

# Hydrometallurgical digestion and leaching of Iranian monazite concentrate containing rare earth elements Th, Ce, La and Nd



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## ABSTRACT

Rare earth elements (REEs) have an ever-increasing variety of applications in modern technology. In the present work, the possibility of extraction of rare earth elements cerium (Ce), lanthanum (La), neodymium (Nd) and thorium (Th) from a monazite concentrate containing 25% rare earth elements' oxide was investigated. In this regard, the acidic digestion and leaching step, and their effective parameters were studied in detail using design of experiment (DOE) which hadn't been investigated before. The optimum conditions for the performed experiments in acidic digestion step were: temperature of 225 °C, time of 3.5 h and sulfuric acid to concentrate weight ratio (L/S) of 2.5. Using these conditions, the recovery of 92.34%, 91.44%, 92.1% and 88.94% were obtained for cerium, lanthanum, neodymium and thorium, respectively. In leaching step, the optimum conditions were: temperature of 75 °C, time of 15 h and water to digested mass weight ratio of 7.5. Using these conditions, recovery of 88.47%, 89.45%, 87.35% and 82.19% were obtained for rare earth elements cerium, lanthanum, neodymium and thorium, respectively. In precipitation step, rare earths' oxalates were precipitated from the leach liquor using oxalic acid. Then, the precipitate was calcined in a furnace at 800 °C for 1 h and finally, a mixture of rare earth elements' oxides with a purity of about 84% was obtained.

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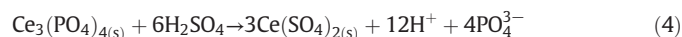
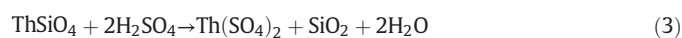
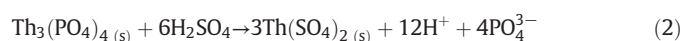
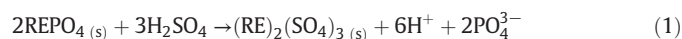
## 1. Introduction

Nowadays, the rare earth elements' (REEs) applications in industry, such as, permanent magnets, superconductors, glass polishing, and lasers (Kim and Osseo-Asare, 2012; Abreu and Morais, 2014) are increasing. Thus, separation and purification of different rare earth elements from their sources have become very important. Principal sources of REEs are bastnasite (LnFCO<sub>3</sub>), monazite (Ln, Th) PO<sub>4</sub> and xenotime (Y, Ln) PO<sub>4</sub> (Cotton, 2006; Panda et al., 2012; Abreu and Morais, 2014). Lanthanides (Ln) belong to the REEs' group and due to the similarities between these elements, the separation process is very difficult. For instance, all of them adopt 3<sup>+</sup> oxidation states in aqueous solution and the further separation will be difficult (Cotton, 2006).

There are different ways to separate the lanthanides from each other in solutions: chemical separation, fractional crystallization, ion-exchange and solvent extraction. Among them only solvent extraction is used on a commercial scale. In all of these methods, a digestion and leaching process should be done in advance. These two steps are very important because they control the concentration of REEs in the

obtained liquor, which is used for the separation/purification process. The digestion process is performed in two main paths: acidic with sulfuric acid and basic with sodium hydroxide (Gupta, 1990, Kim and Osseo-Asare, 2012). This step is then followed by a leaching process.

Monazite lattice is made of strong bonds between REEs and oxygen and therefore, they are not soluble in water. In acidic digestion process, these strong bindings are broken and soluble REEs' sulfates are formed. The following reactions occur during the digestion process (Gupta, 1990):

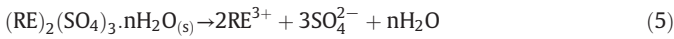


The reactions show that the dry gray digested mass is mainly consisted of sulfate salts of REEs and Th. In the leaching step, the REEs sulfates are dissolved in water; however, some of the impurities and unreacted monazite which consists of REEs phosphates and also insoluble compounds which were produced in the digestion process such as

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thorium pyrophosphate remain as a precipitate. The following reactions occur in the leaching step (Gupta, 1990):



Studies about the solvent extraction of REEs have been extensively carried out (Jorjani and Shahbazi, in press; Banda et al., 2012; Panda et al., 2012; Lee et al., 2005; El-Nadi, 2012; Ablihash et al., 2014). However, researches on the digestion and leaching step are infrequent.

The aim of present research is to investigate the optimum conditions of digestion and leaching of the solid residue from digestion process of the monazite concentrate using design of experiment. The parameters evaluated for optimum conditions of both digestion and leaching were time, temperature and liquid to solid ratio (L/S). To separate rare earths from the leach liquor, precipitation was performed by means of oxalic acid. Finally, a mixture of rare earth elements' oxides was acquired.

## 2. Materials and methods

### 2.1. Materials

The monazite concentrate sample was provided kindly by the Geological Survey of Iran. The feed of the monazite concentrate was from monazite placer deposits in Marvast, Yazd (central Iran). Microscopic studies showed that the monazite concentrate accumulates in the fine size fraction (<2 mm). The sample used in this study was taken directly from the 0–2 mm size fraction. Sulfuric acid with a purity of 93–95% (Merck) was used in both digestion and leaching experiments. Oxalic

acid with the purity of 99.6% was used in the precipitation step. De-ionized water was used in all experiments.

### 2.2. Methods

Fig. 1 shows a suggested flowsheet displaying the process. As it can be seen the process of extraction of valuable metal species from monazite includes three stages: acidic digestion, leaching and precipitation. The monazite concentrate is subjected to grinding followed by acidic digestion using sulfuric acid. The digested mass (produced in the digestion stage, sulfate cake containing REEs) is leached with deionized water followed by filtration. The leach liquor is precipitated by oxalic acid followed by filtration. The residue is calcined and a precipitate containing a mixture of rare earth oxides (REOs) is produced. In this flowsheet oxalic acid was used to precipitate the REE oxalates from the leach solution to obtain a more concentrated rare earth elements concentrate with a higher selectivity than previous researches (Jorjani and Shahbazi, in press). Besides, by oxalic acid precipitation, the impurities entering to the latter solvent extraction process will be eliminated.

#### 2.2.1. Digestion experiments

In order to investigate the optimum conditions of digestion step, certain amount of sulfuric acid was heated in a Pyrex beaker on a heater. After reaching the required temperature (based on the designed conditions in Table 1), 3 g of the monazite concentrate was added to the sulfuric acid in the beaker. A mechanical stirrer was used to agitate the mixture for a specific time. Then, the digested sample was left in air until dried and a “dry gray solid residue” or the digested mass was obtained. Afterwards, the digested mass was re-leached with deionized water in the fixed leaching parameters of liquid to solid ratio of 10:1 for 15 h in the room temperature. Then, the leach liquor was filtered with a centrifuge (EDA-20, Hettich, Germany) and finally, the leach liquor and the precipitate were analyzed with ICP.

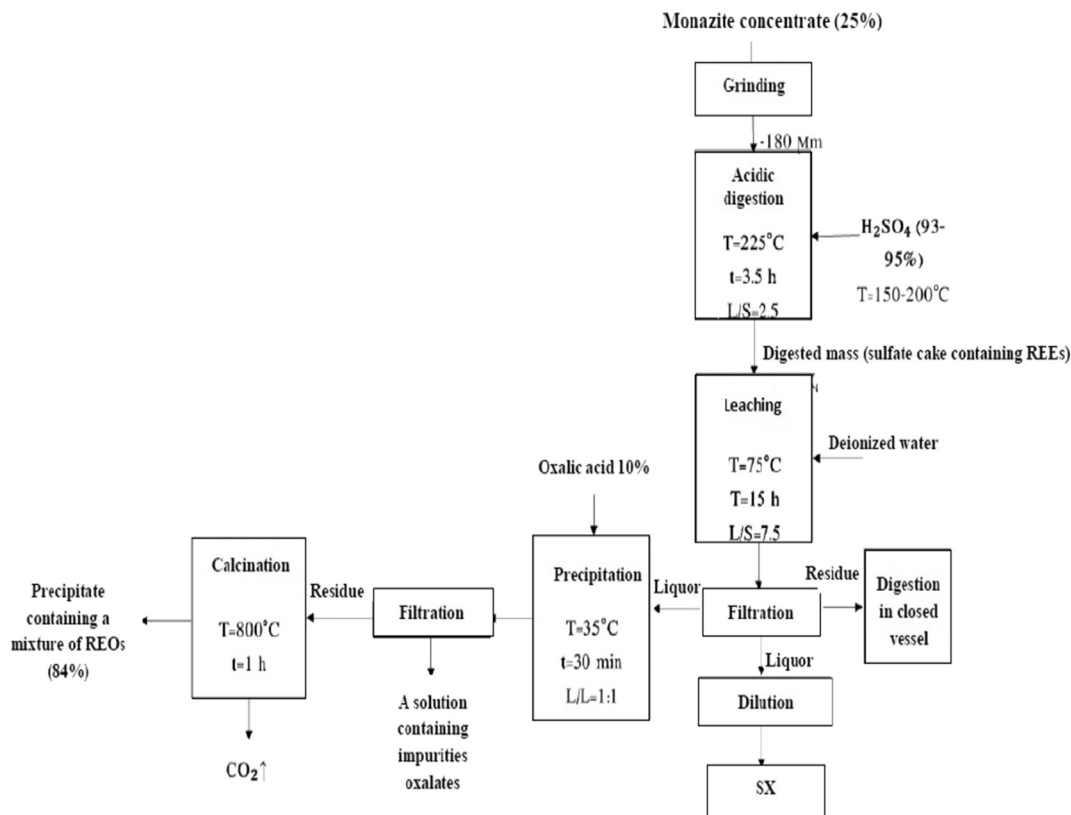


Fig. 1. A schematic flowsheet of the procedure which was applied in this work in order to produce a mixture of REOs.

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