



Recovery of rare earth elements from aqueous solution obtained from Vietnamese clay minerals using dried and carbonized parachlorella



Josiane Ponou^{a,*}, Li Pang Wang^a, Gjergj Dodbiba^a, Katsunori Okaya^a, Toyohisa Fujita^a, Kohei Mitsuhashi^b, Takafumi Atarashi^b, Gouki Satoh^c, Masayoshi Noda^c

^aDepartment of Systems Innovation, Graduate School of Engineering, University of Tokyo, 7-3-1 Hongo, Bunkyo-ku, Tokyo 113-8656, Japan

^bNittetsu Mining Co. Ltd., Research and Development Department, 8-1-Hirai-Hinode-machi, Nishitama-gun, Tokyo 190-0182, Japan

^cPanac Advance Co. Ltd., OX Tamachi Building, 5-31-19 Shiba, Minato-ku, Tokyo 108-0014, Japan

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ABSTRACT

The ongoing development of new advanced technologies, created increasing demands for rare earth elements (REEs) in the international market. The available conventional technologies for concentration and recovery of REEs are expensive making biosorption an efficient and low-cost technology for the recovery of REEs from aqueous solution. Thus, the biosorption and desorption of multi-component solution containing Y(III), La(III), Sm(III), Dy(III), Pr(III), Nd(III), Gd(III) were investigated using dried or 250 °C and 350 °C carbonized parachlorella. Evaluating the effect of pH with respect to contact time indicated a dependency of the system with those parameters. The optimum pH for dried and 250 °C carbonized parachlorella was 7 whereas 350 °C reaches its maximum uptake at pH 4. Rapid adsorption within the first 5 min of contact followed by a slight variation in the following 20 min characterized the sorption processes onto parachlorella by-products. The mechanism of the biosorption is explained by a combination of complex reactions occurring simultaneously in the biosorption process.

In addition, desorption process has been investigated using various concentrations of HCl, HNO₃, and H₂SO₄ at different temperatures. It was found that the reversible process is rapid, less temperature and pH dependent with high desorption percentage. Moreover, only light REEs were desorbed regardless of the kind of acid and the solution temperature. Parachlorella is found to be good and low-cost biosorbent for the recovery of above REEs from aqueous solutions.

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Introduction

Rare earth elements (REEs) are often referred as the “seeds of technology” because of their uses in high-tech strength permanent magnets, lasers, automotive catalytic converters, fiber optics/superconductors, electronic devices, and green energy sectors [1,2]. Due to the ongoing development of new advanced technologies, there is an over-increasing demand for REEs in the international markets, with emphasis on identifying new resources to ensure adequate supply for present and future use.

The designation “rare earths” refers to the 15 elements of the periodic table known as “lanthanides” with yttrium and scandium, further divided as a function of their atomic number into two categories. Light rare earth elements (LREEs), which accounted for 66.8% of global demand in 2010 [2] referred to lanthanum, cerium, praseodymium, neodymium, promethium and samarium. Heavy rare earths elements

(HREE), less common and more valuable, referred to the rest of lanthanides elements with yttrium. The 17 REEs are found in all REE geological deposits because they share many similar properties but their distribution and concentrations vary [2]. REE mineral deposits are usually rich in either LREE or HREE, but rarely contain both in significant quantities [2]. The term “rare earth” is actually a misnomer, because these elements are more abundant in the earth’s crust compared to silver, gold or the platinum-group metals and are part of more than 200 rock-forming minerals [1,3]. They are “rare” because they do not naturally occur in metallic form, only as mixed and scattered in minerals and are difficult to separate from each other due to very similar physico-chemical properties [1].

Several expensive methods exist to recover REEs from ores including pyrometallurgy, hydrometallurgy, etc. processes in case of REEs rich ores. In addition, different methods have been proposed for separation and preconcentration of REEs including precipitation, liquid–liquid extraction, solid–liquid extraction, super critical extraction, electro-winning, electro-refining, etc. [4–7] but, those methods are inefficient or required a large volume of solvent or intensive labor [7–9]. Thus, find a low-cost and efficient technology for the

* Corresponding author.

E-mail address: ponoujosiane21281@yahoo.co.jp (J. Ponou).

concentration and recovery of REEs led to the choice of biosorption. Some biomasses including *Pseudomonas aeruginosa*, *Saccharomyces cerevisiae*, etc. have been tested [10,11] and are found efficient for the recovery of Dy(III), Sc(III), La(III), Pr(III), Nd(III), and Eu(III). However, due to the large number of candidate biomasses, biosorption of REEs needs to be deepened and more biomasses need to be tested.

Therefore, in this study parachlorella, an alga used by PANAC Co. Ltd. to enhance the bio-energy production efficiency, was used as biosorbent. In addition, a solution of REEs containing a mixture of Y(III), La(III), Sm(III), Dy(III), Pr(III), Nd(III), and Gd(III) under various concentrations was prepared from Vietnamese rare earth ore and used for the experiment. Thus, in order to improve the sorption efficiency of parachlorella, the alga is carbonized at 250 °C and 350 °C and the REEs uptake of carbonized parachlorella is compared to the dried one. Desorption process was also evaluated and the mechanism was clearly described.

Materials and methods

Equipments

Thermogravimetric differential thermal analysis (TG-DTA) (Thermo plus EVO TG 8120, Rigaku) was carried out under nitrogen condition. The carbonization of parachlorella was made out using electric furnace (HPM-OG/AS ONE). The surface texture of the biosorbent before and after experiment was investigated using SEM (scanning electron microscope JSM-6510LA/JEOL). The zeta potential was measured by electrophoresis analysis (LS-2000E, Photal OTSUKA Electronic). The reciprocal shaking bath (FTB-01, AS ONE) was used for the batch experiment process. The pH of the solution was measured with pH meter (HM-25R, TDA DK) previously calibrated. The REEs content in treated solution was analyzed using ICP-OES (inductively coupled plasma-optimal emission spectrometer, PerkinElmer, Optima 5300). In order to point out the structure of the biosorbents and the functional group involved in the adsorption process Fourier transform infrared spectrometer (FT/IR 4100/DR410/ATR PRO 450-S) was used.

Biosorbents preparation

Parachlorella was received from Panac. Co. Ltd. in dried form (the sample was subject to spray dried for 3 days). One part of the material is subjected to the thermal decomposition analysis under N₂ atmosphere (decomposition rate 10 °C/min) on TG-DTA in order to point out the optima temperatures at which the carbonization should be made. As a result, the dried parachlorella was carbonized at 250 °C or 350 °C in the electric furnace, for 30 min. The heating was done under N₂ atmosphere (gas flow: 15 L/min) to avoid any oxygenation during the carbonization. Then the carbonized materials are desiccated until there are completely cooled. The sample was then sieved to obtain a fraction of fine particles all passing 0.6 mm.

Batch experiments

A solution containing the mixture of Y(III), La(III), Sm(III), Dy(III), Pr(III), Nd(III), and Gd(III) rare earth ions and others elements as Mg, Si, K, Ca, Mn, and Fe in diverse concentrations and initial pH = 5.63 at 30 °C is shown in Fig. 1. This solution was prepared by dissolving the Vietnamese rare earth ore in 2 wt% of ammonium sulfate aqueous solution. The mixture was kept for 60 h under room conditions. This aqueous solution (Fig. 1) was used as an initial one for the batch experiments.

Batch studies were conducted to investigate the parametric factors such as pH, solution temperature, biosorbent dosage and contact time for both biosorption and desorption processes. The pH of the solution was adjusted by using 1 mol/L of HCl or NH₃ aqueous solution. The

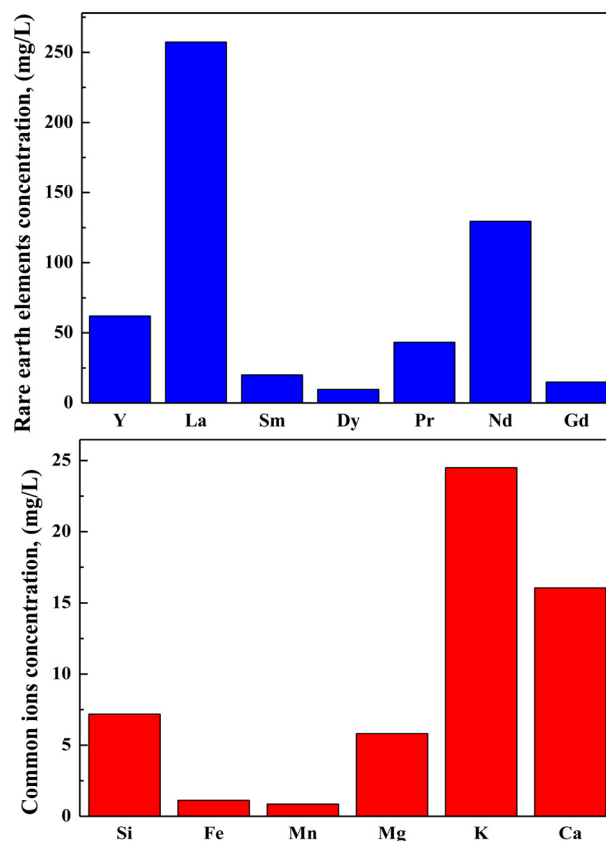


Fig. 1. Vietnamese rare earth aqueous solution composition.

biosorbent was then mixed with REEs aqueous solution, resulting in the concentration of 5 g/L. The mixture was then shaken in the bath at 30 °C and 150 rpm. The sample was filtered with 0.2 μm filter and the filtrate was analyzed using ICP-OES in terms of remaining REEs and other elements concentration. The amount of REEs or other elements adsorbed q (mg/g) was calculated by using the following equation:

$$q = \frac{V(C_{init} - C_{fin})}{M} \quad (1)$$

V , volume of the solution (L); M , mass of the biosorbent used (g); C_{init} , initial REEs or others common ions concentration (mg/L); C_{fin} , final concentration of rare earth ions or others common ions in solution (mg/L).

The percentage of removed rare earth ions or others was interpreted as:

$$\text{Removal}(\%) = \frac{C_{init} - C_{fin}}{C_{init}} \quad (2)$$

Samples for desorption experiments were prepared by putting 2 g of biosorbents and 100 mL of REEs solution into contact for 2 h (to saturate as much as possible the available biosorbents sites with rare earth ions). The biosorption process was conducted at pH 7 and 30 °C until equilibrium was reached. The solution was analyzed using ICP-OES and Eq. (1) helps to determine the desorption density at equilibrium q_0 for the selected biosorbents. The REEs loaded samples, which will be subjected to desorption experiment were dried in the oven at 80 °C for 72 h.

0.05 g of the prepared sample was mixed with 10 mL of HCl, HNO₃, and H₂SO₄ (various concentration of acid have been used) and shaken at various temperatures depending on the parameter study. The solution obtained was analyzed using ICP-OES and the results

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