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Dissolution of Uranium and Rare Earth Elements from a Low-Grade Phosphate Ore Using Different Acids

Farkli Asitler Kullanilarak Düşük Tenörlü Fosfat Cevherinden Uranyum ve Nadir Toprak Elementlerinin Çözündürülmesi

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ABSTRACT

Herein, it was aimed at determining the best leaching agent for extracting uranium (U) and rare earth elements (REEs) from a low-grade phosphate ore. Before leaching tests, the beneficiation of U and REEs contents in the raw ore were investigated by using the Falcon concentrator. The U content in the preconcentrated ore increased to 1629.66 ppm from 392 ppm with a recovery of 92% and the amount of REEs was found to be 747.20 ppm. These values suggested that the preconcentrated ore can be used as REEs source due to its content. It was then dissolved in different acid mediums at a temperature of 40 °C for 2 hours to evaluate the effect of $HClO_4$, CH_3COOH, H_3PO_4, HCl, and HNO_3 on the extraction of U and REEs. Among all leaching agents, the use of H_3PO_4 as the leaching agent suggested more promising results compared to others at the fixed experimental conditions. The influences of leaching temperature and acid concentration were also studied and the optimal experimental conditions to dissolve all of U and REEs from the ore are as follows: H_3PO_4 concentration of 4 M, temperature of 50 °C, time of 4 hours.

Keywords: Uranium, Rare Earth Elements (REEs), Acid Leaching, Phosphoric Acid

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INTRODUCTION

Phosphate rock is listed as one of the most strategic raw materials depending on its supply risk versus economic importance (Commission 2017). It contains a considerable amount of uranium (U, 0.005 – 0.02%) together with rare earth elements (REEs, 0.05%) (Paschalidou and Pashalidis 2019). It is known that U recovery from a sedimentary phosphate rock was conducted on an industrial scale until the late 1990s. Since then, it has not been preferred to be used as U sources due to the decrease in the price of U (Steiner et al. 2020). Nowadays, the demand for U causes an increase in its price and it is expected that this demand will tend to grow in the near future. Furthermore, the recovery of U from phosphate ores is not only for economic reasons but also for the protection of human health (Haneklaus et al. 2017).

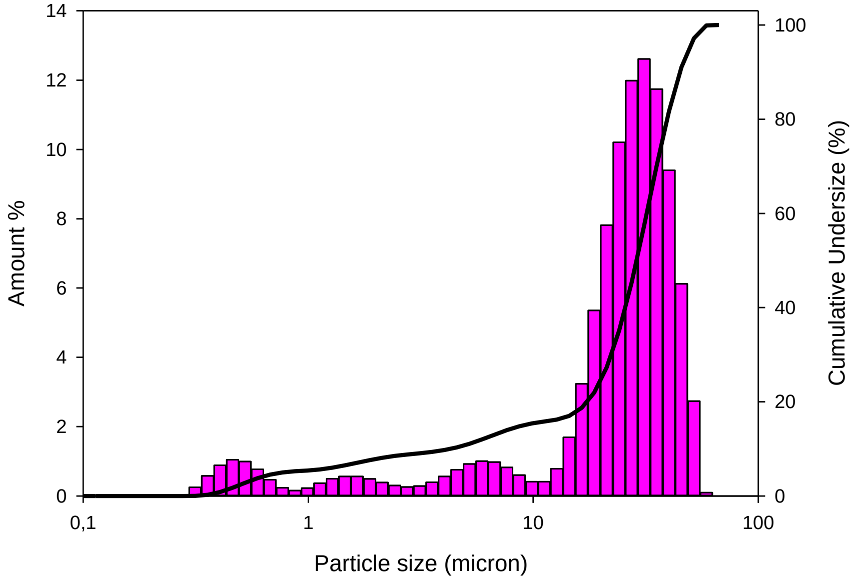

Figure 1. The particle size distribution of the sample prepared for use in the Falcon concentrator

Şekil 1. Falcon konsantratörü için hazırlanan numunenin tane boyut dağılımı

Many studies have been conducted to evaluate the potential of phosphate rocks in terms of their U content and import\export amounts (López et al. 2019, Tulsidas et al. 2019). The amount of U contained in phosphate rocks imported into the EU from different countries has been estimated and it can be concluded that a maximum of 334 t U can be recovered theoretically from those of resources if any beneficiation process is carried out since 2017. According to the study of Lopez et al. (López et al. 2019), domestic phosphate reserves in

Argentina have big potential due to their high U content (39 – 135 mg/kg) and their recovery may be economically feasible. Ulrich et al. (2014) suggested phosphate rock as an unconventional U source to provide a long-term U supply safely. These make it an important alternative and lead to an increase in the number of studies focusing on the evaluation of phosphate ores as an unconventional uranium source. For example; the extraction of U from a low-grade uranium ore prepared in different particle sizes was investigated by using bacteria-assisted leaching for 150 days. A decrease in the particle size led to an increase in the extraction recovery of U. This bioleaching process was explained by a diffusion-controlled kinetic model (Wang et al. 2019). Furthermore, Singh et al. (2016) reviewed numerous studies focusing on the recovery of U from phosphoric acid by using the solvent extraction process including different stages. A number of studies have been carried out on the recovery of REEs from phosphate rock and phosphogypsum (Shlewit 2010;Z.H. İsmail et al. 2015; Hammas-Nasri, et al. 2016; El-Mottaleb et al. 2016; Liang, et al. 2017; Lambert et al. 2018; Amine et al. 2019). Different leaching agents such as H_2SO_4 (Z.H. İsmail, et al. 2015; Hammas-Nasri et al. 2016; Liang et al. 2017; Battsengel et al. 2018), HCl (Shlewit 2010; İsmail et al. 2015; El-Mottaleb et al. 2016; Stone et al. 2016; Lambert; et al. 2018; Amine et al. 2019; Nie et al. 2019), HNO_3 (Bandara et al. 2016), H_3PO_4 (Soltani et al. 2019; Wu et al. 2019), NaCl treatment followed by Na_2CO_3(Hammas-Nasri et al. 2019), and $HClO_4$ (Stone et al. 2016) were used for the recovery of U and REEs. When HNO_3 was used as a leaching agent for the extraction of REEs, better results were obtained compared with the use of H_2SO_4 or HCl as a leaching agent in the leaching test (İsmail et al. 2015). Similar results were observed in those of a previous study (Walawalkar et al. 2016).

Some pretreatment methods including mechanical grinding, ultrasonic impact, and resin-in-pulp were carried out to increase the extraction rate of REEs from the aforementioned sources and its rate increased to higher than 70% from 15% (Rychkov et al. 2018). Moreover, microwave pretreatment had a strong effect on the extraction rate and resulted in an increase in the leaching efficiency of REEs from phosphogypsum in HCl media by as much as 20% (Lambert et al. 2018). In another work, the REE content in the phosphate rock increased to 866.31 ppm from 161.82 ppm with a recovery of 55% via a gravitational separation method before the leaching test (Liang et al. 2018) and this makes the extraction of REEs from phosphate ores more profitable.

The purpose of this study was to investigate the extraction behavior of U together with REEs from a low-grade phosphate rock in the presence of different acids, individually. Before the leaching test, the U and REEs contents in the ore sample were upgraded by using the Falcon concentrator. The effects of acid concentration and temperature on the leaching test were also studied.

EXPERIMENTAL PROCEDURE

Materials

A 100 kg of the raw ore was collected from an east region of Turkey. The major content of the sample was determined by XRF (X-ray Fluorescence, MiniPal 4 Panalytical) and the detailed chemical analysis of the ore was carried out using ICP-MS (Inductively Coupled Plasma-Mass Spectrometer, Perkin Elmer NEXION 2000 P) after conducting HCl + HF + HNO_3 digestion followed by boric acid neutralization methods in a microwave digester (CEM, Mars 6) (Table 1). The X-ray diffraction (XRD) pattern of the sample obtained using a Rigaku Miniflex II diffractometer shows that the raw ore was mainly composed of calcite, quartz, and hydroxyapatite minerals. The loss on ignition (LOI) value of the raw ore determined using thermogravimetric analysis (TGA, Mettler Toledo TGA 3+]) was found to be 20.23%, which is in good agreement with the high temperature-sensitive mineral contents found in the XRD analysis. All chemicals (HCl, H_3PO_4, CH_3COOH, $HClO_4$, and HNO_3) used in this study were of analytical grade and used without purification.

Table 1. The chemical composition of the sample used in this study
Çizelge. 1 Bu çalışmada kullanılan örneğin kimyasal içeriği

Chemical Composition (%)							
CaO	SO_3	SiO_2	CuO	MgO	P_2O_5	Fe_2O_3	LOI
58.24	1.32	11.80	0.03	0.05	8.01	0.62	20.23
Uranium and Rare Earth Elements (REEs, ppm)							
Gd	Ce	La	Y	U	Dy	Er	Eu
9.65	35.01	51.20	120	392	4.01	4.1	0.62
Lu	Nd	Pr	Gd	Ho	Sc	Sm	Tb
0.15	11.33	2.68	9.65	0.25	12	2.15	0.54
Tm	Yb	Pm	Total REEs			Total (U+REEs)	
0.43	1.19	U.D	255.31			647.31	

Method

Pre-concentration of a phosphate ore

The ore samples were crushed using a jaw crusher and ground in a ball mill to obtain samples with a particle size (d_{100}) of 60 µm prior to enhanced-gravity separation tests. The representative sample from the ground ore was prepared by coning and quartering for each test and stored for use. The particle size distribution of the sample determined by Malvern Mastersizer 300U is seen in Figure 1. It is clear that the raw sample had a uniform particle size with Gaussian distribution ranging from 10 to 60 µm.

The pre-concentration of the raw sample was carried out using the Falcon L40 laboratory model concentrator (Sepro Mineral Systems, Canada), which is widely used for many purposes such as beneficiation of REEs (Filippov et al. 2016), nickel (Farrokhpay et al. 2019), copper (Kademli and Aydogan 2019), and tungsten ores (Foucaud et al. 2019). The working principle of the concentrator explained in detail by Farrokhpay et al (2019). The experimental conditions conducted in this study are listed in Table 2.

Table 2. Experimental conditions for the Falcon concentrator
Çizelge 2. Falcon konsantratörü için deney şartları

Experiment No	Rotary speed		Pressure (psi)
	Hz	G	
E1	35	60	2
E2	35	60	3
E3	40	78	2
E4	40	78	3
E5	50	123	2
E6	50	123	3

The slurry prepared with the mixing of 100 g ore and 2000 mL water was pumped into the Falcon concentrator via the pump at a flow rate of 0.5 L/min. After each test, light materials discarded from the bowl were identified as tailings, whereas heavy materials present in the equipment were pre-concentrate. Each product was dried, weighed, and dissolved in the Mars 6 microwave digester to determine the amount of U and REEs in the pre-concentrate by ICP-MS. The pre-concentration recoveries of these elements were calculated as follows:

$$\mathrm{PR} = \frac{C \times c}{F \times f} \times 100 \qquad (1)$$

Where PR is the pre-concentration recovery (%), C is the weight of the pre-concentrate (%), c is the U or REEs contents in the pre-concentrate (ppm), F is the weight of the feed material (%), f is the U or REEs contents in the feed material (ppm).

Approximately 2000 g of the pre-concentrate sample was prepared at the optimal experimental conditions for use in a subsequent test.

Leaching of a pre-concentrate ore with different acids

The pre-concentrate ore obtained by the Falcon concentrator was leached using different acids at the fixed experimental conditions that were as follows: 300 rpm stirring speed, 2 M acid concentration, 40 °C temperature, 1:6 solid-to-liquid ratio, and 2 h reaction time. The leaching tests were performed in a 250 mL glass reactor equipped with not only temperature-controlled circulating water to stabilize the solution temperature but also a glass condenser to prevent evaporation. The slurry was stirred at 250 rpm by a magnetic stirrer. A required amount of sample was added into the reactor to provide the desired solid-to-liquid ratio of 1:6 when the solution temperature increased to 40 °C. The volume of the solution was kept as 150 mL.

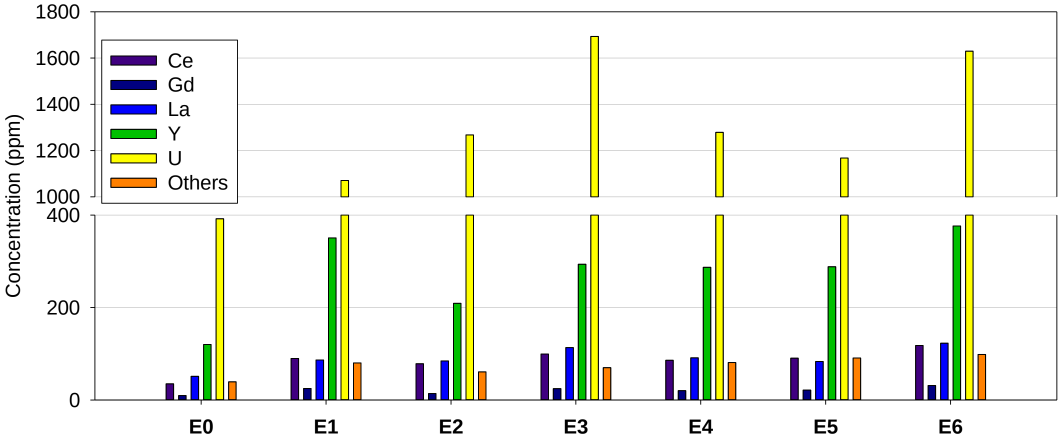

Figure 2. The distribution of U together with Ce, Gd, La, and Y in the pre-concentrated ore

Şekil 2. Ön konsantre içinde U ile birlikte Ce, Gd, La ve Y dağılımları

Different acids (HCl, H_3PO_4, HNO_3, CH_3COOH, and $HClO_4$) were used to determine their effect on the extraction of U and REEs under the fixed leaching conditions. When the predetermined time was over, the reaction was completed. The leach residues were filtered through a Whatman 1 filter paper and washed four times with hot pure (distilled) water. The solution obtained after each leaching test was diluted 100 times to analyze in ICP-MS. A blank solution was prepared to prevent possible experimental errors due to the possibility of U present in H_3PO_4 (Singh et al. 2016). The U and REEs extractions based on the analysis of leaching solutions were calculated by using the following equation.

$$\mathrm{ER}\ (\%) = \frac{A}{B} \times 100 \qquad (2)$$

Where ER is the extraction rate of U or REEs (%), A is the concentration of U or REEs in the leachate solution (ppm), B is the initial concentration of U or REEs in the pre-concentrate (ppm).

Considering all experimental results, the best leaching agent was selected and used in the following tests.

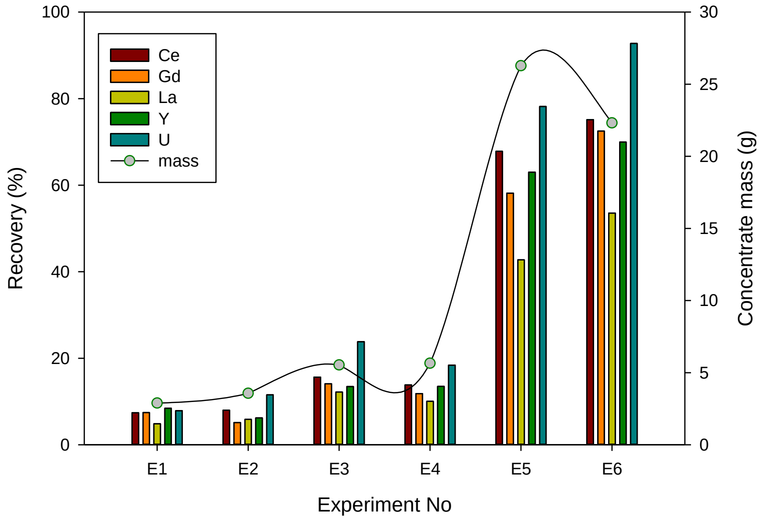

Figure 3. The recovery of U and REEs with mass changes in the pre-concentrated ore

Şekil 3. Ağırlık değişimi ile birlikte ön konsantredeki U ve REEs kazanımı

Leaching of a pre-concentrate ore with H_3PO_4

Among all leaching agents, H_3PO_4 was found to be the best leaching agent for this study. A series of leaching experiments were performed in the presence of different H_3PO_4 acid concentrations in a range of 1 – 4 M and the other parameters were kept constant as aforementioned. To demonstrate the effect of reaction temperature on the extraction behavior of U and REEs, three leaching tests were performed at a temperature of 20, 40, and 50 ºC under the best experimental conditions, which were determined before. After each test, the residue was filtered, dried, and dissolved using the same procedures and the extraction rates of U and REEs were calculated.

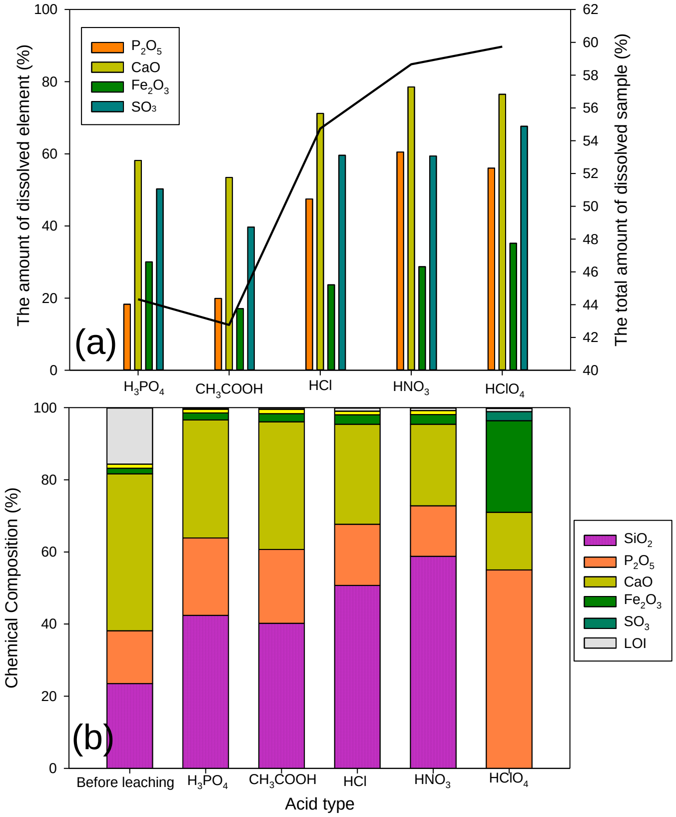

Figure 4. (a) The amount of dissolved major compounds from the preconcentrated ore and their weight losses (%) (b) The major chemical composition of the residues obtained after the leaching test (other parameters: 300 rpm stirring speed, 2 M acid concentration, 40 °C temperature, 1:6 solid-to-liquid ratio, and 2 h reaction time)

Şekil 4. (a) Ön konsantreden çözünen major element miktarı ve ağırlık kaybı değerleri (b) liç işlemi sonrasında geriye kalan kalıntının major element içeriği (diğer parametreler: 300 dev/dk karıştırma hızı, 2 M asit konsantrasyonu, 40 °C sıcaklık, 1:6 katı-sıvı oranı ve 2 saat reaksiyon süresi)

RESULTS AND DISCUSSION

Pre-concentration test results using the Falcon concentrator

The effect of Falcon concentrator's working condition on the separation efficiency of U and REEs from a low-grade phosphate ore was investigated. Figure 2 shows U and REEs distributions of heavy materials obtained after applying the Falcon test.

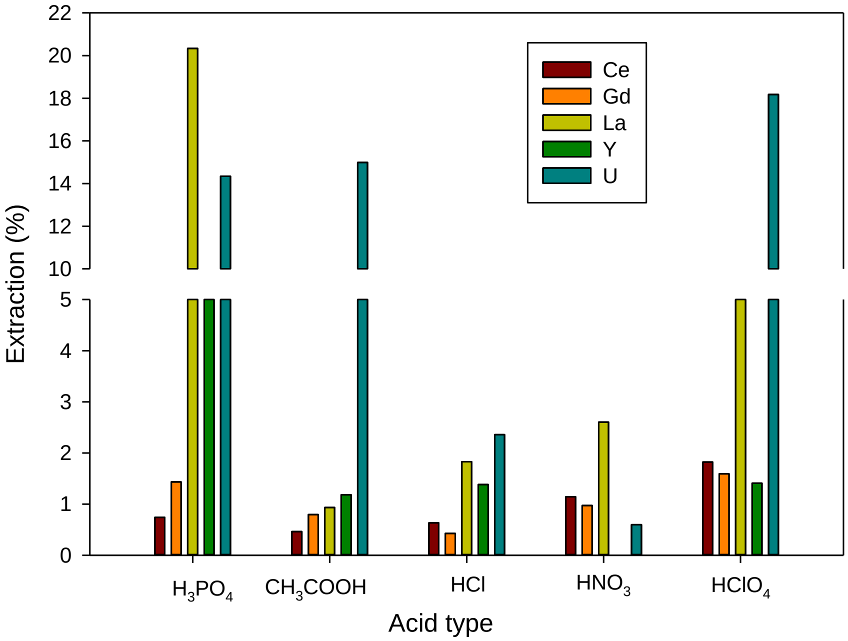

Figure 5. The dissolution recovery of U together with Ce, Gd, La, and Y from the preconcentrate using different acids (other parameters: 300 rpm stirring speed, 2 M acid concentration, 40 °C temperature, 1:6 solid-to-liquid ratio, and 2 h reaction time)

Şekil 5. Ön konsantreden farklı asitler kullanılarak U ile birlikte Ce, Gd, La ve Y elementlerinin çözünme verimleri (diğer parametreler: 300 dev/dk karıştırma hızı, 2 M asit konsantrasyonu, 40 °C sıcaklık, 1:6 katı-sıvı oranı ve 2 saat reaksiyon süresi)

The obtained results suggest that REEs grade in the pre-concentrate ore increase linearly with increasing rotary speed and water pressure. The U grade in the pre-concentrate increased to a value higher than 1000 ppm in each Falcon test but its recovery (<20%) by conducting experiment E1 was much lower compared to the obtained recoveries of U, which belong to experiments E5 (78.18%) and E6 (92.76%) (Figure 3). To obtain U in the preconcentrate with a recovery of higher than 50%, the rotary speed should be 50 Hz, which corresponds to 123 G. The concentrate mass increased with increasing rotary speed that creates centrifugal and hydrodynamic forces, which play a key role in separating heavy materials from light ones in the raw ore.

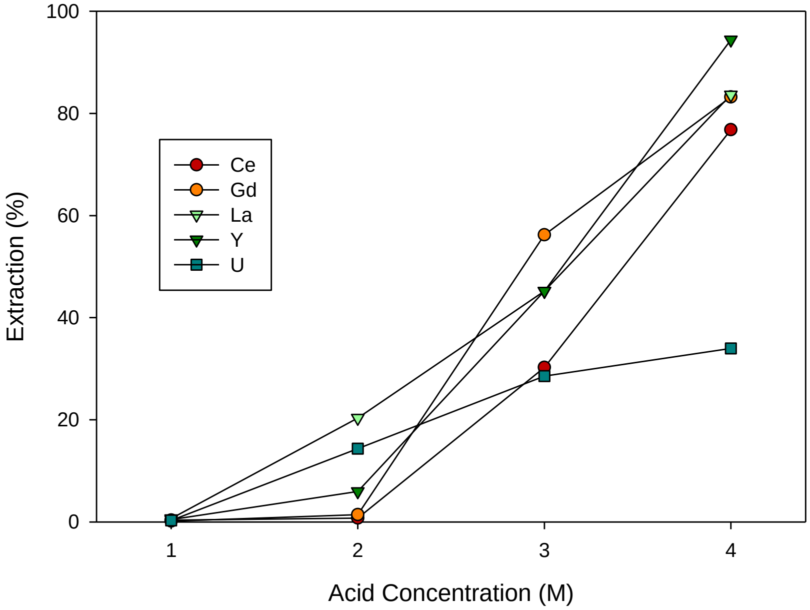

Figure 6. Effects of H_3PO_4 concentrations on the extraction recovery of U together with Ce, Gd, La, and Y from the preconcentrate (300 rpm stirring speed, 40 °C temperature, 1:6 solid-to-liquid ratio, and 2 h reaction time)

Şekil 6. Ön konsantreden U ile birlikte Ce, Gd, La ve Y'nin çözünmesine H3PO4 asit konsantrasyonun etkisi (diğer parametreler: 300 dev/dk karıştırma hızı, 2 M asit konsantrasyonu, 40 °C sıcaklık, 1:6 katı-sıvı oranı ve 2 saat reaksiyon süresi)

The maximum concentrate mass was about 25 g when 100 g of the sample was fed into the concentrator under the condition of experiment E5. However, increasing water pressure from 2 psi to 3 psi at the same rotary speed led to a decrease in the concentrate mass, which was about 20 g (experiment E6). This could be explained by escaping light materials from the bowl due to the buoyancy of water. This is in line with those of a previous study (Aydogan and Kademli 2019). As such, the U content in the pre-concentrate could be upgraded up to 1693.64 ppm with a recovery of 92.76% at the following conditions: water pressure of 3 psi and rotary speed of 50 Hz, which were determined as optimal parameters for this study. Additionally, the REEs showed similar behaviors in the beneficiation test, but their recoveries were lower compared to those of U recoveries. At the condition of experiment E6, the highest recoveries were found to be 75.14% for Ce, %72.53 for Gd, %53.56 for La, and %69.99 for Y.

When the raw ore was fed into the concentrator at the condition of experiment E6, the total amount of REEs in the preconcentrated ore increased from 255.30 ppm to 747.20 ppm, corresponding to 117.91 ppm Ce, 31.31 ppm Gd, 122.91 ppm La, 376.42 ppm Y and 90.92

ppm others. These values make the pre-concentrated ore being evaluated as REEs source. Researchers indicate that there are different REEs groups (critical [Nd, Eu, Tb, Dy, Y, and Er], uncritical [La, Pr, Sm, and Gd], and excessive [Ce, Ho, Tm, Yb, and Lu]) used to evaluate REEs quality.

Table 3. Evaluation of the pre-concentrate obtained in this study
Çizelge 3. Bu çalışmada elde edilen ön konsantrenin değerlendirilmesi

Experiment No	Critical (Nd, Eu, Tb, Dy, Y and Er)	Uncritical (La, Pr, Sm, and Gd)	Excessive (Ce, Ho, Tm, Yb, and Lu)	C/E (Critical/Excessive)	CEP (C / C+U+E)
E0	140.60	60.76	36.60	3.84	0.59
E1	391.32	123.71	93.02	4.21	0.64
E2	236.80	106.20	81.45	2.91	0.56
E3	324.07	151.76	103.33	3.14	0.56
E4	325.68	122.72	89.14	3.65	0.61
E5	330.85	116.29	93.46	3.54	0.61
E6	432.59	170.31	122.06	3.54	0.60

The ratio of critical elements and outlook coefficients (critical/excessive ratio, C/E) are important parameters and should be greater than 0.3 and 0.7, respectively (Seredin and Dai 2012). These values are important parameters for determining the potential of material as REEs source. Based on this approach, we evaluated the REEs distribution in the pre-concentrate ore and obtained results were summarized in Table 3. The C/E ratios calculated in this study were in a range of 2.91 – 4.21, indicating that the pre-concentrate ore has a potential to be used as REEs source.

Leaching Experiments

Effect of acid types on the extraction of U and REEs

The preconcentrated ore was leached in different acid mediums at a temperature of 40 °C for 2 h to evaluate the effect of acid types on the extraction of U and REEs. The other parameters were kept constant as follows: 300 rpm stirring speed and 1:6 solid-to-liquid ratio. The major oxides of the leachate residue determined by XRF were listed in Figure 4.

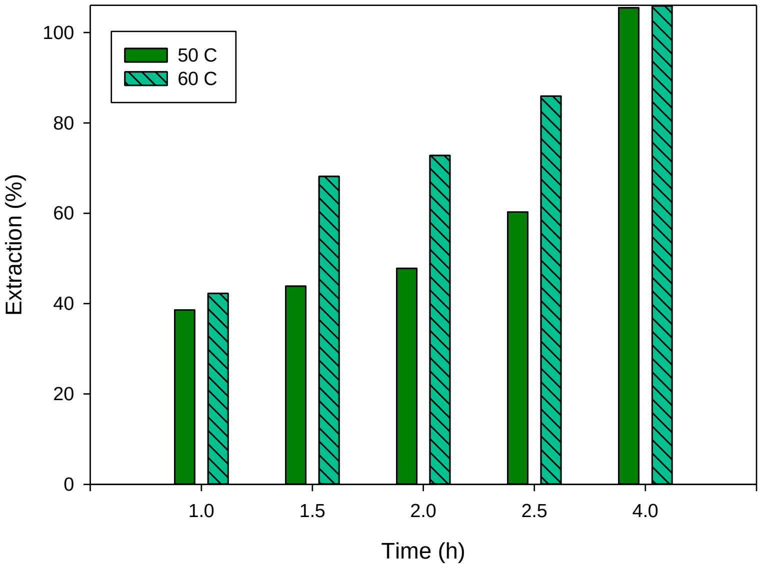

Figure 7. Dissolution of U from the preconcentrate in the presence of H_3PO_4 at different temperatures

Şekil 7. H3PO4 varlığında farklı sıcaklılıklarda ön konsantreden U çözünmesi

The weight loss after leaching tests indicated that more than 50% of the ore was dissolved, except for the ore leached with H_3PO_4 and CH_3COOH acid. The carbonate content in the preconcentrated ore released during the leaching tests, as shown in Figure 4b. The effect of acid types on the amount of dissolved P_2O_5 was in descending order as $HNO_3 > HClO_4 > HCl > CH_3COOH > H_3PO_4$.

To evaluate the extraction behavior of REEs, the following elements Ce, Gd, La, and Y were selected due to their high content in the preconcentrated ore. Figure 5 shows the extraction recoveries of U together with Ce, Gd, La, and Y in the presence of different acids. It is clear that the recovery of these elements was lower than 25%. Except for La that dissolved with the highest recovery (20.5%), the extraction recoveries of Ce, Gd, and Y was below 5%. The recovery value for U was in a range of 0 – 18%. The maximum U extraction was achieved by using $HClO_4$ acid as the leaching medium. However, the experimental findings for the U were lower compared to a previous study (Cánovas et al. 2019), in which U with a recovery of 21% can be obtained from phosphogypsum in the presence of HNO_3 (3M). In addition, only %10 U was recovered by using H_2SO_4 (0.5 M) in the same study. These indicate the importance of acid types used in the leaching test. In this study, the effect of acid types on the extraction of U was ordered as follows (from highest to lowest): $HClO_4$ > CH_3COOH > H_3PO_4 > HCl > HNO_3.

Considering all experimental findings given in Figure 4 and Figure 5, H_3PO_4 was selected as the best leaching agent at the fixed experimental conditions.

Effects of H_3PO_4 concentration and temperature on the extraction of U and REEs

The leaching experiments were performed in a range of 1 – 4 M H_3PO_4 concentrations at the following fixed conditions: 300 rpm stirring speed, 40 °C temperature, 1:6 solid-to-liquid ratio, and 2 hours reaction time. Figure 6 shows the effect of acid concentration on the dissolution yields of U and REEs. It is obvious that the recoveries increased linearly with the increase of acid concentration. More than 60% of REEs dissolved, when 4 M H_3PO_4 was used but U was barely extracted from the preconcentrated ore (<25%). It was understood from experimental findings that elements were ordered as follows Y > Gd ≥ La > Ce > U based on their dissolution yield.

Additional leaching experiments were performed at a temperature of 50 °C and 60 °C for different durations. The extraction recovery of U increased in extended times (Fig 7). The temperature increased from 50˚C to 60˚C enhanced both the rate and degree of U recovery. The required time to dissolve all of U ions decreased within the increase of reaction temperature.

CONCLUSION

The aim of this study was to evaluate the potential of a low-grade phosphate ore in terms of its U and REEs content and to recover the U and REEs from the ore by conducting the Falcon concentrator followed by acid leaching method. The effect of Falcon concentrator's working conditions was investigated. It was determined that the U content in the preconcentrated ore increased to 1629.66 ppm from 392 ppm with a recovery of 92% and the amount of REEs was found to be 747.20 ppm. A series of leaching experiments were carried out at the fixed leaching conditions to determine the effect of acid types on the extraction recovery of U and REEs from the preconcentrated ore. Experimental findings indicated that acids were ordered as follows (from highest to lowest) : $HClO_4$ > CH_3COOH > H_3PO_4 >HCl, and HNO_3, according to the extraction recovery of U, while the order of acids considering the extraction REEs+U was $H_3PO_4 > HClO_4 > CH_3COOH > HCl > HNO_3$. Among all acids, H_3PO_4 was selected as the best leaching agent. The effect of acid concentration and temperature in the presence of H_3PO_4 was investigated and REEs leached with high recoveries, but the extraction recovery of U was quite lower than that of those REEs. Thus, the temperature had to be increased to at least 50 °C for 4 hours in order to extract 100% of U. The residue obtained after leaching can be further used in fertilizer applications due to its high P content.

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