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Beneficiation of bastnaesite by a multi-gravity separator

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Abstract

The preconcentration of bastnaesite of Beylikahir ore was achieved by attrition scrubbing of the original ore crushed to −1.65 mm. After scrubbing the sample at 50% solid pulp density for 1 h, the pulp was diluted and deslimed by cycloning. The slime (cyclone overflow) that was collected as a preconcentrate, assayed 28% REO with 72.6% recovery. The preconcentrate was fed to a Mozley multi-gravity separator (MGS) for further upgrading. As a result, a bastnaesite concentrate with 35.5% REO grade and 48% recovery (on original ore basis) was produced. Chemical analysis of the concentrate was as follows: Ce, 13.75%; La, 11.81%; Nd, 2.30%; Pr, l%; Sm, 0.15%; Y, 0.064%. H_2SO_4 curing and water leaching of the preconcentrate indicated that >75% of REE could be taken into solution.

Keywords: Bastnaesite; Attrition scrubbing; Cycloning; Multi-gravity separator; Yates technique

1. Introduction

Monazite (Ce,La,Th,Y)PO_4 (a rare earth phosphate) and bastnaesite (Ce,La)FCO_3 (a rare earth fluorocarbonate) are two principal commercial rare earth ore minerals. Monazite is present in beach sands around the coastal belt of India, Brazil, Australia, South Africa and the USA. Baotou, Mianning and Weishan deposits in China, and Mountain Pass, California, in the United States are the important bastnaesite deposits.

Previously, monazite was the principal source of rare earths. However, for the last 33 years, the production of bastnaesite has been the world's major source of rare earths [1]. Bastnaesite is a mineral consisting predominantly of the lighter rare earths, distributed as follows: cerium 50%, lanthanum 34%, neodymium 11%, praseodymium 4%, samarium 0.5%, gadolinium 0.2% and europium 0.7%. The rare earth oxides (RE_2O_3) constitute 75% of the mineral bastnaesite.

Flotation is the standard method for recovering rare earth minerals from igneous and hydrothermal deposits, while physical methods, such as gravity and magnetic/electrostatic separation, are currently employed for the treatment of rare earth containing placer deposits.

Turkey has a complex ore deposit in Eskişehir-Beylikahir (mid-west Turkey), which contains about 1 million tons of REO reserves with an average concentration of 3.42% REO. The complex ore is of hydrothermal origin and consists of rare earth minerals and fluorite and barite with thorium. Bastnaesite is the main rare earth mineral in the ore. It can easily be distinguished only by scanning electron microscopy (SEM), ion microprobe (IP), inductively coupled plasma (ICP), or other quantitative analytical methods (XRF, EDX, etc.).

The purpose of this study was to beneficiate rare earth minerals using a Mozley multi-gravity separator (MGS).

2. Materials and methods

2.1. Characteristics of the ore

A complete chemical analysis of a representative sample is given in Table 1. Analysis of the ore by SEM, XRD and optical microscopy showed that bastnaesite present in the Beylikahir deposit is either as a cementing material between the fluorspar and barite particles or intimately associated with these minerals. The calculated REE and REO contents of the ore are 6.5 and 7.9%, respectively. Bastnaesite comprises 10.2% of the original ore. The distribution of REE, fluorite and barite in the ore is shown in Table 2. As can be seen, REE tend to accumulate in the fine fractions.

2.2. Methods

Under the light of mineralogical and sieve analyses, selective enrichment of the REE was determined to be in

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Table 1
Chemical analysis of the ore [2]

Element or compound	Ce	La	Nd	Pr	Sm	Gd	Dy	Y
Grade	3.00%	2.70%	0.55%	0.18%	220 ppm	120 ppm	60 ppm	300 ppm
Element or compound	CaF_2	$BaSO_4$	Al_2O_3	Fe_2O_3	SiO_2	ThO_2	SrO	P_2O_5
Grade	55.00%	25.40%	4.00%	3.00%	1.30%	0.07%	0.60%	1.00%

the fines. Therefore, concentration by size classification seemed to be a viable beneficiation technique. Attrition scrubbing followed by cycloning and MGS processing was employed to concentrate the ore and raise the REO grade to around 60%, which is preferable for downstream processing.

3. Experimental results and discussion

3.1. Preconcentration of REE by attrition scrubbing and cycloning

The sub-sieve analysis technique applied on the −38 μm fraction demonstrated that most of the bastnaesite particles are in the −5 μm size range. As cryptocrystalline bastnaesite particles are present in the cementing material between fluorite and barite particles, attrition scrubbing was applied to liberate them. For this purpose, the ore crushed to −1.65 mm (10 mesh) was subjected to attrition scrubbing for 1 h at a solid/liquid ratio of 1:1 by weight. Then, the pulp was deslimed by three hydrocyclones operating in series. The overflow (slime) was collected as preconcentrate with 28% REO grade and 72.6% recovery.

3.2. Concentration by the multi-gravity separator

The MGS represents the latest development in fine grain mineral separation. As it can recover ultrafine grained minerals, it was selected to increase the REE grade of the cyclone overflow. The MGS consists of a slightly tapered open ended drum that rotates in a clockwise direction and is shaken sinusoidally in an axial direction [3].

Inside the drum is a scraper assembly which rotates in the same direction as the drum but at a slightly faster speed. Feed slurry is introduced continuously midway onto the internal surface of the drum via an accelerator ring cleaner. Wash water is added via a similar launder positioned near the open end of the drum. As a result of the high centrifugal forces and the added shearing effect of the sinusoidal shaking, the dense particles migrate through the slurry film to form a semisolid layer against the wall of the drum. This dense layer is conveyed by the scrapers towards the open end of the drum where it discharges into the concentrate cleaner. The less dense minerals are carried by the flow of wash water downstream to the rear of the drum to discharge via slots into the tailings cleaner. Wash water flow rate, vibrational amplitude, vibrational frequency, tilt angle and drum rotational speed are critical variables which affect concentrate grade and recovery.

The success of concentration with MGS depends on the selection of suitable parameters and minerals. The optimization of these parameters necessitates many tests. The total number of experiments required can be reduced by employing a factorially designed series using the Yates technique [4]. This test series provides an indication of optimum parameters. The first step in this technique is the selection of reasonable parameters for preliminary tests. In the preliminary tests the rotational drum speed and tilt angle were kept constant at 240 rpm and 8° from the horizontal axis, respectively. Shake frequency, shake amplitude and wash water flow rate were chosen as the major variables. The Yates technique for 2^3 experiments (three being the number of parameters) was used for statistical design and analysis of the results. Table 3 shows the experimental conditions and the grade and recovery responses for each experiment. The experimental conditions were arranged in the so-called Yates order.

Table 2
Screen analyses of barite, fluorite and major REE

Size fraction (μm)	Weight (%)	Barite (%)		Fluorite (%)		Cerium (%)		Lanthanum (%)	
		$BaSO_4$	Dist.	CaF_2	Dist.	Ce	Dist.	La	Dist.
−1700+600	28.76	17.78	18.77	68.20	37.37	1.70	14.89	1.40	14.14
−600+200	22.36	23.59	19.36	61.60	26.07	1.360	10.94	1.50	12.01
−200+75	19.63	41.72	30.09	38.10	14.26	2.90	17.33	2.80	19.43
−75+38	10.07	50.09	18.52	32.00	6.14	3.60	19.45	3.50	12.37
−38	19.18	18.83	13.26	44.20	16.16	6.40	37.39	6.20	42.05
Total	100.00	27.22	100.00	52.47	100.00	3.29	100.00	2.83	100.00

Table 3
Experimental conditions and responses

Test No.	Code	Shake frequency (cycles/min)	Wash water flow (dm^3/min)	Shake amplitude (mm)	Grade Ce (%)	Recovery (%)
1	(1)	4	4	10	12.11	83.01
2	a	5.6	4	10	12.88	71.02
3	b	4	8	10	12.18	79.69
4	ab	5.6	8	10	12.50	77.59
5	c	4	4	20	13.11	72.69
6	ac	5.6	4	20	12.54	77.27
7	bc	4	8	20	12.52	76.87
8	abc	5.6	8	20	12.54	76.87

Tables 4 and 5 combine the Yates technique with ANOVA (analysis of variance) to simplify the decision on the significance of the parameters investigated. The procedure for preparing the table is as follows.

(i) In column (3), the upper half is obtained by adding successive pair responses, and the lower half is obtained by subtracting successive pairs. Columns (4) and (5) are calculated in the same way.
(ii) Tests are repeated three times at center points to estimate the error associated with the determination of an individual response, which is required to test statistical significance.
(iii) The ANOVA procedure is applied.
(iv) The table value of $F(1,2)$ for $\alpha = 0.05$ is compared with the calculated F value.

Tables 4 and 5 indicate that the selected parameters have no significant role in the grade and recovery responses of the concentration process. As seen in Table 3, the concentrates' grades and recoveries were almost the same within the given operating conditions.

In order to increase the grade of the product, the rougher concentrate was cleaned in the MGS at center points of operational parameters in Table 4 and produced a concentrate with an average grade of 29.30% REE (35.50% REO)

Table 4
Yates technique combined with ANOVA for recovery response[a]

Yates order (1)	Recovery Ce (%) (2)	Column (3)	Column (4)	Column effects total (5)	$(5)_3^2/2$ (6)	Degrees of freedom (7)	F calculated $(6)/(7)S_e^2$ (8)	F table (1,2,0.05)	Decision
1	83.01	154.03	311.31	615.01	–	–	–	–	NS[b]
a	71.02	157.28	303.7	−9.51	11.31	1	3.47	18.51	NS
b	79.69	149.96	−14.09	7.03	6.18	1	1.89	18.51	NS
ab	77.59	153.74	4.58	5.31	3.52	1	1.07	18.51	NS
c	72.69	−11.99	3.25	−7.61	7.23	1	2.22	18.51	NS
ac	77.27	−2.10	3.78	18.67	43.57	1	13.26	18.51	NS
bc	76.87	4.58	9.89	0.53	0.05	1	0.02	18.51	NS
abc	76.87	0.00	−4.58	−14.47	26.17	1	8.03	18.51	NS
Column total		605.5	617.84	614.96					
Alternative total		308.92	307.48	0.0					

[a] Frequency (a), 4.8 cycles/min; wash water flow (b), 6 l/min; amplitude (c),15 mm; center point responses, (1) 78.58%, (2) 75.52%, (3) 75.31%; average recovery, 76.47%

$$S_e^2 = \sum_{i=1}^{3} \frac{(R_i - R_{\text{average}})^2}{2}$$

$S_e^2 = 3.26$.

[b] NS, not significant.

Table 5
Yates technique combined with ANOVA for grade response[a]

Yates order (1)	Recovery Ce (%) (2)	Column (3)	Column (4)	Column effects total (5)	$(5)_3^2/2$ (6)	Degrees of freedom (7)	F calculated $(6)/(7)S_e^2$ (8)	F table (1,2,0.05)	Decision
1	12.11	24.99	49.67	100.38	–	–	–	–	NS[b]
a	12.88	24.68	50.71	0.54	11.31	1	1.89	18.51	NS
b	12.18	25.65	1.09	−0.90	6.18	1	5.31	18.51	NS
ab	12.50	25.06	−0.55	0.14	3.52	1	0.10	18.51	NS
c	13.11	0.77	−0.31	1.04	7.23	1	7.10	18.51	NS
ac	12.54	0.32	−0.59	−1.64	43.57	1	17.68	18.51	NS
bc	12.52	−0.57	−0.45	−0.28	0.05	1	0.52	18.51	NS
abc	12.54	0.02	0.59	1.04	26.17	1	7.36	18.51	NS
Column total		100.92	100.16	100.32					
Alternative total		50.08	50.16	0.08					

[a] Frequency (a), 4.8 cycles/min; wash water flow (b), 6 l/min; amplitude (c), 15 mm; center point responses, (1) 12.20%, (2) 12.47%, (3) 12.38%; average recovery, 12.35%

$$S_e^2 = \sum_{i=1}^{3} \frac{(\text{grade}_i - \text{grade}_{\text{average}})^2}{2}$$

$S_e^2 = 0.019$.

[b] NS, not significant.

and recovery 47.78%. The contents of REE in the concentrate were as follows: Ce, 13.75%; La, 11.81%; Nd, 2.30%; Pr, 1.00%; Sm, 0.15%; and Y, 0.064%.

3.3. Hydrometallurgical recovery of REE from the preconcentrate

The leaching of preconcentrate directly into mineral acid solution did not give satisfactory leach recoveries, however acid curing the preconcentrate with concentrated acid (98% H_2SO_4) provided easier sulfation. The sulfated sample was then leached with water at room temperature for 3 h at a solid/liquid ratio of 1:4 in a constant-temperature water bath with a mechanical stirrer. The optimum amount of H_2SO_4 needed was twice the stochiometric amount (585 kg/ton of preconcentrate), while further acid additions resulted in decreasing recoveries.

Acid curing and water leaching processes resulted in recoveries of up to 80% cerium, 77% lanthanum, and 73% neodymium.

4. Conclusions

Turkey's Beylikahir complex ore deposit contains barite, fluorite and rare earth minerals. The principal rare earth mineral is bastnaesite. Bastnaesite is concentrated in the −5 μm fraction of the ore.

Preliminary beneficiation of bastnaesite is performed by attrition scrubbing and cycloning of the ore crushed to −1.65 mm. Three-stage cycloning produced an overflow preconcentrate with a 28% REO grade and 72.6% recovery. This product was upgraded by the use of a multi-gravity separator. The grade of the preconcentrate was increased by MGS to 35.5% REO grade with 48% recovery. The metallurgical recovery of REE by acid curing and water leaching of the preconcentrate resulted in the extraction of REE with a 73–80% recovery.

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