



# Effect of a novel compound on leaching process of weathered crust elution-deposited rare earth ore



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## ABSTRACT

The leaching process of weathered crust elution-deposited rare earth ore is a typical noncatalytic heterogeneous reaction in liquid–solid systems. In order to intensify the leaching process of rare earth and reduce the impurity leaching accompanying with rare earth, ammonium sulfate and ammonium formate were mixed as a novel compound leaching agent, in which ammonium formate was used as an inhibitor for aluminum, as well as an aid agent to speed up rare earth leaching. Effects of ammonium formate concentration, liquid/solid ratio, leaching agent pH and leaching temperature on the leaching process of rare earth and aluminum were investigated, and then were systematically evaluated by the leaching kinetic theory. It is found that ammonium formate could effectively enhance the leaching rate of rare earth and significantly inhibit the leaching efficiency of aluminum. Leaching agent pH has a greater impact on the leaching efficiency of aluminum, and no effects on that of rare earth. A higher leaching temperature in the tested range could enhance the leaching rate of rare earth and aluminum, but it will largely increase the production cost. The optimum conditions of leaching rare earth and aluminum are 0.1 mol/L ammonium sulfate compounded with 0.032 mol/L ammonium formate, 1:1 liquid:solid (mL/g), pH 5.0 ~ 8.0 and room temperature. At these conditions, the leaching efficiency of rare earth and aluminum are 92.97% and 37.79% respectively. Moreover, the leaching equilibrium time for rare earth was 320 min shorter than the traditional leaching process by the ammonium sulfate, confirming an acceleration from this novel compound on the leaching of rare earth, especially for the difficult-to-infiltrate weathered crust elution-deposited rare earth ore. The leaching results are well fitted with the shrinking core model with apparent activation energy of 15.83 kJ/mol for RE and 9.60 kJ/mol for Al in the temperature range of 283 ~ 323 K, indicating a diffusion controlled reaction, and the reaction orders are 1.06 for RE and –1.77 for Al.

## 1 Introduction

The weathered crust elution-deposited rare earth ore was first discovered in Jiangxi Province, China in 1969, and widely distributed in southern China, such as Jiangxi, Fujian, Guangdong, Yunnan and Hunan (Chi et al., 2008; Zhou et al., 2017). The weathered crust elution-deposited rare earth ore as a typical ion-adsorbed-type rare earth ore is formed through a series of chemical, physical and biological processes with rare earth hydroxyl aqueous ions absorbed onto clay minerals during the weathering and infiltration process (Qiu et al., 2014; He et al., 2016; Tian et al., 2010). The rare earth in this ore can be leached by an electrolyte solution using the ion-exchange method based on the rare earth ions adsorbed characteristics (Tian et al., 2010; He et al., 2016b). The exploitation of this ore has been developed from the initial barrel dipping and pool dipping process to heap leaching and in-situ leaching process. At present, more mature heap leaching and in-situ

leaching processes are used to recover rare earth resources in actual mine production. Ammonium sulfate can enhance the selectivity of the leaching process, resulting from metal ions such as calcium and barium to form insoluble sulfates and remain in the ore body. It is thus commonly applied as a leaching agent.

A high consumption of leaching agent and a low rare-earth leaching efficiency can be predicted due to the poor permeability and the varied characteristics of clay minerals. The weathered crust elution-deposited rare earth ore contains some special mineralogy characteristics similar to the silicate clay mineral, such as small mineral particles, high specific surface, small porosity, and liability for forming an electric double layer, which makes a bad permeability of this ore. It would result in a low diffusion driving force in the leaching process, and then the worse leaching efficiency and the more leaching agent consumption. Thus, it is imperative to find a novel leaching agent to enhance the leaching process, particularly for the difficult-to-infiltrate ore. The difficult-to-

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infiltrate weathering crust elution-deposited rare earth ore is sometimes given up for mining due to its longer leaching time or no leachate collected. At present, the efficient mining of this kind of ore has been paid more and more attention for the high-effective utilization.

Due to the complex chemical composition of clay minerals, the selectivity of electrolyte ion exchange is poor, resulting in the presence of a large amount of impurity ions, especially aluminum ions, in the rare earth leachate. These impurities can be coprecipitated together with the rare earth ions when the leachate was processed by ammonium bicarbonate to produce rare earth carbonate. Thus, the aluminum ion, in the leachate, not only makes the subsequent process harder, but also affects the quality of rare earth products. To inhibit the aluminum leaching from the weathered crust elution-deposited rare earth ore is a cost-effective method for decreasing the adverse effect of aluminum and improve the efficiency of leaching agent.

We compared several chemicals, such as ammonium formate, hydroxypropyl methyl cellulose, sesbaniagum. The results showed that the addition of ammonium formate improves the leaching rate of rare earth to a greater extent, while an inhibition on aluminum leaching by the ammonium formate is exceptionally discovered, so a detailed study on the impact of ammonium formate on the whole leaching process of weathered crust elution-deposited rare earth ore has been discussed in this study. The effect of ammonium formate concentration, the liquid/solid ratio, the leaching agent pH and the leaching temperature on the leaching process of rare earth and aluminum were investigated and discussed by the kinetic theory.

## 2. Experiment

### 2.1. Materials

The weathered crust elution-deposited rare earth ore samples used in this study were from Guangdong province, China. The rare earth ore sample was shaped into a cone, flattened by a trowel and quartered. Then, the last two diagonal quarters were collected and remixed. The sample preparation steps were repeated until the fully mixed sample was enough for the experiments. Finally, the rare earth ore sample was air dried at 60 ~ 70 °C for 12 used for column leaching experiments. Part of the ore sample was sieved using standard sieves for sample characterization.

Ammonium sulfate ((NH<sub>4</sub>)<sub>2</sub>SO<sub>4</sub>), ammonium formate (HCOONH<sub>4</sub>), zinc oxide (ZnO), ethylenediaminetetraacetic acid disodium, ascorbic acid, sulfosalicylic acid, xylenol orange, hexamethylenetetramine, hydrochloric acid and ammonium hydroxide were purchased from Sinopharm Chemical Reagent Co., Ltd., Shanghai, China, with analytical grade. Standard solution of zinc was purchased from National Center of Analysis and Testing for Nonferrous Metals and Electronic Materials (NCATN).

### 2.2. Sample characterization

250 g of dried rare earth ore samples with different sieving particle sizes were packed uniformly in the glass column and leached by 0.1 mol/L ammonium sulfate with liquid to solid ratio of 4:1. Then leachates from different sieving particle size ores were collected separately and the contents of rare earth and aluminum in these leachates were analyzed to calculate the grade of ore sample shown in Table 1. 500 g of dried rare earth ore samples without sieving was packed in the glass column and leached by 0.1 mol/L ammonium sulfate with liquid to solid ratio of 4:1. The contents of different rare earth ions in the leachate were analyzed with ICP-MS (Agilent 7700x, Agilent Technologies Inc.), and the partitioning of ion-exchangeable rare earth was obtained.

**Table 1**

Particle size distribution of rare earth ores and distribution of ion-exchangeable rare earth and aluminum (%).

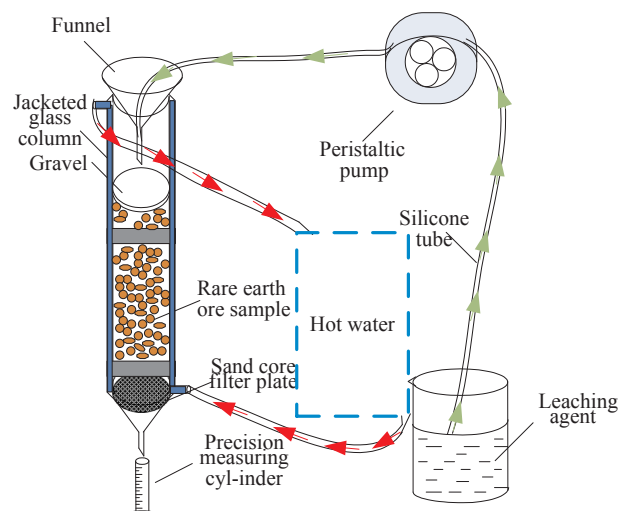
Particle size/mm	+0.83	-0.83	-0.25	-0.15	-0.109
		to	to	to	
		+0.25	+0.15	+0.109	
Mass distribution	32.84	20.90	11.52	6.35	28.39
Rare Earth Distribution ratio	16.48	13.98	13.65	8.41	47.48
Rare Earth Grade	0.072	0.096	0.17	0.19	0.24
Aluminum Distribution ratio	2.36	10.32	13.19	11.68	62.45
Aluminum Grade	0.00064	0.0044	0.010	0.016	0.018

### 2.3. Column leaching experiment

The leaching experiments were processed in a home-made glass column covered by a glass jacket. The temperatures of leaching experiment were controlled by the hot water pumped by using a precision type thermostat. In carrying out the experiments, 250 g of dried rare earth ore sample was packed uniformly in the glass column and the packed ore height was measured. A sand core filter plate was fixed at the bottom of leaching column to retain soil particles and two layers of filter paper were placed on the upper of the rare earth ore sample to resist preferential flow. The leaching agent was added from the top of leaching column at a constant flow rate by a peristaltic pump. The schematic diagram of experimental apparatus is shown in Fig. 1. When there is no leachate can be collected from the bottom of leaching column, the leaching experiment is finished. The collected leachate was analyzed by titration method to determine the concentrations of rare earth and aluminum. The experiments were conducted at ambient temperature of 25 ± 0.5 °C.

### 2.4. Analytical methods

The total content of rare earth (RE) in the leachate was determined by ethylene diamine tetraacetic acid (EDTA) titration using xylenol orange as indicator and hexamethylenetetramine as buffer. After that, excess EDTA solution was added into the studied liquid sample and placed in a water bath at 90 °C for 10 min. Then the content of aluminum (Al) was analyzed by back titration using a known concentration of zinc solution. The detailed description of the method also can be found in the literatures (Zhou et al., 2017; Yang et al., 2015; He et al., 2016c). The content of RE and aluminum was also determined by the EDTA titration method, and then the leaching efficiency ( $\alpha$ ) can be calculated according to the following equation:



**Fig. 1.** Schematic diagram of experimental apparatus.

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