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Cadmium extraction from phosphate ore. Effect of microwave

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Abstract This study discusses the operating variables for removal of cadmium from phosphate ore using Na₂EDTA. These variables include the reaction time, Na₂EDTA concentration, liquid/phosphate ore ratio, number of extractions and microwave extraction. Na₂EDTA induced a two-step extraction process including a rapid extraction within the first hour, and a subsequent gradual release that occurred over the following hours. The cadmium extraction efficiency increased progressively with the increasing of Na₂EDTA concentration. The extraction efficiency of cadmium increased with increasing liquid/phosphate ratio in the 5–200 range. Consecutive extractions using low concentrations were more effective than a single soil extraction with concentrated Na₂EDTA. Microwave was beneficial to improve the removal in soil washing, and using microwave could partly substitute for agitation.

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1. Introduction

Phosphate fertilizers contain traces of cadmium that can be accumulated in soil with repeated application of phosphate fertilizer. Cadmium can cause adverse animal and human

health impacts at high levels or at lower levels if exposure occurs over a prolonged period. To prevent soil from cadmium contamination of phosphate fertilizer, it is primordial to remove cadmium from phosphate ore. Two techniques are proposed to eliminate cadmium from phosphate ores: calcinations and leaching, but leaching technique is economical, better than calcinations (Keith Syers, 2001a,b). Several studies have focused on cadmium removal using various extracting agent. One of the most suitable and successfully used techniques for the treatment of soils contaminated with heavy metals on-site ex situ is soil washing technique. Soil washing often needs the use of different extracting agents for heavy metals decontamination. These agents include acids, bases, chelating agents, electrolytes, oxidizing agents and surfactants (Hong et al., 1995; Schramel et al., 2000; Reddy and Chinthamreddy, 2000; Sun et al., 2001). Acid washing leads to a decrease of soil productivity and a number of changes in the chemical and physical structures of soils due to mineral dissolution (Reed

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et al., 1996). Chelating agents, such as ethylenediamine tetraacetic acid (EDTA), nitriloacetic acid (NTA), diethylenetriamine pentaacetic acid (DTPA) and S,S-ethylene-diaminedisuccinic acid (EDDS) are considered as the most attractive alternative because they can form strong metal-ligand complexes and are thus highly effective in remediating heavy metal-contaminated soils (Norvell, 1984; Elliott and Brown, 1989; Kim and Ong, 1998). EDTA is also extensively used for soil remediation because of its ability to mobilize metal cations efficiently coupled with only a minor impact on the physical and chemical properties of the soil matrix (Lee and Marshall, 2002). Studies focused on the cadmium extraction from phosphate ore, which would be reduced to lower rates, were rarely investigated by researchers so far. Our study was carried out to discuss a scheme in order to assess the suitability of Na₂EDTA operating variables and compare cadmium extraction efficiencies in different washing conditions. The influence of some parameters was taken into consideration, such as liquid/phosphate ratio (*L/P*), reaction time, concentration of Na₂EDTA, number of extractions and microwave extraction.

2. Experimental

2.1. Materials

The phosphate samples were collected from Djebel El Onk, Tebessa north east Algeria. The samples are ground to less than 2 mm particle size. All the chemicals used in the experiments were in pure form (Merck and Sigma brand). Bidistilled water was also used in all experiments.

2.2. Analytical procedures

2.2.1. Mineralization

Before the determination of the phosphate ore chemical composition, the acid digestion (HF/HClO₄/HNO₃) was added to 1.00 g of the phosphate samples, the suspension was then centrifuged at 3000 rpm for 15 min in EBA 20 Hettich centrifuge; the supernatant was filtered through Whatman filter paper (45 μm) and then analyzed.

2.2.2. Extraction procedure

The extraction tests were conducted in 25 mL beaker. 1.00 g of the sample and a measured volume of EDTA was agitated using an end-over-end shaker at a speed of 400 rpm at room temperature (25 °C) for a given time. The suspensions were centrifuged at 3000 rpm for 15 min and the supernatants were then filtered through a 45 μm membrane for heavy metal analysis.

The washing solution was prepared from analytical grade reagents. All tests were performed in triplicates and the results were presented as averages of the triplicate extracts. As a typical experimental procedure, the mixture of 1.00 g phosphate ores and volume of Na₂EDTA was treated by microwave irradiation in a domestic microwave oven for pre-set time. During the test processes, the samples were obtained at a predetermined time, and the solution samples were analyzed for cadmium.

2.2.3. Analysis methods

Cd, Pb, Cr concentrations were measured by absorption atomic spectrometry (spectrometer Shimadzu AA 6200). A UV-visible (Jenway) spectrophotometer was used for phosphate analysis according to the standard methods (vanadomolybdophosphoric acid colorimetric method) (Olsen and Sommers, 1982). Ca, Mg, and Fe were measured by fluorescence X spectrometry.

3. Results and discussion

3.1. Composition of phosphate ore

The analytical results of some elements in the phosphate ore are presented in Table 1. The content of cadmium in phosphate is 16.5 ppm. This level exceeds the recommended value (10 ppm). In this work, we will study the possibility of reducing the cadmium content by simple extraction and microwave assisted extraction.

3.2. Conventional cadmium extraction

3.2.1. Influence of consecutive extractions

Although Na₂EDTA could remove cadmium from the phosphate ore, it is not adequate to completely decontaminate the mineralization. To remove more cadmium, three consecutive extractions with 0.25 M Na₂EDTA (*L/S* = 25, 2 h) were carried out on the same sample. The results are illustrated in Fig. 1.

Renewing the extracting solution greatly increased the dissolution of cadmium; the cumulative removals of cadmium from phosphate after 1st and 3rd extraction cycles were 38.9% and 55.9% respectively. Three reasons that explain the greater removal of cadmium with the increasing renewal of the solution: (1) the removal of some residual metal chelates which may be trapped in the soil pores (Sun et al., 2001) or re-adsorbed onto the sediment particles during the first step of the sequential extraction procedure; (2) the reduction the interference in metal-chelant complex formation after removing the major content of the competition cations in the first steps (Poletini et al., 2007); and (3) the reduction of readsorption of the desorbed metals and increase of the dissolution rate of metal-containing particles by maintaining undersaturated conditions (Strawn and Sparks, 2000). As the number of consecutive extractions increased, only a small percentage of the total cadmium was extracted. After the previous extractions where the mobile forms were extracted, the Metal was released from the less mobile forms (i.e., bound to organic matter, bound to Fe/Mn oxides or residual fraction). This is presumably related to the dissolution of mineral constituents of sediment (including oxides and silicates) initially retaining the contaminants (Poletini et al., 2007).

3.2.2. Influence of concentration

The effect of Na₂EDTA concentration on the rate of cadmium extraction was tested using a concentration range of 0.01–0.25 M. It was noticed that the extraction of cadmium increased with increasing Na₂EDTA concentration and reached 36.6% (Fig. 2). From Fig. 2, it can be seen that when the concentration of Na₂EDTA varies from 0.01 to 0.125 M, the concentration of the extracted cadmium increases accordingly and becomes

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