



Separation of rubidium from boron containing clay wastes using solvent extraction



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ABSTRACT

Rubidium was predominately identified in boron clay (BC) as a result of the analyses of trace elements in boron industrial wastes that were investigated. In the first step, rubidium in the BC was removed from the solution by leaching in 1 M H₂SO₄ for 2 h. After a filtering operation, rubidium was separated from the solution by precipitation in the form of tetraphenylborate. In the second step, rubidium was extracted with 18-Crown-6 (0.1 M) in nitrobenzene from picric acid and stripped with 2 M of hydrochloric acid from the organic phase. 89.52% of the best rubidium extraction was achieved.

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1. Introduction

As mining activities increase, the amount of waste materials readily increases. During the beneficiation of valuable metals and industrial minerals from their ores, large volumes of waste materials or tailings may be produced and these tailings may be harmful to the environment [1]. Of the boron mines operating in the United States, taking the necessary measures to prevent damage to the environment is one of the leading concerns [2]. Our country is rich in boron reserves and during the processing of ore in concentrators, large quantities of boron wastes are discharged. Consequently, many researchers have carried out the evaluations of wastes.

Investigations on boron tailings demonstrate that the most suitable way to evaluate wastes is first to recover boron minerals from tailings and then utilize the remainder of the mainly clay minerals in suitable sectors [3]. Recovering boron from boron wastes by leaching using various solutions [4–6] was studied. Many researchers use boron wastes as an additive material in some sectors like cement [7,8], concrete [9], brick [10–14], tile [15,16], and ceramic [17,18]. Several studies demonstrate that boron wastes have been also used for absorption [19,20]. Other studies on boron aim to determine the trace elements in the structure of the boron minerals and clays. Helvacı et al. differentiated Emet borate deposits and they reported high Ca borate (colemanite) levels, very low

Na levels and relatively high concentrations of Mg, Sr, As and S [21]. The clay mineralogy and whole-rock chemistry of the borate-bearing layers of the Hisarcık and Espiye mines were examined and the clay fraction of the studied profile contains predominantly Li-bearing saponite that accounts for 60–90 wt.% of the clay fraction (<2 μm) by Çolak et al. [22]. Büyükburç et al. aimed to achieve cost reduction by extracting lithium from boron clays using natural (limestone–clay) materials and the wastes of boric acid were used as a source of gypsum [23].

Rubidium is used interchangeably or together with cesium in many applications. Its principle application is in specialty glasses used in fiber optic telecommunication systems. Rubidium's photoemissive properties have led to its use in night-vision devices, photoelectric cells and photomultiplier tubes. It has several uses in medical science, such as in positron emission tomographic (PET) imaging, the treatment of epilepsy and the ultracentrifugal separation of nucleic acids and viruses. It has been used in electron tubes as a deoxidizer for research and development studies and as a fuel in plasma jet engines. A dozen or more other applications are known, that include its use as a catalyst for several organic reactions and in frequency reference oscillators for telecommunication network synchronization [24–26].

Although rubidium is more abundant in the earth's crust than copper, lead or zinc, it forms no minerals of its own. It is, or has been, produced in small quantities as a byproduct of the processing of cesium and lithium ores taken from small deposits in Canada, Namibia and Zambia [27]. Ion exchange, precipitation, and extraction are the three main methods to recover rubidium from muscovite ore [28,29]. Shan et al. achieved 90.1% Rb extraction for the mixture roasted with a

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mass ratio of muscovite/NaCl/CaCl₂ of 1.00/0.25/0.25 at 850 °C for 30 min [30]. Direct acid leaching and chlorination roasting-waste leaching were used to leach rubidium from kaolin clay waste by Libo Zhou et al. [31]. M.R. Tavakoli Mohammadi et al. extracted rubidium from gold waste with a three-step process (acid washing, followed by salt roasting and water leaching) [32].

Solvent extraction is a basic separation process to isolate elements in a substance next to the unwanted contaminants. Industrial application of solvent extraction first began with uranium, but now has an important place in nuclear applications and analytical chemistry. Its selectivity is due to its solvent extraction ability that outclasses other separation methods such as precipitation and ion exchange [33].

In the present study, we attempted to obtain rubidium (detected in BC as the result of analyses) by using a solvent extraction method with crown ether.

2. Experimental

2.1. Materials, reagents and the characterization of BC

18-Crown-6 (1,4,7,10,13,16-Hexanoxacyclooctadecane; MW = 264,32 g/mol), picric acid (2,4,6-Trinitrophenol; MW = 229,11 g/mol) were purchased from Sigma-Aldrich; RbCl (MW = 120,92 g/mol) was purchased from Merck. Other chemicals such as nitrobenzene, benzene, toluene, carbontetrachloride, chloroform were reagent grade. Doubly distilled deionized water was used in the preparation of the solutions. The chemical structure of 18-Crown-6 and picric acid are given in [Schema 1](#). BC was obtained from the Emet Etibor Factory (Espey region) in Turkey. The BC was dried at 110 °C for 2 h in an oven and ground in a ring mill at under 63 μm. The analysis of the chemical composition of the BC was performed using XRF and AAS. XRD was used for determining the mineral phases.

2.2. Solvation of the BC

Optimum parameters of solvation are leaching in 1 M H₂SO₄, with a 0.02 solid/liquid ratio for 2 hours in a shaker with a mixing speed of 200 rpm at room temperature. After the filtration of the solution, the concentration of rubidium was analyzed using ICP-OES (Perkin Elmer 4300DV). This part will be discussed in a separate paper.

2.3. Methods

A total volume of 10 mL of 0.05 M picric acid solution containing 5 mg of rubidium chloride was prepared and transferred into a separating funnel (aqueous phase). 18-Crown-6 was dissolved in 10 mL of 0.1 M nitrobenzene (organic phase) and was transferred into a separating funnel as well. The mix was extracted twice by strongly shaking the solution together for 5 min. The two phases were allowed to settle and separate. The extracted rubidium concentration in the organic phase was calculated by the determination of rubidium in the aqueous phase using ICP-OES. The organic phase was taken into the another separating funnel, 10 mL 2 M hydrochloric acid was added (aqueous phase) and

extracted samely to strip the rubidium from the organic phase. The back extracted rubidium in the aqueous phase was also determined by ICP-OES. All the analytical results, expressed as ppm are mg/L.

We investigated the effects of the operational conditions; pH, a concentration of crown ether, a concentration of picric acid, kind of diluent, and kind of stripping acid.

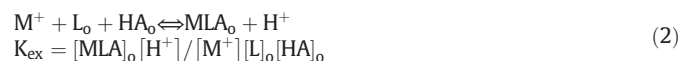
3. Theory and calculation

3.1. Mechanism of the extraction

For practical purposes, as in industrial applications, it is often more popular to use the percentage extraction %E (sometimes named the extraction factor), which is given by:

$$\%E = 100D/D + (V_a/V_o). \quad (1)$$

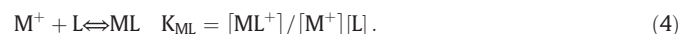
V_a and V_o are the volumes of the aqueous and organic phases respectively. If the volume in the two phases were equal, the denominator would be “D + 1”. If the extraction efficiency approaches 100%, then the dispersion ratio approaches infinity. The extraction yields ranging from 99 to 100% mean that the dispersion ratio is variable from 99% to infinity. It is observed that with widely varying disintegration rates, the nearly completed extraction would be incorrect [34]. The overall reaction equilibrium between an aqueous phase containing the univalent metal ion M⁺, a picrate anion A⁻, the hydrogen ion H⁺ and an organic phase of the crown ether L may be represented by the following equations [35–39].



where MLA represents the ion pair between a univalent metal-crown ether cation, an ML and the picrate anion; the subscript “o” and the absence of subscript denote the organic phase and the aqueous phase, respectively. The distribution of the free crown ether between the two phases is:

$$L \rightleftharpoons L_o \quad K_{D,L} = [L]_o / [L]. \quad (3)$$

The complex reaction of the univalent metal ion with the crown ether



The distribution of picric acid between the two phases



The extraction of picric acid

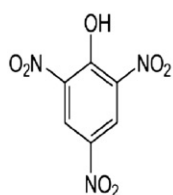


K'_{ex} can be written by using Eqs. (2) and (6)

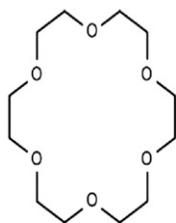
$$K'_{ex} = [MLA]_o / [M(L)^+] [A^-].$$

Table 1
Chemical compositions of BC (mass fraction %).

Na ₂ O	MgO	Al ₂ O ₃	SiO ₂	P ₂ O ₅	SO ₃	Cl
1.300	8.138	10.540	44.150	0.165	0.414	0.002
K ₂ O	CaO	TiO ₂	MnO	Fe ₂ O ₃	B ₂ O ₃	Loss on ignition
4.010	1.783	0.530	0.101	5.403	3.060	20.400



A



B

Schema 1. Chemical structure of picric acid (A) and 18-Crown-6 (B).

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