

# Biosorption of yttrium, lanthanum, cerium, and neodymium from apatite leaching aqueous solution using *platanus orientalis* leaf powder

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## Abstract

In this work, *platanus orientalis* (*P. orientalis*) leaf powder was used to extract yttrium, lanthanum, cerium, and neodymium from aqueous solution produced by nitric acid leaching of apatite concentrate. The effects of pH, contact time, temperature, and quantity of biosorbent used in the sorption process, as well as the concentration of hydrochloric acid in the desorption stages, were investigated. The results show that the leaf powder can take up 23.0, 6.0, 6.9, and 3.2 mg/g of cerium, lanthanum, neodymium, and yttrium, respectively, from the aqueous solution; extractions of 97% of the cerium, 82% of the lanthanum, 87% of the neodymium, and 51% of the yttrium were achieved. Hydrochloric acid can desorb about 99% of the lanthanum and yttrium, 98% of the neodymium, and 97% of the cerium from *P. orientalis*. According to thermodynamic studies, the values of  $\Delta H^\circ = 0.54, 4.76, 0.22, \text{ and } 0.23 \text{ kJ/mol}$  and of  $\Delta G^\circ = -9.12, -9.48, -9.44, \text{ and } -7.64 \text{ kJ/mol}$  (at  $30^\circ\text{C}$ ) for La, Ce, Nd, and Y, respectively, suggest that the biosorption of La, Ce, Nd, and Y on *P. orientalis* leaf powder is an endothermic, spontaneous process. The results indicate that *P. orientalis* is a suitable biosorbent for the extraction of rare earth elements from apatite leaching aqueous solution.

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## 1. Introduction

The rare earth elements are a group of 17 chemically-similar, metallic elements, including scandium and yttrium, with atomic numbers of 21 and 39, respectively, and the lanthanides with atomic numbers 57 to 71 (Spedding 1978).

The rare earths are typically soft, malleable, ductile, and usually reactive. They have an ever-increasing variety of applications in modern technology. These applications range from mundane applications (lighter flints, glass polishing) to high-technology applications (phosphors, lasers, magnets, batteries, magnetic refrigeration) to futuristic applications (high-temperature superconductivity, safe storage, and transport of hydrogen in the post-hydrocarbon economy) (Haxel et al., 2002).

The most common minerals that contain rare earth elements are apatite, monazite, and xenotime. The general formula for apatite is  $\text{Ca}_{10}(\text{PO}_4)_6\text{X}_2$ , where X can be a fluorine ion, a chlorine ion, or a hydroxyl group (Martina et al., 1999). Apatite, which contains an average of 0.1 - 0.8% of rare earth oxides, is the main source of phosphate fertilizers and phosphoric acid (Li et al., 2005).

Nitric, sulfuric, and hydrochloric acids have been used as leaching agents for the extraction of rare earth elements from apatite.

Usually, the rare earths are recovered from leach liquor by solvent extraction and finally precipitated as rare earth oxalates from the strip solution. The oxalates are calcined in a furnace to yield a mixture of rare earth oxides. Also, fractional crystallization, fractional precipitation, ion exchange, and selective oxidation or reduction are used for separating individual rare earths (Gupta and Krishnamurthy, 2005). All of these methods have some advantages, as well as disadvantages, such as disposal of harmful materials in the environment.

Biosorption is a symbol of biotechnological novelty as well as a cost-effective method for recovery of metals from industrial effluents and aqueous solutions using certain types of inactive or dead microbial biomass that bind and concentrate metal ions in their cell walls (Dobson and Burgess, 2007, Mao et al., 2009). Dead biomass, such as tree leaves, sawdust, bark, and cone biomass are used for biosorption

processes (Aoyama, 2003). These products, which are relatively inexpensive and available in large quantities, eliminate any nutrient requirement and can be exposed to high-toxicity environments (Volesky, 1990).

Palmieri et al. (2002) used *Sargassum fluitans* for lanthanum recovery from a synthetic aqueous solution and investigated the dynamics of the biosorption process, the influence of pH, and the desorption process. It was reported that biosorption by non-living *Sargassum fluitans* is a good alternative method for lanthanum recovery from solutions.

Diniz and Volesky (2005) applied *Sargassum polycystum* Ca-loaded biomass for biosorption of lanthanum, europium, and ytterbium from single-component and multi-component batch systems. For the multi-component mixtures, the metal affinity sequence established was  $\text{Eu} > \text{La} > \text{Yb}$ , and the maximum metal uptakes obtained were 0.29, 0.41, and 0.28 m mol g<sup>-1</sup> for La, Eu, and Yb, respectively.

Diniz et al. (2008) studied the biosorption of lanthanum and europium using protonated *Sargassum polycystum* biomass in batch and column systems. It was found that *Sargassum polycystum* biomass could be used in at least three consecutive uptake/desorption cycles with no loss of metal uptake capacity, and it can be regenerated by 0.1 N HCl.

Tree leaves have been used extensively for the recovery of heavy metals, such as chromium (Aoyama, 2003; Aoyama et al., 1999; Aoyama et al., 2000), cadmium (Sharma and Bhattacharyya, 2005), and lead (Bhattacharyya and Sharma, 2004; Al-Subu, 2002), from aqueous solutions.

Sert et al. (2008) used leaf powder made from *P. orientalis* for the biosorption of lanthanum and cerium from a synthetic aqueous solution. The effects of initial pH, contact time, initial metal ion concentration, and temperature were investigated and optimized. The Langmuir monolayer capacity of the sorbent was reported as 28.65 mg g<sup>-1</sup> and 32.05 mg g<sup>-1</sup> for La and Ce, respectively.

*P. orientalis*, a tree in the Platanaceae family, has been an important tree in Persian gardens from the earliest days. There, it is known as the Chenar. Iran is an rare earth element importer country. One of the by-products of the Chadormalu iron ore concentrator plant is apatite concentrate, which is rich in rare earth

elements (Jorjani et al., 2007; Jorjani et al., 2008). The objectives of the present work are: (a) to use the powdered leaves of *P. orientalis* as a biosorbent for recovering yttrium (Y), lanthanum (La), cerium (Ce), and neodymium (Nd) ions from aqueous solution produced by nitric acid leaching of apatite concentrate; (b) optimization of the effects of pH, contact time, temperature, and quantity of biosorbent in sorption, as well as the hydrochloric acid concentration in the desorption stages; (c) to determine thermodynamic parameters and conduct isotherm investigations. This is the first time the extraction of the mentioned REEs directly from apatite leach solution has been investigated using *P. orientalis* and reported in the literature.

## 2. Materials and methods

### 2.1. Sampling and preparation of pregnant liquor

The examined sample was prepared from apatite concentrate obtained from the Chadormalu plant; the apatite concentrate is a byproduct of iron-ore production in the Chadormalu iron-ore concentrator plant. A representative sample, for which 80 wt% of the sample was made up of particle sizes that were less than 50  $\mu\text{m}$ , was used for the characterization and leaching studies without any further size reduction. The results of the assay of the light and heavy rare earth elements in the apatite concentrate are shown in Tables 1 and 2. As can be seen, La, Ce, Nd, and Y are the major rare earth constituents, so the evaluation of their extraction behavior from the aqueous phase is the subject of the current work.

Table 1. Assay of light REEs in the apatite concentrate

Light Rare earth elements	La	Ce	Pr	Nd	Sm	Eu	Total
Assay (ppm)	1514	4204	455	1738	293	24.5	8228.5
Assay (%)	18.39	51.1	5.52	21.12	3.58	0.29	100

Table 2. Assay of heavy REEs in the apatite concentrate

Heavy Rare earth elements	Gd	Tb	Dy	Ho	Er	Tm	Yb	Lu	Y	Total
Assay (ppm)	233	28.9	145	24.2	63.2	7.76	40.9	4.94	855	1402.9
Assay (%)	16.61	2.07	10.34	1.73	4.51	0.55	2.9	0.35	60.94	100

The apatite sample was leached by using a solution of 60% nitric acid at 60 °C and a solid-to-liquid ratio of 30% with agitation for 30 minutes at 200 rpm (Jorjani et al, 2008). The slurry was filtered to remove the insoluble constituents; the pregnant liquor (phosphoric acid) that contained the dissolved rare earth elements was recovered. The chemical analysis of the pregnant liquor is shown in Table 3. As can be seen in Table 3, the pregnant liquor contains different unwanted ions, such as  $\text{Ca}^{+2}$ ,  $\text{Mg}^{+2}$ , and  $\text{Fe}^{+3}$ , that can influence the rare earth elements biosorption process; therefore, the precipitation of impurities (mainly as hydroxides, oxides, and hydrated nitrates) was evaluated with the  $[\text{H}^+]$  values of the pregnant liquor at 10, 1, 0.1, 0.01, 0.001, and 0.0001, and the precipitate was centrifuged. Maximum precipitation of impurities and minimum precipitation of rare earth elements were used as evaluation criteria; finally pH = 3 was selected as the optimum pH value for the precipitation of impurities; Table 4 shows the amounts of impurities and REEs removed with the precipitation at pH = 3.

Table 3. Chemical analysis of pregnant liquor

Element	Ca	Fe	Mg	P	S	F	La	Ce	Nd	Y
Content (ppm)	100000	1280	2450	56400	1015	6880	362	1221	394	290

Table 4. Removal of impurities from pregnant liquor using precipitation at pH = 3

Element	Ca	Mg	Fe	P	La	Ce	Nd	Y
Removal (%)	97	83	99	99	19	22	20	14

## 2.2. Preparation of *P. orientalis* leaf powder

The leaves of *P. orientalis* used in this study were collected from trees at Ekbatan town, Tehran, Iran. They were washed with deionized water to remove dust and impurities, dried at 80 °C for 24 h, and ground to -125 µm powder (Sert et al., 2008). Calcium ions tend to compete with rare earth elements in solution to absorb on the biosorbent; so, the biomass was then loaded with calcium in a solution of 1000 mgL<sup>-1</sup> CaNO<sub>3</sub> for 12 hr under mild agitation. Later, the biomass was washed with distilled, de-ionized water to remove excess Ca ions. Finally, the biomass was dried again overnight at 55 °C and stored in a desiccator prior to use.

## 2.3. Metal biosorption capacity and distribution coefficient

The biosorption capacity of *P. orientalis* leaves (Q) and distribution coefficient (K<sub>d</sub>) of metal ions between the solid and aqueous phases can be calculated using the following equations (Sert et al., 2008):

$$Q = (C_i - C_e) \times V/M \quad (1)$$

$$K_d = ((C_i - C_e)/C_i) \times V/M, \quad (2)$$

where Q is the metal sorption capacity (mg/g), C<sub>i</sub> and C<sub>e</sub> are initial and equilibrium metal concentrations in the solution (mg/l), respectively, V is the solution volume (L), and M is the mass of biosorbent (g).

## 3. Results and discussion

### 3.1. Effect of initial pH

The effect of pH on metal biosorption has been studied by many researcher (Holan and Volesky, 1994; Kratochvil et al., 1998; Tsezos and Volesky, 1981; Guibal et al., 1992; Darnall et al., 1986; Aksu and Kutsal, 1991; Garnham et al., 1993; Holan et al., 1993; Kuyucak and Volesky, 1989), and the results demonstrated increasing cations uptake with increasing pH values. The pH can affect the degree of ionization of metal ions in solution and the surface charge of the biosorbent (Aksu and Isoglu, 2005).

Sert et al. (2008) studied the effect of pH on the biosorption of La and Ce from synthetic aqueous solutions by *P. orientalis* leaf powder; the highest biosorption was obtained at pH = 4.

In this work, the pH of the aqueous solution was adjusted to 0, 1, 2, and 3 using nitric acid and ammonia solution, and the biosorption experiments were conducted using 10 ml of the pregnant liquor and 0.2 g of biosorbent at a temperature of 30 °C, a contact time of 2 h, and a mixing rate of 150 rpm. Fig.1 shows the effect of pH on the biosorption capacity of *P. orientalis*. It is apparent that pH is an important parameter for the sorption process. The REEs biosorption capacities were higher at pH = 3. (At pH = 4 or greater, the precipitation of REEs was significant.)

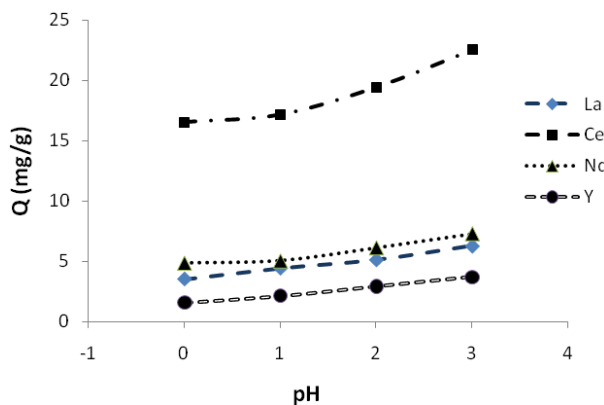


Fig. 1. Effect of initial pH on biosorption of REEs by *P. orientalis* leaf powder; 10 ml of pregnant liquor and 0.2 g of biosorbent at a temperature of 30 °C, a contact time of 120 min, and a mixing rate of 150 rpm

### 3.2. Effect of temperature

The biosorption of cadmium (II) ions by *C. vulgaris* was studied by Aksu (2001); it was shown that the algal biomass exhibited the highest cadmium (II) uptake capacity at 20 °C. Kutahyali et al. (2010) studied La and Ce biosorption on *Pinus brutia* leaf powder and showed that their uptakes increased slightly with increasing temperature. Takahashi et al. (2005) studied the adsorption of rare earth elements onto bacterial cell walls and showed that the distribution coefficient of rare earth elements between the bacterial and the aqueous phases increased with increasing temperature.

In the current work, the effects of different temperatures (20, 30, 40, and 50 °C) on the biosorption capacity of *P. orientalis* were examined. The other variables were kept constant, and the pH was adjusted

to pH = 3. Fig. 2 shows that the biosorption capacity of cerium increased and those of lanthanum, neodymium, and yttrium remained virtually unchanged as the temperature was increased.

The Van't Hoff equation was used to measure the enthalpy of adsorption. It relates the change in temperature ( $\Delta T$ ) to the change in the equilibrium constant ( $\Delta K$ ) given the standard enthalpy change ( $\Delta H^\ominus$ ) for the process. Since:  $\Delta G^\ominus = \Delta H^\ominus - T\Delta S^\ominus$ ,  $\Delta G^\ominus = -RT \ln K$ , it follows that:

$$\ln K = -\frac{\Delta H^\ominus}{RT} + \frac{\Delta S^\ominus}{R}, \quad (3)$$

where  $\Delta S^\ominus$  is the standard entropy change; therefore, a plot of the natural logarithm of the equilibrium constant versus reciprocal temperature gives a straight line. The slope of the line is equal to the negative standard enthalpy change divided by the gas constant,  $-\Delta H^\ominus/R$ , and the intercept is equal to the standard entropy change divided by the gas constant,  $\Delta S^\ominus/R$ . The values of  $\Delta H^\ominus$ ,  $\Delta S^\ominus$ , and  $\Delta G^\ominus$ , which were calculated from the information presented in Fig. 3, are presented in Table 5 for the biosorption of lanthanum, cerium, neodymium, and yttrium. As shown, the values of  $\Delta H^\ominus = 0.54, 4.76, 0.22$ , and  $0.23$  kJ/mol and  $\Delta G^\ominus = -9.12, -9.48, -9.44$ , and  $-7.64$  kJ/mol for La, Ce, Nd, and Y, respectively, at  $30^\circ\text{C}$  suggest that the biosorption of La, Ce, Nd, and Y on *P. orientalis* leaf powder is an endothermic, spontaneous process. Also, during the adsorption process, the positive sign of  $\Delta S^\ominus$  causes an increase in the entropy (Ünlü and Ersoz, 2006).

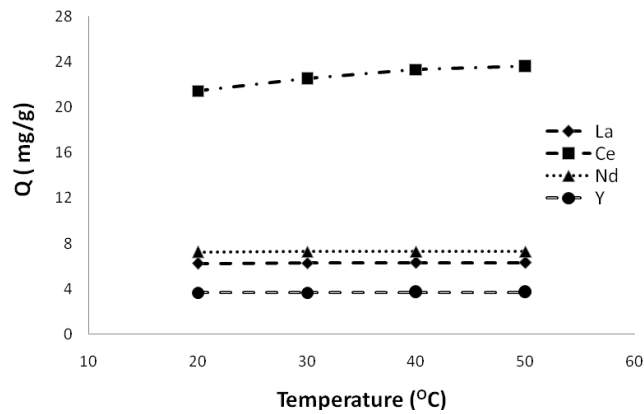


Fig. 2. Effect of temperature on biosorption of REEs on *P. orientalis* leaf powder; 10 ml of pregnant liquor, 0.2 g of biosorbent, pH = 3, contact time of 120 min, and a mixing rate of 150 rpm



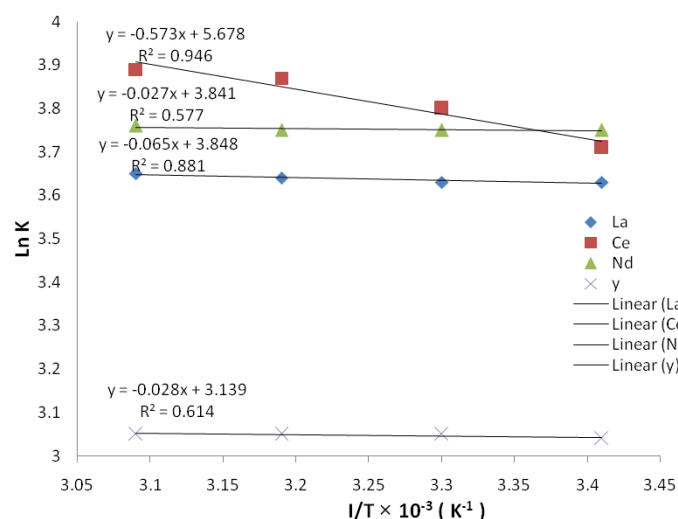


Fig. 3. Ln K vs. 1/T for the calculation of thermodynamic parameters for the biosorption of La, Ce, Nd, and Y on *P. orientalis* leaf powder

Table 5. Thermodynamic parameters of La, Ce, Nd, and Y biosorption on *P. orientalis*

	$\Delta H^\circ$ (kJ/mol)	$\Delta S^\circ$ (kJ/mol K)	$\Delta G^\circ$ (KJ/mol)			
			293 °K	303 °K	313 °K	323 °K
La	0.54	0.0319	- 8.80	- 9.125	-9.44	- 9.763
Ce	4.76	0.047	-9.011	-9.481	-9.951	-10.42
Nd	0.22	0.0319	-9.126	-9.445	-9.764	-10.083
Y	0.23	0.026	-7.388	-7.648	-7.908	-8.168

### 3.3. Effect of contact time

Sert et al. (2008) examined different time intervals, ranging from 5 to 360 min on the biosorption process for La and Ce from synthetic aqueous solutions by *P. orientalis* leaf powder; the highest biosorption occurred at a contact time of 60 min. Kutahyali et al. (2010) proposed contact times of 30 and 15 minutes for La and Ce (III) biosorption, respectively, on *Pinus brutia* leaf powder.

In this study, 10 ml of aqueous solution with pH and temperature values of 3 and 50 °C, respectively, were shaken with 0.2 g of biosorbent for different time intervals ranging from 10 to 120 min, and the biosorption capacity of *P. orientalis* leaf powder for different rare earth elements, La, Ce, Nd, and Y was evaluated. The results are shown in Fig. 4. As the Figure shows, equilibrium was reached at a contact time

of 45 min, and further increases of the contact time did not improve the biosorption capacity of the REEs because the active sites on biosorbent had been filled.

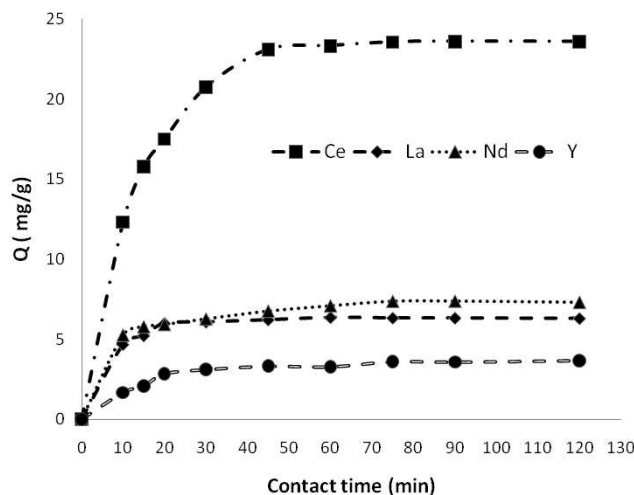


Fig 4. Effect of contact time of biosorbent and aqueous solution on biosorption of La, Ce, Nd, and Y on *P. orientalis* leaf powder

### 3.4. Effect of mass of biosorbent

In this stage, the effects of 0.2, 0.3, and 0.4 g of *P. orientalis* on the extraction of La, Ce, Nd, and Y from 10 ml of aqueous solution at a contact time of 45 min, a temperature of 50 °C, and a mixing rate of 150 rpm were investigated. The results are shown in Fig. 5. As the Figure shows, increasing the mass of *P. orientalis* leaf powder increased the percentage of REEs absorbed from the aqueous phase. Nevertheless, the capacity of biosorbent decreased (Table 6).

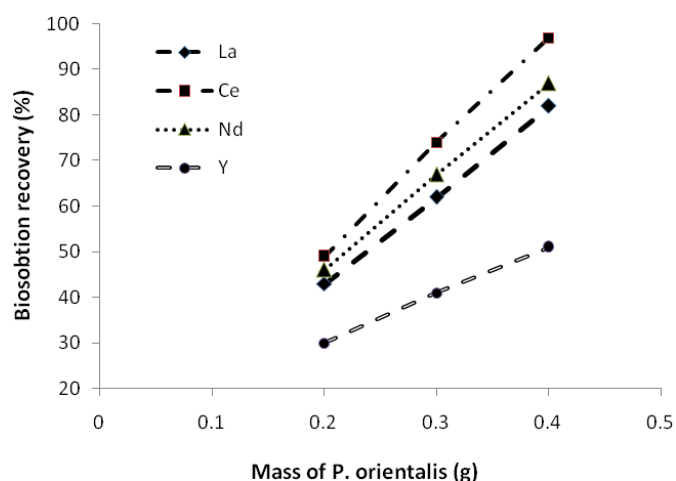


Fig 5. Effect of mass of *P. orientalis* leaf powder on biosorption recovery of La, Ce, Nd, and Y

Table 6. Biosorption capacity (mg/g) of La, Ce, Nd, and Y with different mass of *P. orientalis*

Mass of biosorbent (g)	La	Ce	Nd	Y
0.2	6.31	23.59	7.28	3.68
0.3	6.07	23.38	7.10	3.42
0.4	6.00	23.20	6.89	3.21

### 3.5. Desorption study

The biosorbent should be recovered to the maximum extent possible from the extracted valuable elements, Y, La, Nd, and Ce. In this study, HCl was used to desorb rare earth element ions from the *P. orientalis* leaf powder. The loaded biomass was suspended in 0.2, 0.3, and 0.5 N solutions of HCl for 15 minutes. The results are shown in Fig. 6, and it is evident that the 0.5 N HCl had the best recovery of REE ions from the *P. orientalis* leaf powder.

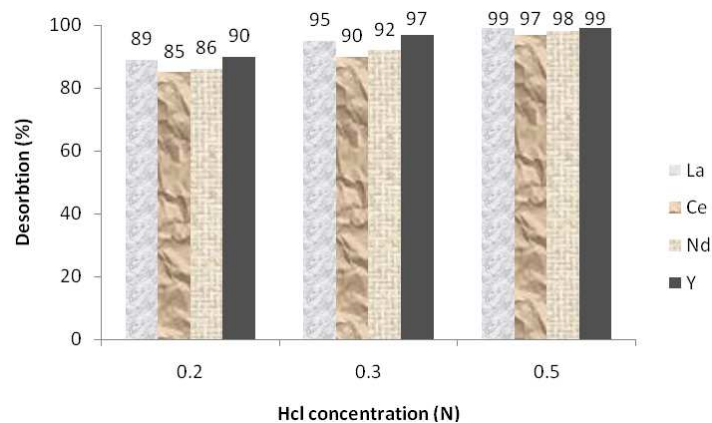


Fig 6. Effect of HCl concentration on desorption of loaded *P. orientalis*

### 3.6. Precipitation of rare earth elements

The HCl stripping solution was then subjected to precipitation of rare earth elements with 10%  $\text{H}_2\text{C}_2\text{O}_4$  solution when agitated slowly for 1 hr at pH = 1 (Le et al., 2006). Then, the precipitate was filtered and heated up to at 800 °C to obtain rare earth oxide (REO) concentrate. The REO was dissolved in nitric acid and analyzed for REEs and impurities. Table 7 shows the results of the analysis for REOs, the purity of which was greater than 86%.

Table 7. Analysis results of REO

Elements (%)	Contents (%)
REEs	86.79
Ca	10.27
Mg	2.56
P	0.1
Fe	0.01
S	0

#### 4. Conclusions

- The biosorption of four rare earth elements, i.e., Y, La, Ce, and Nd, from pregnant liquor prepared from apatite acid leaching was studied.
- The pH is an important parameter for the sorption process. The REEs biosorption capacities reached their maximum values at pH = 3.
- The biosorption capacity of cerium increased with increasing temperature, while the biosorption capacities of lanthanum, neodymium, and yttrium did not change significantly with increasing temperature.
- The thermodynamic studies showed that the biosorption of La, Ce, Nd, and Y on *P. orientalis* leaf powder is an endothermic, spontaneous process.
- The contact time of 45 min was optimized for the extraction of rare earth elements.
- Increasing the mass of the *P. orientalis* leaf powder had a positive effect on the amount of rare earth elements absorbed on the biosorbent; however, the biosorption capacity decreased.
- The best reagent for desorbing REEs ions from the *P. orientalis* leaf powder was 0.5 N HCl.
- Mixed rare earth oxides with an assay of 86.8% can be achieved after precipitation and calcination.

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