficiency could not exceed 83.3% when leaching with the single  $\text{MgSO}_4$  or  $(\text{NH}_4)_2\text{SO}_4$  leaching agent. The colloid sediment and mineral phases accounted for 7.8 wt% and 8.9 wt %, respectively. The rare earth partition in the different states was tested by inductively coupled plasma-atomic emission spectrometry (ICP-AES, PerkinElmer, co., LTD. Optima 8300) and is shown in Table 2. According to the rare earth partition of the ion-exchangeable phase, it could be easily seen that the LT ore was a kind of rare earth ore with Middle Y and Rich Eu, and the Ce partition in the colloidal sediment phase was 55.84%, more than half of the total rare earth. Furthermore, the chemical composition of ore samples was tested by XRF (XRF-1800, SHIMADZU Co., Ltd.), and the results were listed in Table 3. The main minerals in the ore were clay minerals and quartz sand, so that  $\text{SiO}_2$  and  $\text{Al}_2\text{O}_3$  were the main composition.

### 2.2. Apparatus and experimental procedure

All chemicals used in the experiments were of analytical grade. All the solutions were prepared with deionized water.

In the column leaching process, columns of 40 mm inner diameter were used as the apparatus, and the precision pump (Baoding Longer Precision Pump Co., Ltd., BT100-1F) was used to control the flow rate of the leaching agent. The experimental setup is shown in Fig. 1. First, 300 g of dried rare earth ore obtained by the quadrature method, was packed into the column. And then, the column was slightly tapped to make a packed bed height of 20 cm. The column with ore was eluted with the leaching agent of a given concentration and pH at a desired leach flow rate. The initial pH of the leaching agent was adjusted by sulfuric acid and magnesium oxide. The leaching experiments were performed at ambient temperature (approximately 25 °C). After the addition of the leaching agent, 120 mL deionized water was used to wash the ore at the same flow rate. Finally, all the effluent was collected as leaching liquor, and it was diluted to 500 mL for testing. The rare earth grade of the ore was only 1.80wt.%, and the leaching process was mainly a process of ion exchange. Therefore, in the leaching of the ion-adsorption type rare earth ore, the quality of the raw ore and leaching tailings changed very little, so they were considered to be approximately the same approximately (Xiao et al. 2015e). Moreover, it should be noted that the conventional XRD/SEM test was of little significance to the study of the column leaching process because of the low rare earth grade and special rare earth phase in the ion-adsorption type rare earth ore (Chi and Tian 2006). Hence, the existing state of the rare earth in the leaching tailings was determined with sequential fractionating extraction methods, such that the leaching effect could be measured by comparing the rare earth content of each phase in the raw ore and leaching tailings.

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