

Concentration and recycling of rare earth elements (REEs) from iron mine waste using a combination of physical separation methods

M. Moghise¹, M. Pourrahim¹, B. Rezai¹ and M. Gharabaghi^{2*}

1. Department of Mining and Metallurgical Engineering, Amirkabir University of Technology, Tehran, Iran

2. School of Mining Engineering, College of Engineering, University of Tehran, Tehran, Iran

Received 18 July 2015; received in revised form 2 November 2015; accepted 10 November 2015

*Corresponding author: gharabaghi@ut.ac.ir (M. Gharabaghi).

Abstract

This study aims to investigate and optimize the effects of the main parameters including the particle size, gravity and magnetic separation combination, high gradient magnetic separation, magnetic field intensity, shaking table slope, washing water flow, and electrostatic separation upon the rare earth element (REE) recoveries from iron mine waste. The electron microprobe showed that high amounts of REEs were distributed on the fluorapatite mineral, and hence, it was necessary to remove the high magnetic minerals by a low-intensity magnetic separation using a magnetic drum in an experimental procedure. A cyclic magnetic separator was used for the low-gradient magnetic separation. Moreover, a shaking table and an electrostatic separator were used to expand the recovery and grade of REEs. A combination of these methods was considered to optimize the REE recoveries based on the best combination including two steps of low magnetic separation, one step of medium intensity magnetic separation, a shaking table, and an electrostatic separator. Two low-intensity magnetic of 800 and 2000 gauss, one medium-intensity magnetic of 8000 gauss, a one-step shaking table with a water flow of 90 mL/s and a table slope of 3 degree, and one electrostatic separator of 25000 V with a blade angel of 20 degree had the best performance to separate REEs. The microscopic studies carried out showed that the monazite degree of freedom was between 75 and 105 micron. The results obtained showed that a particle size of $-75 + 63$ micron was a proper one to separate REEs. The total recovery and grade of the REE (Ce, La, Nd, Er, and Gd) concentrate obtained from the sample with a grade of 1499 ppm of REEs were 67.1 and 1.2%, respectively, at the optimum conditions. The results obtained showed that there was a direct relation between the phosphor grade and the REE recoveries, and that the REE recoveries increased by increasing the quantity of phosphor.

Keywords: Rare Earth Elements (REEs), Choqart Iron Waste, Fluorapatite, Magnetic Separation, Shaking Table.

1. Introduction

The term rare earth elements (REEs) refers to the fifteen metallic elements of the lanthanide series, coupled with the chemically similar yttrium, and occasionally scandium. These elements are typically split into two sub-groups, the cerium sub-group of "Light" rare earth elements (LREEs), which includes La to Eu, and the yttrium sub-group of "Heavy" rare earth elements (HREEs), which include the remaining lanthanide elements, Gd to Lu, as well as yttrium. Scandium, when classified as an REE, is not

included in either the LREE or HREE classifications [1, 2].

The problem with the exploitation of rare earth minerals is that they are not commonly found in economic concentrations. REEs are never found as pure metals but in a variety of minerals including silicates, oxides, carbonates, phosphates, and halides [3]. There are more than 250 different kinds of rare earth minerals that have been discovered so far, many containing very low concentrations of REEs varying from 10 to 300 ppm [1, 4]. There are only three major

REE-bearing minerals that are currently exploited commercially (bastnäsite, monazite, and xenotime).

Several methods have been used for the industrial processing of REEs [5]. These methods include a combination of flotation, gravity, magnetic, and electrostatic techniques along with the leaching methods. REE minerals are separated commercially from their associated minerals in placer deposits using a combination of gravity, magnetic, and electrostatic techniques. Flotation is a standard method used for recovering rare earth minerals from igneous and hydrothermal deposits, while physical methods such as the gravity and magnetic/electrostatic separations are currently employed for the treatment of REEs containing placer deposits [6].

Rare earth minerals are good candidates for gravity separation since they have relatively large specific gravities ($4\text{--}7\text{ kg/m}^3$), and are typically with a gangue material (primarily silicates) that is significantly less dense [7]. The most commonly utilized application of gravity separation is in monazite beneficiation from heavy mineral sands. Beach sand material is typically initially concentrated using a cone concentrator to produce a heavy mineral pre-concentrate (20–30% heavy minerals) before a more selective gravity separation step. Often a spiral concentrator is used to achieve concentrations of 80–90% heavy minerals [1].

REEs generally have a series of electrons occupying a shielded 4f sub-shell, and these electrons typically have magnetic moments that do not cancel out, resulting in a material with some degrees of magnetism [8]. Thus magnetic separation is a common method to eliminate highly magnetic gangue or to concentrate the desired paramagnetic REE-bearing minerals such as monazite and xenotime [1]. Magnetic separators are used in the beneficiation of Chinese bastnäsite rare earth ores to eliminate Fe-bearing gangue minerals prior to the rare earth-specific separation steps, and as a cleaning step for flotation feeds and concentrates [4]. In the case of China's second biggest rare earth deposit, the Sichuan Maoniuping Rare Earth Ore, magnetic separation has been combined successfully with gravity separation to achieve a bastnäsite recovery greater than 55% without the need for flotation [4].

Electrostatic separation is a beneficial technique that exploits the differences in conductivity between different minerals to achieve separation [9]. An electrostatic separation technique is

typically used only when alternative processing techniques will not suffice, as the comminution steps in mineral processing flow sheets are generally wet processes, and the energy requirements to drive off all the moisture prior to electrostatic separation can be significant [10]. In the context of rare earth mineral processing, the typical use of electrostatic separation is in the separation of monazite and xenotime from gangue minerals with similar specific gravity and magnetic properties [11]. In the rare earth mineral processing, the typical use of electrostatic separation is in the separation of monazite and xenotime from gangue minerals with similar specific gravity and magnetic properties [7, 11]. A specific example of this is when xenotime, which is more strongly paramagnetic than monazite, is concentrated with ilmenite after magnetic separation of heavy mineral sands. In this case, the only method by which xenotime may be removed from ilmenite is electrostatic separation because, unlike xenotime, ilmenite is conductive [12].

Froth flotation is commonly applied to the beneficiation of rare earth ores due to the fact that it is possible to process a wide range of fine particle sizes, and the process can be tailored to the unique mineralogy of a given deposit. From disseminated ores contained in mineral lenses, the recovery of bastnaesite and monazite is accomplished using flotation. The flotation properties of bastnaesite and monazite are similar to the gangue minerals contained in the bastnaesite and monazite such as calcite, barite, apatite, tourmaline, and pyrochlore, which represent difficulties in selective flotation. However, in the recent years, significant progress has been made in the flotation of both monazite and bastnaesite. An example is in Bayan Obo, where gravity separation has been employed between the rougher and cleaner flotation circuits to efficiently separate monazite and bastnäsite from the iron-bearing and silicate gangue material [13].

Valuable heavy minerals can be seen in Egyptian beach sands containing approximately 30 wt% [14]. In this flow sheet, low specific gravity gangue is separated by wet gravity concentration (the authors employed a Wifley shaking table for this purpose), and then low intensity magnetic separation is used to discard any ferromagnetic mineral without removing the paramagnetic monazite. The remaining non-magnetic stream contains most of the valuable monazite, zircon, and rutile as well as a portion of the gangue

minerals which were not removed in the first two steps. A series of gravity, magnetic, and electrostatic separations are then applied to exploit the different properties of the monazite, zircon, and rutile minerals, and produce the final concentrate streams. Rutile is removed by the conductor fraction after electrostatic separation (monazite and zircon are non-conductive), and then diamagnetic zircon may be removed from the paramagnetic monazite via a further magnetic separation [14].

In this study, a sample iron waste obtained from the Ghoghart mine was analyzed by X-ray diffraction, X-ray fluorescence, microscopic study, and electron microprobe to identify the rare earth component. In addition, the parameters affecting the recovery and grade of separation were investigated for optimization; therefrom, the best sequence of physical separation was selected. Flotation has usually been used in the other studies to separate monazite from iron [11] or gravity and magnetic methods have been employed to separate monazite from sand beach [7, 14, 15]. However, the gravity and magnetic methods were used in this study to separate monazite from iron waste. The spiral [1] or multi-gravity [12] has been used in some studies on the monazite separation, while the shaking table was used in this study.

2. Materials and Method

The sample was obtained from the Choghart thickener waste. Table 1 shows the chemical composition of the variant elements and REE distribution in the sample. X-ray diffraction confirmed the presence of quartz (SiO_2), hematite (Fe_2O_3), magnetite (Fe_3O_4), goethite (FeOH), dolomite (CaCO_3), anorthite ($\text{CaAl}_2\text{Si}_2\text{O}_8$), and microcline (KAlSi_3O_8), and in the other side, optical mineralogical analysis comprised monazite, bastnasite, xenotime, alunite, fluorapatite, hematite, magnetite, and calcite. Electron microprobe showed that high amounts of REEs were distributed on the fluorapatite mineral (Figure 1), and hence, it was necessary to remove the high magnetic minerals by low-intensity magnetic separation with magnetic drum (ERIZMAGNETIC) in the experimental study. A cyclic magnetic separator (BOXMAGRAPID) was used for the low-gradient magnetic separation. Moreover, a shaking table and an

electrostatic separator were used to expand the recovery and grade of REEs. The circuit of separation was arranged using two low-intensity magnetic (650 & 1500 gauss) and one shaking table.

Then a medium-intensity magnetic separator was added to the circuit to eliminate the iron oxide minerals with a low magnetic ability. The effect of magnetic field intensity on the REE separations in this circuit was investigated using different ranges including 500-1000-6000 gauss, 650-1500-6500 gauss, 800-2000-8000 gauss, and 1000-3000-12000 gauss for the low magnetic intensity, low magnetic intensity, and medium magnetic separators, respectively. After that, the values for the shaking table slope and water flow rate were changed to optimize the REE recoveries and assays. Subsequently, the electrostatic separation test was performed on the shaking table concentrate to expand the REE assays at an intensity of 25000 V and a blade angel of 20° . More detailed conditions were referred in the figure for each test. The concentrations of REEs in the solid phase were determined by XRF. Measured quantities of the dry sample and water were mixed in the slurry tank to obtain the required feed solids. A pump was used to feed the slurry for the separation devices at the same flow rate. After collection of the streams, it was filtered, dried, and weighed for analysis of the grade and recovery. It should be noted that the sample was driven off from all moisture in the electrostatic separation.

3. Result and discussion

The sample was processed using a combination of shaking table, LIMS (650 G), and LIMS (1500 G), as can be seen in Figure 2, to separate the different minerals based on their differences in specific gravity and magnetic properties.

The gravity preconcentration steps were designed to eliminate the low specific gravity gangue minerals, and the magnetic separation steps were designed at varying magnetic field strengths to either remove the unwanted ferromagnetic gangue minerals or concentrate the valuable paramagnetic REE minerals.

Table 1. Content of variant element and distribution REEs.

Size (μ)	Weight		Assay (ppm)						Distribution (%)					
	gr	%	Er	Gd	Y	Nd	La	Ce	Er	Gd	Y	Nd	La	Ce
(+400)	17.2	7.6	27	110	630	328	78	111	7	6.8	7	6.8	5.7	6.5
(-400 + 250)	13	5.7	27	124	512	330	98	200	5.3	5.8	4.3	5.2	5.4	8.9
(-250 + 177)	11.2	4.9	28	98	700	298	76	113	4.8	3.9	5.1	4	3.6	4.3
(-177 + 150)	11	4.8	30	113	513	350	67	115	5	4.4	3.6	4.7	3.1	4.3
(-150 + 105)	16	7	26	130	600	300	70	103	6.3	7.4	6.2	5.8	4.8	5.6
(-105 + 75)	23	10.1	31	121	590	412	115	134	10.8	9.9	8.8	11.5	11.3	10.5
(-75 + 63)	27	11.9	30	120	630	350	98	120	12.3	11.6	11	11.4	11.3	11
(-63 + 44)	29	12.8	30	120	780	360	90	123	13.2	12.4	14.6	12.6	11.1	12.2
(-44)	80	35.2	30	135	755	380	123	134	36.4	38.6	39.1	36.8	42	36.5
Feed	277	100	29	123	680	363	103	129	101	100	100	99	98.5	100

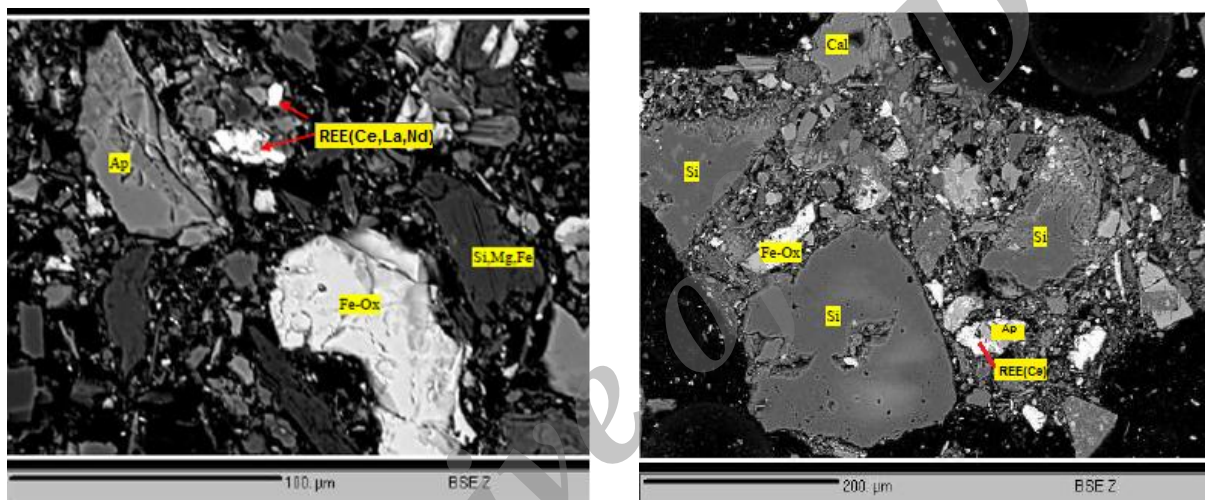


Figure 1. Back scattered electron (BSE) images by EPMA of iron waste.

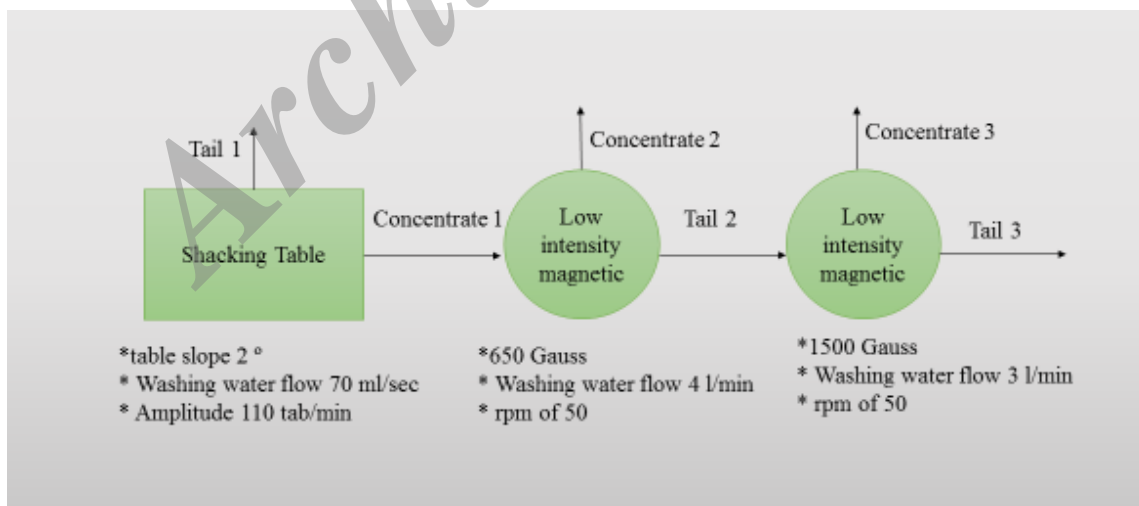


Figure 2. Diagram of first separation flow sheet (SHLL).

3.1 Effect of particle size

It is well-known that the particle size is a main parameter involved for mineral separation. Thus the effect of particle size on the REE separations was investigated at -150 + 105, -105 + 75, and -

75 + 63 micron (Table 2). Microscopic studies showed that the monazite degree of freedom was between 75 and 105 micron.

Table 2. Effect of particle size on REE separations.

Unit		%			ppm						
Size (micron)		Fe	P	SiO ₂	Er	Gd	Y	Nd	La	Ce	ΣREE
(-150 + 105)	Assay	16	5.8	10	89	450	2100	1020	250	350	4259
	Recovery (%)	19.5	39.3	5.3	ΣREE = 53.9%						
(-105 + 75)	Assay	17	6.1	13	112	450	2134	1456	423	487	5062
	Recovery (%)	21.6	45.3	7.2	ΣREE = 56.6%						
(-75 + 63)	Assay	17	6.8	12	93	382	2046	1141	317	364	4343
	Recovery (%)	24.8	63.4	8.1	ΣREE = 62.2%						

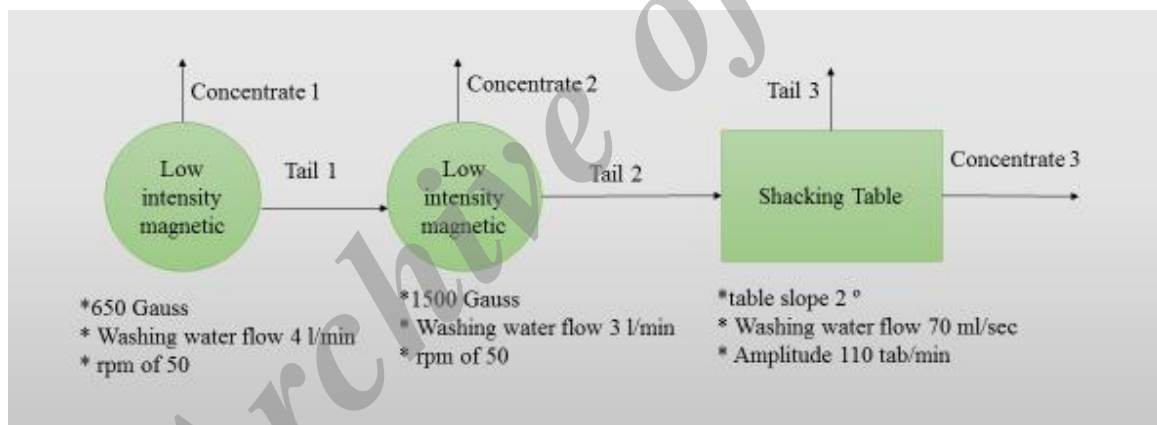
The results obtained showed that decreasing the particle size increased the separation recovery, and so -75 + 63 micron was selected as the optimum size. Since decreasing the particle size makes rare earth minerals release from gangue, the results obtained were predictable.

3.2 Effect of gravity and magnetic separation combination

The sample was concentrated by combination of LIMS (650 G), LIMS (1500 G), and shaking table, as shown in Figure 3, to introduce the best

arrangement between the three separations at this stage. Note that the particle size was in the optimum range (-75 + 63).

Table 3 shows the test results of the gravity and magnetic separation combination. Combination of (LLSh) had a more potential to separate REEs from gangue because the shaking table was very sensitive to slimes, and the amount of slimes was probably minimum in the shaking table feed after two steps of washing water at the magnetic separation process (Tail 2).

**Figure 3. Effect of gravity and magnetic separation combination on REE separation flow sheet (LLSh).****Table 3. Effect of gravity and magnetic separation combination.**

Unit		%			ppm						
Combination		Fe	P	SiO ₂	Er	Gd	Y	Nd	La	Ce	ΣREE
LLSH	Assay	11	5.3	15	99	401	2111	1211	321	346	4489
	Recovery (%)	17.6	54.2	11.1	ΣREE = 70.2%						
SHLL	Assay	17	6.8	12	93	382	2046	1141	317	364	4343
	Recovery (%)	24.8	63.4	8.1	ΣREE = 62.2%						

3.3 Effect of medium gradient magnetic separation

The amount of iron was high in the shaking table concentrate (Conc. 3). Thus a medium gradient magnetic separator was added in the separation

process to eliminate the iron oxide minerals with low magnetic ability (Table 4).

The iron recovery decreased about 3 percent after this step. Therefore, three magnetic separation steps were selected as the optimum flow sheet (Figure 4).

Table 4. Effect of high gradient magnetic separation on REE separation.

Unit		%			ppm						
Combination		Fe	P	SiO2	Er	Gd	Y	Nd	La	Ce	ΣREE
LLSH	Assay	11	5.3	15	99	401	2111	1211	321	346	4489
	Recovery	17.6	54.2	11.1	ΣREE = 70.2%						
LLHSH	Assay	7.5	4.7	14	84	337	1790	999.4	276	321	3807.4
	Recovery	14.6	58.2	12.6	ΣREE = 72.8%						

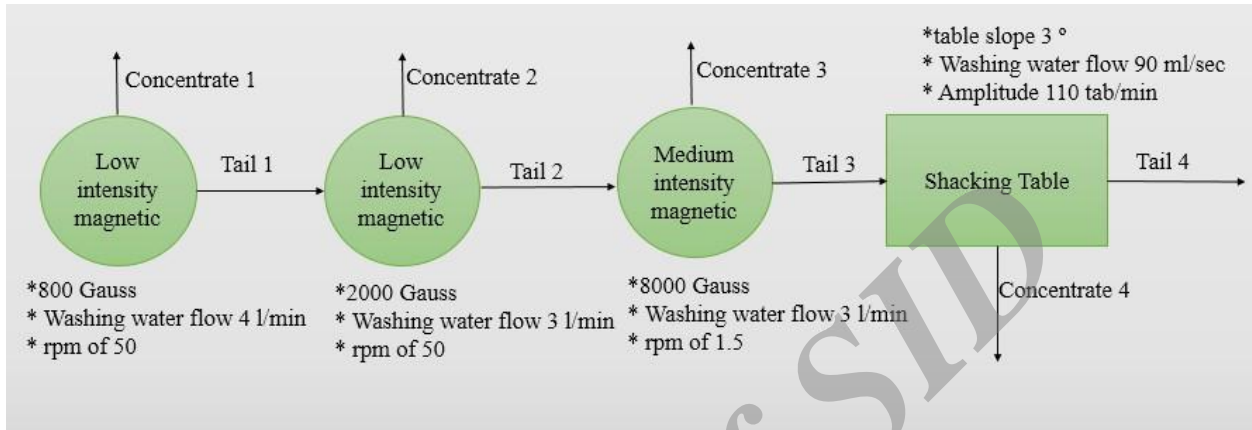


Figure 4. Effect of high gradient magnetic separation on REE separation flow sheet (LLMSH).

3.4 Effect of magnetic field intensity

Effect of magnetic field intensity on the REE separations was investigated in the LLMSH flow sheet with three different ranges (Table 5).

The range of 800-2000-8000 gauss had the best recovery. Decreasing the magnetic field intensity had negative effects on the adsorption of low magnetic ability minerals. Accordingly, low magnetic ability minerals moved to the shaking table concentrate side, whereas increasing the

magnetic field intensity had positive effects on the adsorption of low magnetic ability minerals. Hence, these minerals moved to the medium magnetic concentrate side. High amount of REEs in the magnetic concentrate (12000 gauss) proved that they had low magnetic potentials (Table 6). Thus this property is a proper item for the separation process design.

Table 5. Effect of magnetic field intensity on REE separations in the LLHSH flow sheet.

Unit		%			ppm						
Magnetic field intensity(G)		Fe	P	SiO2	Er	Gd	Y	Nd	La	Ce	ΣREE
500-1000-6000	Grade	6.9	2.9	18.3	40	201	1000	600	165	201	2207
	Recovery (%)	20.4	54.6	24.9	ΣREE = 63.7%						
650-1500-6500	Grade	7.5	4.7	14	84	337	1790	999.4	276	321	3807.4
	Recovery (%)	14.6	58.2	12.6	ΣREE = 71.8%						
800-2000-8000	Grade	7	4.9	13	85	332	1793	1005	277	340	3832
	Recovery (%)	13.3	59.5	11.4	ΣREE = 72.2%						
1000-3000-12000	Grade	6.3	4	16	65	350	1349	765	207	254	2990
	Recovery (%)	8.8	35.6	10.3	ΣREE = 42.4%						

Table 6. Effect of magnetic field intensity on REE adsorptions.

Magnetic field intensity(G)	Recovery (%)			
	Fe	P	SiO2	ΣREE
6000	16.9	4.9	3.9	3.6
8000	23.7	9.8	15.4	8.4
12000	38.3	40.2	40.5	39.3

3.5 Effect of shaking table slope

Effect of shaking table slope was investigated on the REE separations in the LLMSH flow sheet with four different slopes including 1°, 2°, 3°, and 5° (Table 7), demonstrating that increasing the

recovery decreases the REE assays. A separator with slope of 3° made the REE recoveries to reach 75.3%, and so this slope was selected as an optimum parameter.

Table 7. Effect of shaking table slope on REE separations.

Unit		%			ppm						
Shaking table slope (degree)		Fe	P	SiO ₂	Er	Gd	Y	Nd	La	Ce	ΣREE
1	Grade	8	5	13	85	356	2000	1002	300	342	4085
	Recovery (%)	11.9	47.5	8.9	ΣREE = 59%						
2	Grade	7	4.9	13	85	332	1793	1005	277	340	3832
	Recovery (%)	13.3	59.5	11.4	ΣREE = 72.2%						
3	Grade	6.5	5.5	15	79	350	1800	1003	290	350	3872
	Recovery (%)	12.8	69	13.6	ΣREE = 75.3%						
5	Grade	10	3.9	15	58	256	1445	710	200	230	2899
	Recovery (%)	24.4	60.7	16.9	ΣREE = 67.3%						

3.6 Effect of washing water flow

Effect of washing water flow on the REE separations was investigated in the LLMSH flow sheet with four different flow ranges including 50, 70, 90, and 130 mL/s (Table 8).

Washing water flow of 90 mL/s made the best REE recoveries, so this flow rate was selected as an optimum parameter. Middle density minerals

were moved to the shaking table waste due to the increase in the washing flow rate. On the other side, high density minerals were transferred to the shaking table concentrate, thus the REE recoveries and REE assays decreased and increased, respectively.

Table 8. Effect of washing water flow on REE separations.

Unit		%			ppm						
Washing water flow (mL/s)		Fe	P	SiO ₂	Er	Gd	Y	Nd	La	Ce	ΣREE
50	Assay	5.8	3	17	50	250	1200	700	200	220	2620
	Recovery (%)	14.3	47.1	19.3	ΣREE = 63.4%						
70	Assay	6.5	5.5	15	79	350	1800	1003	290	350	3872
	Recovery (%)	12.8	69	13.6	ΣREE = 75.3%						
90	Assay	6	5.5	14.7	86	335	1819	1018	293	359	3910
	Recovery (%)	11.6	67.7	13.1	ΣREE = 75.1%						
130	Assay	9	5.3	10	93	410	2221	1240	350	402	4716
	Recovery (%)	13.4	50.5	6.9	ΣREE = 68.4%						

3.7 Effect of electrostatic separation

The electrostatic separation test was performed on the shaking table concentrate to expand the REE assays at an intensity of 25000 V and a blade angle of 20° (Table 9).

REE assays increase in the non-conducted part of the electrostatic separator since the REE minerals have no electrical conduction. The relation

between REE recoveries and phosphor recovery was investigated in all the tests in Figure 6. The results obtained showed that increasing the phosphor quantity expanded the REE recoveries. It can be said that REEs concentrate in the phosphor mineral phases.

Table 9. Effect of electrostatic separation.

Unit	%			ppm							
Part	Fe	P	SiO2	Er	Gd	Y	Nd	La	Ce	ΣREE	
Concentrate 4	Assay	6	5.5	14.7	86	335	1819	1018	293	359	3910
	Recovery (%)	11.6	67.7	13.1	ΣREE = 75.1%						
None conduct	Assay	1	16.6	35	266	1060	5800	3005	893	1090	12114
	Recovery (%)	4.8	87.6	69.1	ΣREE = 89.4%						
Conduct	Assay	8	0.9	6.5	11	39	194	210	47	59	560
	Recovery (%)	94.6	11.6	31.4	ΣREE = 10.4%						

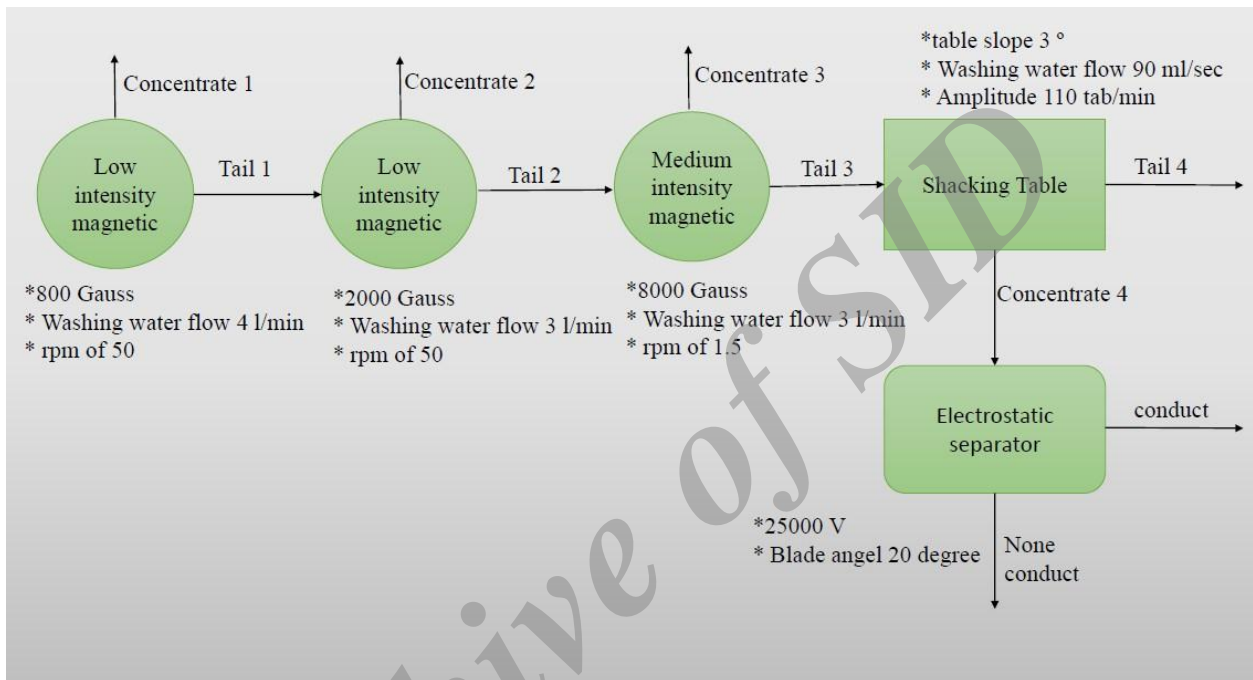


Figure 5. Best flow sheet for REE recoveries.

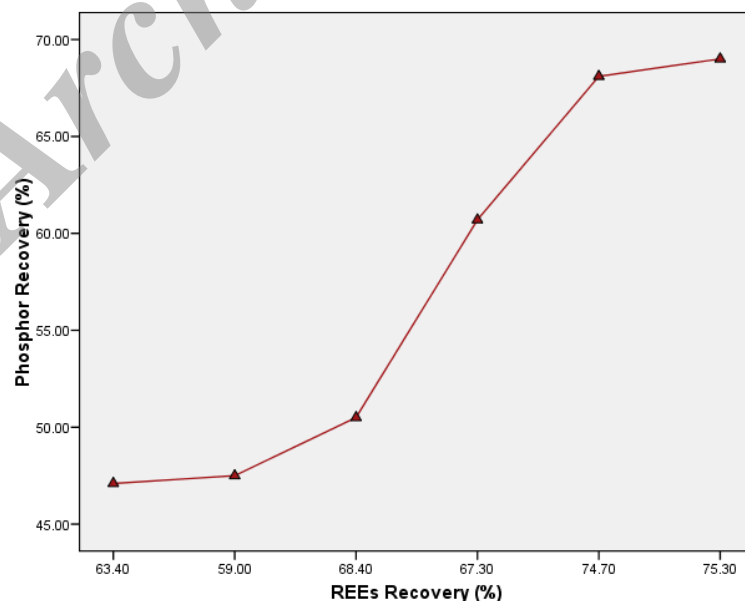


Figure 6. Relation between REE recoveries and phosphor recovery.

4. Conclusions

A number of experiments were carried out to achieve the optimum values for REE separations from iron waste. The main parameters investigated in the REE separations included particle size, gravity and magnetic separation combination, medium gradient magnetic separation, magnetic field intensity, shaking table slope, washing water flow, and electrostatic separation. Two low-intensity magnetic of 800 and 2000 gauss, one medium-intensity magnetic of 8000 gauss, one step shaking table with water flow of 90 mL/s and a table slope of 3 degree, and one electrostatic separator of 25000 V with blade angle of 20 degree had the best performance to separate REEs. Microscopic studies showed that the monazite degree of freedom was between 75 and 105 micron. The results obtained showed that the particle size of $-75 + 63$ micron was a proper range to separate REEs. The best combination of separators included two steps of low magnetic separator, one step of medium magnetic separator, shaking table, and electrostatic separator, respectively. The total recoveries and assays of REEs (Ce, La, Nd, Er, and Gd) were obtained to be 67.1 and 1.2%, respectively at the optimum situations. Also the results obtained proved that increasing the phosphor quantity increased the REE recoveries. It can be said that increasing the recovery of phosphor expands the REE recoveries in the last product.

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بازیابی عناصر نادر از باطله‌های کارخانه فرآوری آهن با استفاده از روش‌های جداسازی فیزیکی

محمد مقیسه^۱، مسعود پوررحیم^۱، بهرام رضایی^۱ و مهدی قراباغی^{۲*}

۱- دانشکده مهندسی معدن و متالوژی، دانشگاه صنعتی امیرکبیر، ایران

۲- دانشکده فنی و مهندسی، دانشکده معدن، دانشگاه تهران، ایران

ارسال ۲۰۱۵/۷/۱۸، پذیرش ۲۰۱۵/۱۱/۱۰

* نویسنده مسئول مکاتبات: gharabaghi@ut.ac.ir

چکیده:

در این تحقیق استحصال عناصر نادر با استفاده از روش‌های فرآوری فیزیکی از باطله‌های معدن آهن بررسی شده است. مطالعات میکروسکوپی و میکروپروب نشان داد که قسمت اعظم عناصر نادر در داخل کانی فلوتورآپاتیت بوده و باطله‌ها دارای محتوی آهن بالایی بودند که ضروری بود توسط روش‌های جدایش مغناطیسی مقدار آن‌ها کاهش یابد. هدف نهایی این مطالعه، بهینه‌سازی روش‌های میز لرزان، جدایش مغناطیسی و الکترواستاتیکی برای استحصال عناصر نادر بود. نتایج نشان داد که با استفاده از دو مرحله جدایش مغناطیسی شدت پایین در شدت میدان‌های ۸۰۰ و ۲۰۰۰ گوس، یک مرحله جدایش مغناطیسی شدت متوسط در میدان ۸۰۰۰ گوس و یک مرحله میز لرزان و یک مرحله جدایش الکترواستاتیکی ۲۵۰۰۰ امکان جدایش عناصر نادر فراهم است. نتایج نشان داد که با استفاده از دانه‌بندی ۶۳-۷۵ میکرون جدایش مناسب بود. در نهایت با بهینه‌سازی نتایج از خوراک با عیار عناصر نادر ۱۴۹۹ پی‌پی‌ام، کنسانتره‌ای با عیار عناصر نادر ۱/۲ درصد و بازیابی ۶۷/۱ درصد به دست آمد.

کلمات کلیدی: عناصر نادر، باطله آهن چغارت، فلوتورآپاتیت، جدایش مغناطیسی، میز لرزان.