



**The Ultimate Mineral Processing Challenge: Recovery of
Rare Earths, Phosphorus and Uranium from Florida
Phosphatic Clay**

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The Ultimate Mineral Processing Challenge: Recovery of Rare Earths, Phosphorus and Uranium from Florida Phosphatic Clay

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Abstract

Phosphate beneficiation in Florida generates over one ton of phosphatic clay (slime) per ton of phosphate rock produced. Since the beginning of the large scale washing and desliming practice for phosphate beneficiation, over two billion tons of such slime have been accumulated, which contain approximately 600 million tons of phosphate rock, 600,000 tons of rare earth elements (REE), and 80 million kilograms of uranium. However, recovery of these valuable

elements from the phosphatic clay may well be the most challenging subject in mineral processing, because the clay is very dilute (averaging 3% solids) and extremely fine in size with over 50% below 2 microns, and contains nearly 50% clay minerals as well as lots of Mg, Fe and Al. With support of the industry and under funding of the Critical Materials Institute (CMI), FIPR, in conjunction with the Oak Ridge National Laboratory, undertook a major task to recover phosphorus, rare earths and uranium from Florida phosphatic clay. This paper covers results from preliminary testing of two approaches. The first approach involved three-stage cycloning using cyclones of 12.4 cm (5 inch), 5.08 cm (2 inch) and 2.54 cm (1 inch) in diameter, respectively, to remove clay minerals followed by flotation and leaching. The second approach is a two-step leaching process. In the first step, selective leaching was conducted to remove magnesium thus allowing production of phosphoric acid suitable for DAP manufacturing in the second leaching step. Results showed that multi-stage cycloning with small cyclones is necessary to remove clay minerals. Selective leaching at about pH 3.2 using sulfuric acid was found effective for removing over 80% Mg from the feed with minimal loss of phosphorus.

Key words: Phosphate, Rare earths, Uranium, Hydrocyclone, Leaching

Introduction

Florida phosphate ore, or “matrix” as it is called locally, contains three different mineral groups which have to be separated from each other. They are phosphate minerals, clay minerals and quartz (sand). As mined, the phosphate and sand particles are embedded in compacted mud, or “clay-balls”. Before separation can begin, all the particles must be liberated from the matrix of mud. The very first unit operation in the beneficiation process is to disaggregate the various

particles; this actually starts while the matrix is flowing through the multi-mile pipeline from the mine to the beneficiation plant. While in the pipeline, the matrix is exposed to shear forces as it passes through the various centrifugal pumps along the pipeline. These intense shear forces cause a significant percentage of the sand and phosphate particles to be liberated from the clay-balls by the time they arrive at the plant. Once reaching the plant, the first goal is to finish disaggregating the clay, and follow that by making a size separation at 1 mm. This processing is conducted in the “washer” consisting of a large rotating trammel screen, a few stages of log washer, screens and hydrocyclones. In all the currently operating beneficiation plants, the washer is a large structure that receives the matrix, screens it, and then discharges a +1.0 mm “pebble” phosphate product and a -1.0 mm slurry of liberated clay, sand, and phosphate particles.

The next process objective is to remove the clays. Since the clays are finer than 0.1 mm, to reject the clay all that is required is to size at 0.1 mm and discard the fine fraction. This is accomplished using hydrocyclones about 50.8 cm (20 inch) in diameter. The minus 0.1mm fraction is called phosphatic clay or slime, which represents at least one third of the mined ore and is pumped as a slurry of 3% solids into clay settling pond.

Rare earths, phosphate and uranium losses in the phosphatic clays. Since the industry started large scale washing/desliming practice for phosphate beneficiation, about 2 billion tons of phosphatic clays have been accumulated in Florida. The waste clays not only create one of the most difficult disposal problems, but also represents a tremendous loss of valuable and critical elements including phosphorus, rare earth elements, and uranium. In some operations about one third of the P_2O_5 mined ends up in the phosphatic clays. This loss has been repeatedly verified by many researchers. One way to comprehend the magnitude of phosphate loss in the phosphatic clays is to analyze the phosphate content in the clay. Table 1 summarizes some early results. It

may be seen that some phosphatic clays have a much higher phosphate content than the phosphate ores currently being processed.

Table 1– Concentration of P_2O_5 in the past phosphatic clays.

Time	Author/Organization	% P_2O_5
1954	National Academy of Science	20.0
1963	Gary et al., USBM	13.1
1969	Davenport and Watkins, TVA	17.7
1971	IMC	12.3
1975	Staff, USBM	9-17
1982	Jordan et al, USBM	6-15.9
1987	Ardaman & Associates	4.6-17.9

As the Florida's phosphate reserves diminish, it becomes more and more important to recover the phosphate values from these slimes.

According to the International Atomic Energy Agency, uranium in world phosphate deposits accounts for over 88 percent of the world's unconventional uranium resources, Figure 1. Florida phosphate contains higher content of uranium than many of the other phosphate deposits in the world.

More importantly, REE in Florida phosphate is of higher value due to higher HREE to LREE ratio. One analysis of a typical phosphate clay shows that the total REE in the sample consists of

28.09% Y, 20.05% HREE, and 50.66% LREE.

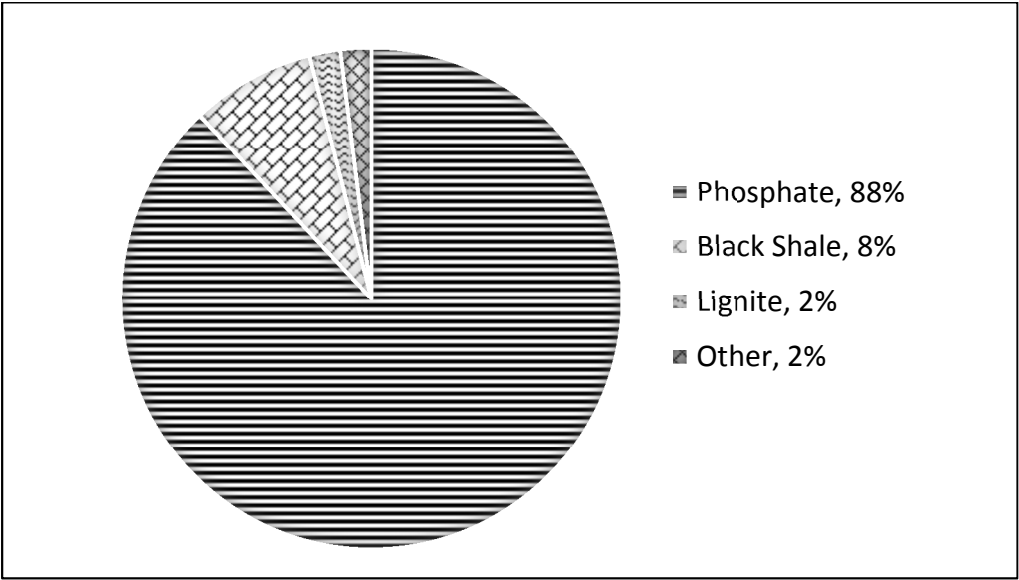


Figure 1 – Various unconventional uranium resources in the world.

Published analyses, decades old, of trace elements in Florida phosphate rock have shown that many of the critical rare earth elements are present. Though only present in trace amounts, because of the tonnage of phosphate produced, these elements are still significant in aggregate mass and have not been recovered.

A comprehensive investigation of REE in Florida phosphate was conducted by Kremer and Chokshi (1989) of Mobil Research & Development Corporation. The total REE in Florida phosphate matrix analyzed 282 ppm (88 ppm neodymium, 68 ppm cerium, 57 ppm yttrium, and 49 ppm lanthanum, accounting for 90%). Distributions of REE in the mining and chemical processing streams were also determined, showing 40% in waste clay, 37.5% in PG, 12.5% in phosphoric acid, and 10% in sand tailings. Data further indicated that REEs were concentrated in fine phosphate particles, as the pebble product analyzed 284 ppm REE versus 575 ppm in the

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3 flotation concentrate. The high REE concentration (336 ppm) in waste clay is another evidence
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5 of REE concentrating in fine phosphate particles.
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8 Another set of data by the USGS (Altschuler et al, 1967) showed that Florida phosphate rock
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10 contained about 500 ppm REE, with 150 ppm lanthanum, 120 ppm cerium and 110 ppm yttrium.
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12 According to a report by the former USBM (May and Sweeney, 1983), Florida
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14 phosphogypsum contained 300 ppm REE, with 130-170 ppm gadolinium, 49 ppm cerium and 39
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16 ppm lanthanum.
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20 Assuming averaging concentrations of 9% P_2O_5 , 300 ppm REE and 40 ppm U, the 2 billion
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22 tons of accumulated phosphatic clay in Florida represent approximately 600 million tons of
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24 phosphate rock, 600,000 tons of rare earth elements (REE), and 80 million kilograms of uranium.
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32 **Previous research.** Recovery of the % P_2O_5 values from the waste clays is not a novel
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34 concept. Hazen Research was issued a patent (Hazen, et al, 1969) on a process to leach the clays
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36 with sulfuric acid, then to recover the % P_2O_5 from the acid leachate with mixtures of amine and
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38 alcohol in a solvent extraction step. Ribas and Nickerson (1976) proposed to recover the
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40 phosphate values by consolidating, calcining at 870-1093°C, and then digesting the clay in
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42 mineral acid to produce phosphoric acid. The process was energy intensive and created severe
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44 dusting problems during calcination because of the dry, finely-divided particles.
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48 The U.S. Bureau of Mines carried out a detailed investigation (Powell and Calhoun, 1963) in
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50 which the clay was first sized using hydrocyclones and hydroseparators. Up to 96% of the plus
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52 400 mesh material was recovered during the sizing step. Subsequent flotation of the plus 400
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54 mesh fraction gave a concentrate of over 30% % P_2O_5 at an 88% recovery from this coarse
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fraction. However, P_2O_5 recovery from the total clay was low.

The Florida Institute of Phosphate Research (FIPR) sponsored a project (Jha et al., 1985), which used an autoclave acidulation technique to recover the phosphate values from clays. At a temperature of $175^{\circ}C$ more than 90% of the % P_2O_5 was extracted in less than an hour. After acid treatment, the clay residue quickly consolidated to a 35-45% solids level. Longer leaching time led to larger phosphogypsum crystals which helped the entire mass to filter more readily. However, the equipment investment and poor acid quality prevented this process from being commercialized.

Other techniques investigated include flocculation (Andersen and Somasundaran, 1993; Arol and Iwasaki, 1987; Gu and Doner, 1993; Sresty, 1977), microbial methods (Davis, 1965), and flotation (Duke, 1961; Greene and Duke, 1962; Smit, 1959; Sun and Smit, 1963). A common conclusion from most of the previous studies was that recovering phosphate from the slimes is either technically impossible or economically prohibitive (Zhang, et al., 1995).

Although desliming at about 150 mesh is carried out at all the plants, the phosphatic clays are extremely fine. Table 2 (Zhang et al., 1995) shows that the minus 1 micron fraction accounts for about 35-50% of the total slime mass.

Table 2 – Size (micron) distribution of four composite phosphatic clay samples.								
Sample	+44	-44+20	-20+15	-15+10	-10+5	-5+2	-2+1	-1
M010a	15.25	11.45	4.68	6.50	7.00	8.82	10.65	35.65

N010a	4.31	7.39	3.87	5.87	7.06	10.03	11.98	49.49
C010a	1.25	8.11	8.32	8.94	6.86	8.00	5.72	52.81
G010a	1.53	7.28	10.41	9.76	9.87	10.55	6.91	43.78

Table 3 lists the major chemical compositions of the four composite samples (Zhang et al., 1995). Perhaps, the first conclusion one can draw from Table 3 is that phosphate loss in current beneficiation operations is still tremendous, with the lowest P_2O_5 content averaging 6.09%. Another noticeable characteristic is the high MgO content. This means that even if one successfully removes the clay minerals, a dolomite separation process may be necessary before the final product would be acceptable.

Table 3 – Major chemical compositions of four composite phosphatic clay samples.

Sample ID	P_2O_5 %	Insol %	Al_2O_3 %	CaO %	MgO %	Fe_2O_3 %
M010a	12.92	32.57	6.43	22.46	4.01	1.63
N010a	14.56	35.36	8.75	22.04	1.79	2.87
C010a	6.97	43.27	4.93	14.82	4.23	1.21
G010a	6.09	48.51	4.88	15.86	5.36	2.01

Experimental

Sample analysis and characterization. Solid sample analysis involves two parts. The first part is analysis of the routine six, P_2O_5 , Insol (acid insoluble), Al_2O_3 , CaO, MgO and Fe_2O_3 by acid digestion using the standard method developed by the Association of Fertilizer and

Phosphate Chemists. After digestion, P_2O_5 and CaO are analyzed using an automatic colorimetric analyzer. Insol is measured by drying and weighing the digestion residue. Al, Mg and Fe are determined using an ICP-OES. The second part is analysis of REE, U and Tr using an ICP-MS. The digestion procedure is a modified method developed by USGS. In all our chemical analyses, the relative deviations of mass balances for P_2O_5 and total REEs were set at less than 2.0 and 3.0. Any results above these limits were subject to either repeat chemical analysis or rerun the experiment. In order to determine REE and P distribution in different size fractions, a laboratory cyclonsizer is used to split a clay sample into six fractions.

Cyclone Testing. A 12.7-cm (five-inch) hydrocyclone was used for separating the fine clay minerals from the coarse, non-clay minerals. In these tests, several drums of phosphatic clay samples were mixed to get uniform slurry of 5-6% solids. This composite sample was then split and placed into different drums. The cyclone pump directly sends the feed to the cyclone while keeping the drum mixed. Both underflow and overflow samples were collected for chemical and mass balance analyses. Each test used one drum of sample. These tests were conducted to evaluate the effect of cyclone parameters. After the batch testing, the 12.7-cm cyclone pilot plant was hauled to an operating mine for continuous testing to generate large samples for downstream process development.

In subsequent testing, the overflow sample from in-plant testing was further sized using a 5.08 cm and a 2.54 cm cyclone, respectively.

Flotation of cyclone underflow sample. Flotation was conducted using a laboratory scale Denver flotation machine. In a flotation test, the pH of flotation pulp of 30 solids is first adjusted

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3 to about 9 with soda ash, and then conditioned with the collector (a tall oil fatty acid) for 4
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5 minutes, followed flotation to completion.
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10 **Leaching of cyclone underflow.** The leaching apparatus consists of a glass reactor, a
11 vacuum system, an acid pumping system, temperature and pH control mechanisms, a vacuum
12 sampling system, and computer display. A two-liter sealed glass reactor is heated by a 500 W
13 heating mantle. The temperature is controlled automatically using a probe placed into the center
14 of the reactor through a thermal well. The accuracy of temperature control is ± 0.2 versus ± 0.1
15 for pH. The pH probe was calibrated as a function of temperature. The slurry is kept suspended
16 during leaching by a shaft driven stirrer. The pH is monitored using an industrial probe, and the
17 pH signals are recorded by a computer which relays control signals to the acid dispensing
18 syringe pump.
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31 One vacuum system is set to expel CO_2 from the reactor through a water cooled condenser,
32 and the condensed water is used to dilute the acid before entering the reactor. Another vacuum
33 system is installed to take the slurry samples with a sample well in the middle of the slurry.
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43 **Results and discussion**

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47 **Waste clay characterization and analysis.** Chemical analysis of four slime samples is
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Table 4 – Chemical analysis of four recent phosphatic clay samples (all in ppm with P₂O₅ in wt%).

Sample	P ₂ O ₅	U	La	Ce	Y	Nd	Gd	Pr	Total REE
A	5.60	37.30	41.66	85.96	60.17	53.67	10.12	2.60	287.62
B	7.12	77.04	39.96	75.51	59.94	45.06	7.44	3.65	301.36
C	12.98	71.10	74.80	132.88	142.31	87.44	17.65	1.65	515.14
D	2.76	--	13.00	24.00	22.00	21.00	4.20	4.20	122.00

These results show that the current phosphatic clay averages about 300 ppm REE, 7% P₂O₅, and 40-50 ppm U.

The hydrocyclone overflow product of phosphatic clay waste from Four Corner was classified using a laboratory cyclosizer with water as the medium. Each time, 20 grams of the overflow clay were added to the sample container of the cyclosizer, and after classification 6 products with different size fraction were produced. A total of 40 grams of the fine clay were used for two tests. The test conditions are as follows: Temperature: 25°C; Water flowrate: 11.6 liters per minute; Time of elutriation: 5 minutes. The sample was split into six size fraction, results are shown in Table 5.

Table 5 – Analysis of different size fractions of a phosphatic clay sample.

Size fraction	Yield/%	Content	Distribution
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/micron		Total REE/ppm	P ₂ O ₅ /%	Total REE/%	P ₂ O ₅ /%
+41	13.25	358.78	14.01	17.16	15.71
-41+31	9.50	210.70	8.72	7.24	7.01
-31+22	9.25	228.70	9.46	7.67	7.40
-22+14	17.00	238.70	11.18	14.60	16.08
-14+10	4.00	229.07	11.62	3.29	3.93
-10	47.00	298.41	12.54	50.04	49.87
Weighted average	100.00	278.70	11.82	100.00	100.00
Clay feed	-	278.58	11.34	-	-

These results show no significant concentration for either REE or P₂O₅ in any size fraction. This also indicates that further sizing below 10 micron is necessary in order to determine the optimum separation size for removing clay minerals and the right cyclone size to achieve the separation.

Chemical and mineralogical analyses under a separate project showed that rare earth elements exist in three major forms: as monazite mineral, as xenotime, and substitution of calcium in phosphate crystals. Nearly all the uranium in sedimentary phosphate exists in isomorphous substitution for Ca in phosphate. Rare earths were also found to follow phosphate. Therefore, the key to recovering REE and U is to concentrate phosphate first.

Cyclone testing results. The in-plant cyclone testing results are summarized in Tables 6 and

7. These results show that the 20.7-cm (5-inch) cyclone is too big for removing the clay minerals in phosphatic clay. However, the process did produce an underflow more manageable in terms of contaminants such as Al and Fe. It should be pointed that higher pressure would improve separation. However, as we will see below,

Table 6 – Effect of apex diameter and pressure on particle distribution.

Pressure (psi)	Apex diameter (mm)					
	9.0		9.5		12.5	
	Overflow	Underflow	Overflow	Underflow	Overflow	Underflow
	D ₉₀ ,µm	D ₁₀ ,µm	D ₉₀ ,µm	D ₁₀ ,µm	D ₉₀ ,µm	D ₁₀ ,µm
4.0	7.946	17.106	6.494	10.387	8.991	3.180
6.0	8.017	17.561	7.302	12.485	9.520	7.025
8.0	8.328	18.241	8.552	17.444	8.070	8.176
10.0	8.396	19.026	7.446	15.372	6.731	7.576

Table 7 – Separation results summary.

Product	% weight	ΣREE	% ΣREE	% P ₂ O ₅	% Fe ₂ O ₃	% Al ₂ O ₃
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	yield	(ppm)	recovery			
Overflow	79.32	326.86	82.47	10.82	4.01	7.83
Underflow	20.68	266.46	17.53	10.10	2.22	2.57
Weighted average	100.00	314.37	100.00	10.67	3.64	6.74

Preliminary flotation results. The underflow product of cyclone testing was subject to anionic flotation with sodium carbonate as a pH modifier and sodium silicate as a depressant. The collector was a mixture of fatty acid and fuel oil at a 1: 0.5 ratio. Figures 2-4 show the effect of pH modifier, collector and depressant dosage on rare earths recovery and grade. It should be noted that REE recovery value of the fourth data point in Figure 6 is smaller than the others. Since it is very small, it may not represent a meaningful change in depression mechanism.

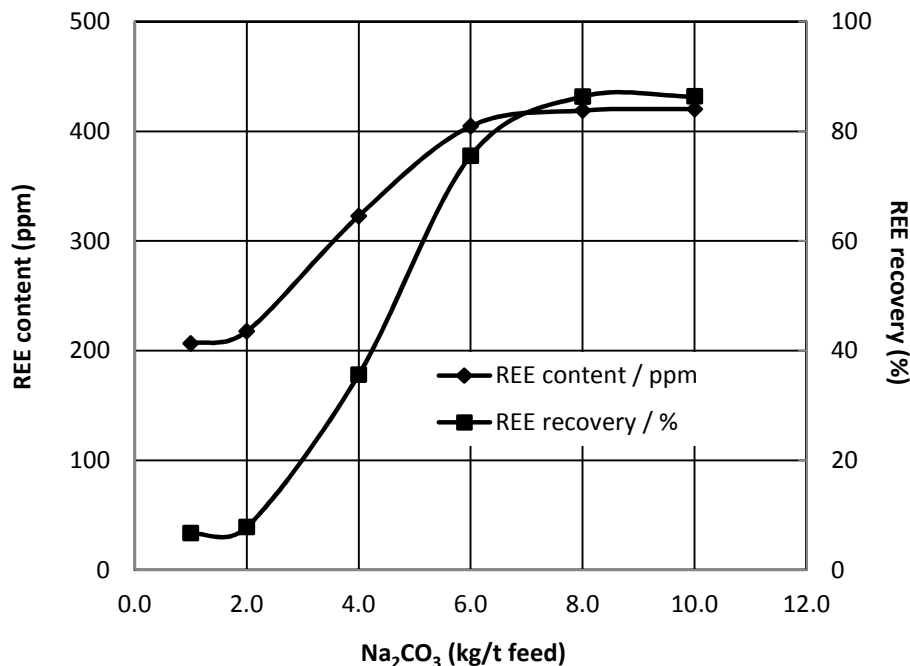


Figure 2 – Effect of pH modifier on REE content in concentrate and recovery at collector

dosage of 2.5 kg/ton and depressant dosage of 1.8 kg/ton of feed.

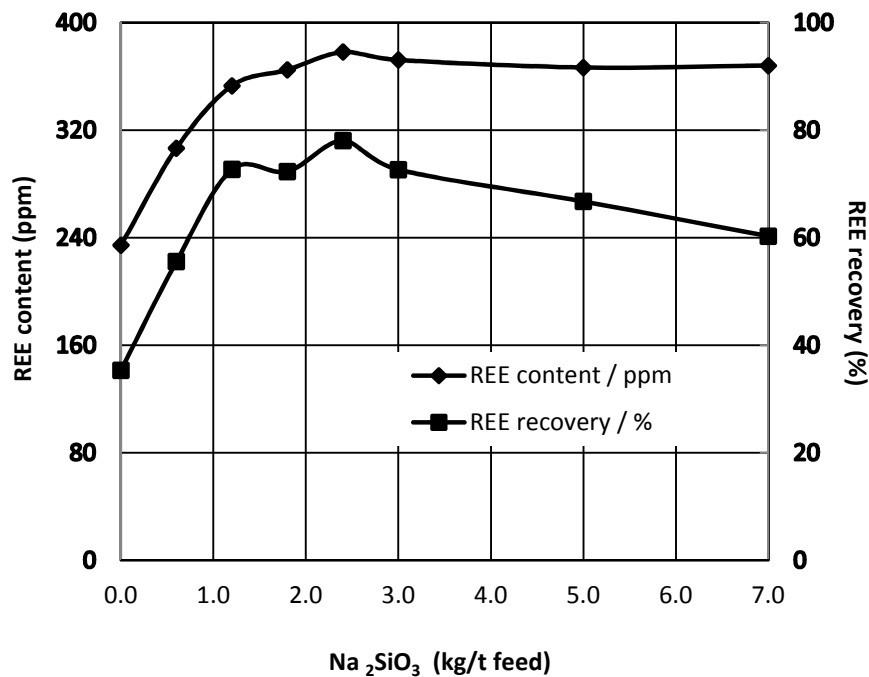


Figure 3 – Effect of depressant on REE content in concentrate and recovery at collector dosage of 0.5 kg/ton and pH modifier dosage of 8 kg/ton of feed.

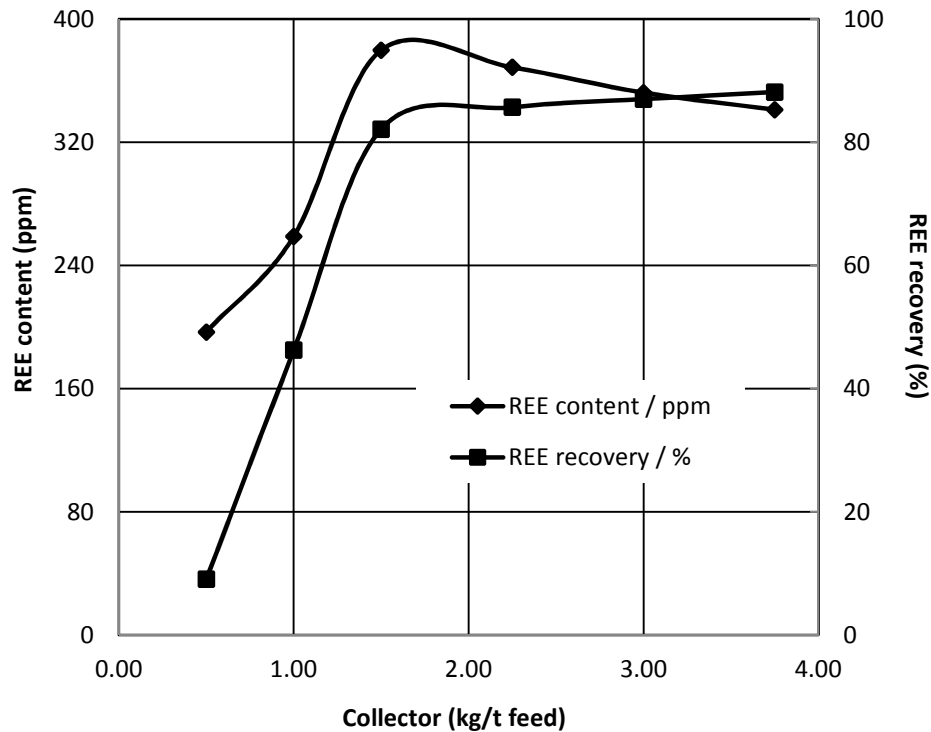


Figure 4 – Effect of collector dosage on REE content in concentrate and recovery at pH modifier dosage of 8 kg/ton and depressant dosage of 2.4 kg/ton of feed.

Table 8 summarizes flotation results from one of the best tests, showing 88% recovery of total rare earths and over 94% P_2O_5 recovery.

Table 8 – Flotation results on the cyclone underflow.

Product	% yield	Σ REE	% Σ REE	% P_2O_5	% P_2O_5
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		(ppm)	recovery		recovery
Concentrate	54.18	420.38	88.16	12.68	94.22
Tailings	45.82	79.50	11.84	0.92	5.78
Weighted average	100.00	209.73	100.00	7.29	100.00

Two-stage leaching. Another approach that we proposed for the cyclone underflow is two-stage leaching. Recovery of the phosphate value is critical to obtaining the industry’s endorsement for a technology to recover REE from waste clay. This requires removal of carbonates (mainly in the form of dolomite). Phosphoric acid manufacturing requires a phosphate rock feed with less than 1% MgO, while the waste clay contains up to 9%. FIPR recently developed a selective leaching technology, which can remove carbonate from phosphate ore by leaching with sulfuric acid at pH about 3.2. After the MgO is removed at low pH level, P and REE can be leached by increasing the acid concentration.

Although H₂SO₄ leaching for MgO removal is nothing new, this technology is innovative in the following respects:

- Carbon dioxide produced by leaching was expelled by boiling to enhance reaction rates.
- pH was accurately controlled by computer relayed control signals, with temperature compensation. Since pH is the most important variable affecting both leaching rate and phosphate loss, close control of the pH is vital for the success of this process.
- The soluble magnesium is converted to magnesium nitrate in the leachate by adding an appropriate amount of nitric acid. It is easier to precipitate MgO from Mg(NO₃)₂ than from MgSO₄.

- The results indicated that phosphate loss could be held as low as 0.01% even with 89% removal of the original MgO. Compared to the conventional process, a net gain could be achieved using this process.

The stage one leaching was done on a clay sample containing about 4% MgO. As is shown in Figure 5, a majority of the MgO was removed rather rapidly using the dilute acid leaching technique with a small phosphate loss.

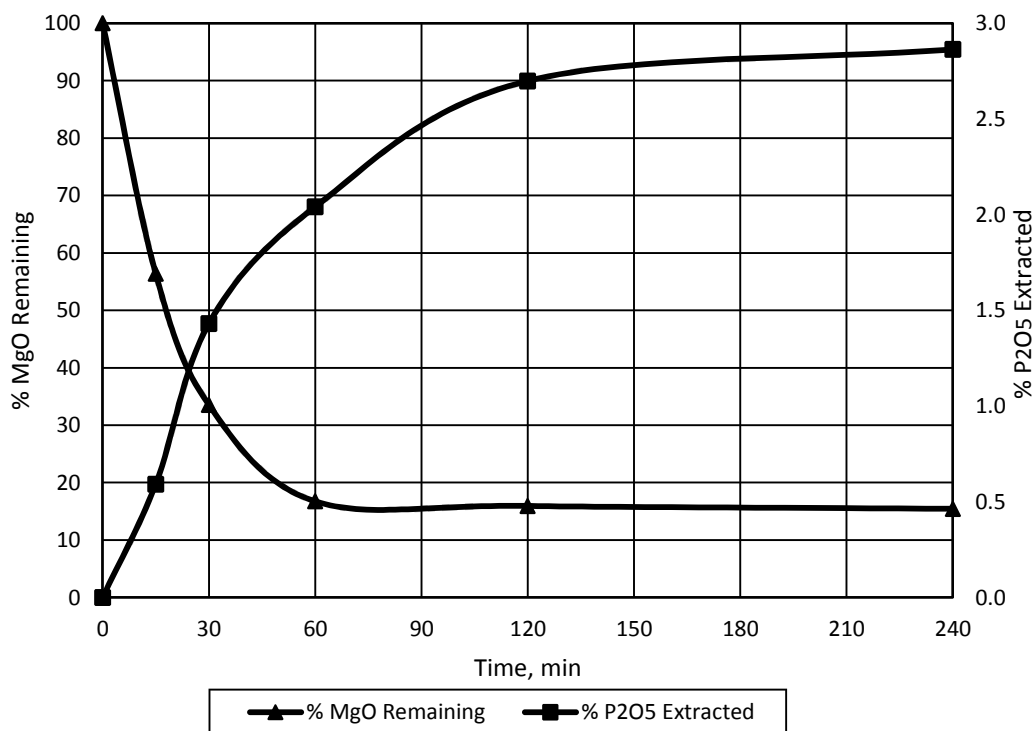


Figure 5 – Effect of leaching time on MgO removal and phosphate loss.

Conclusions

The accumulated 2 billion tons of phosphatic clay contain approximately 600 million tons of phosphate rock, 600,000 tons of rare earth elements (REE), and 80 million kilograms of uranium.

Removal of the clay minerals in phosphatic clay requires small cyclones of 2.54cm to 5.08 cm in diameter and cut at near one micron.

If a majority of the clay minerals is removed, the coarse fraction can be processed by flotation, with over 80% recovery of phosphate and rare earths. It should be pointed out that should be about the same as that of phosphate, because nearly all the uranium reports to phosphate mineral.

Selective leaching using dilute phosphoric acid is very effective for removing MgO in pre-concentrated phosphatic clay, which dramatically improves the economics for recovering rare earths and uranium because phosphoric acid quality would be more suitable for high value fertilizer production.

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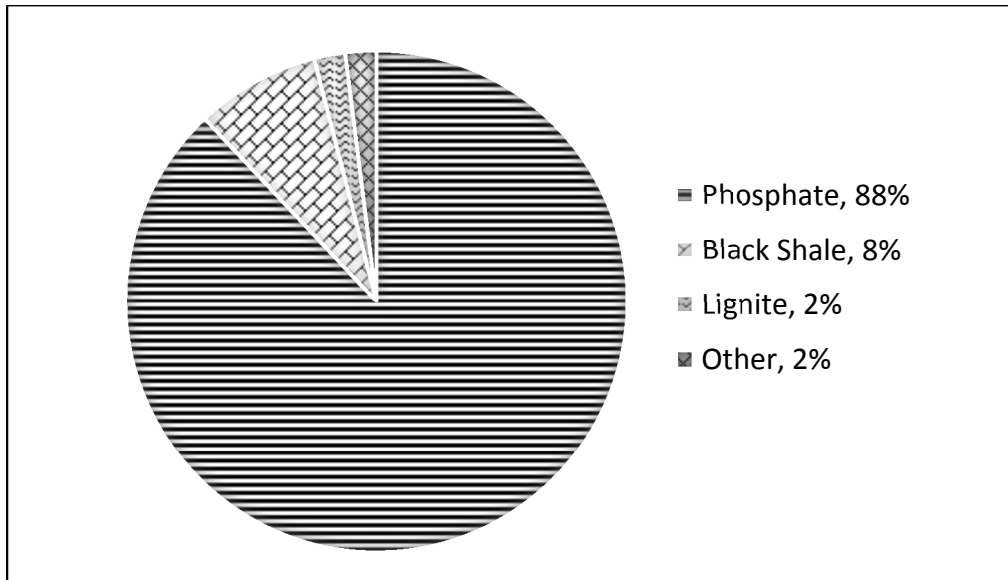


Figure 1 – Various unconventional uranium resources in the world.

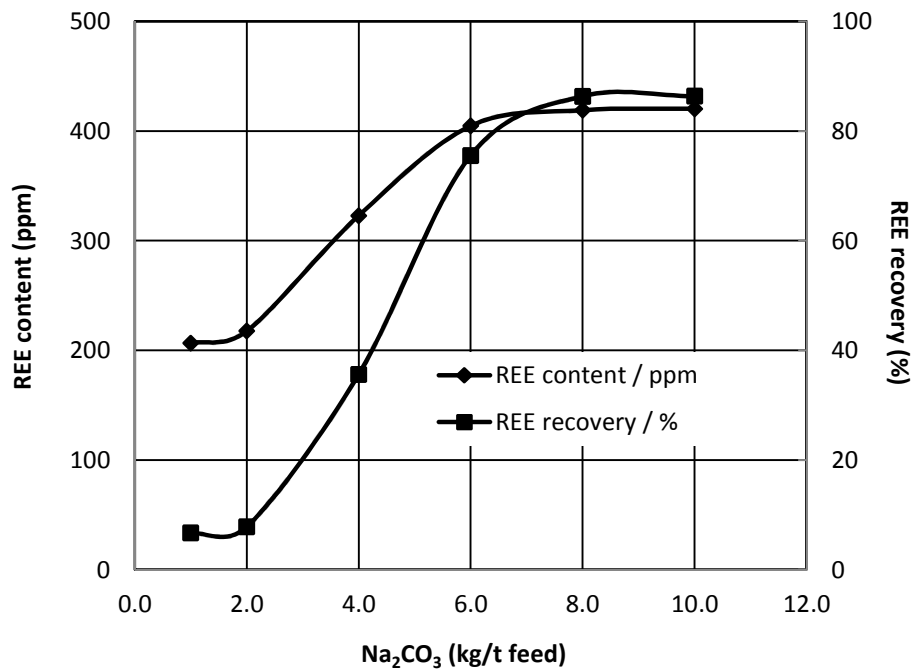


Figure 2 – Effect of pH modifier on REE content in concentrate and recovery at collector dosage of 2.5 kg/ton and depressant dosage of 1.8 kg/ton of feed.

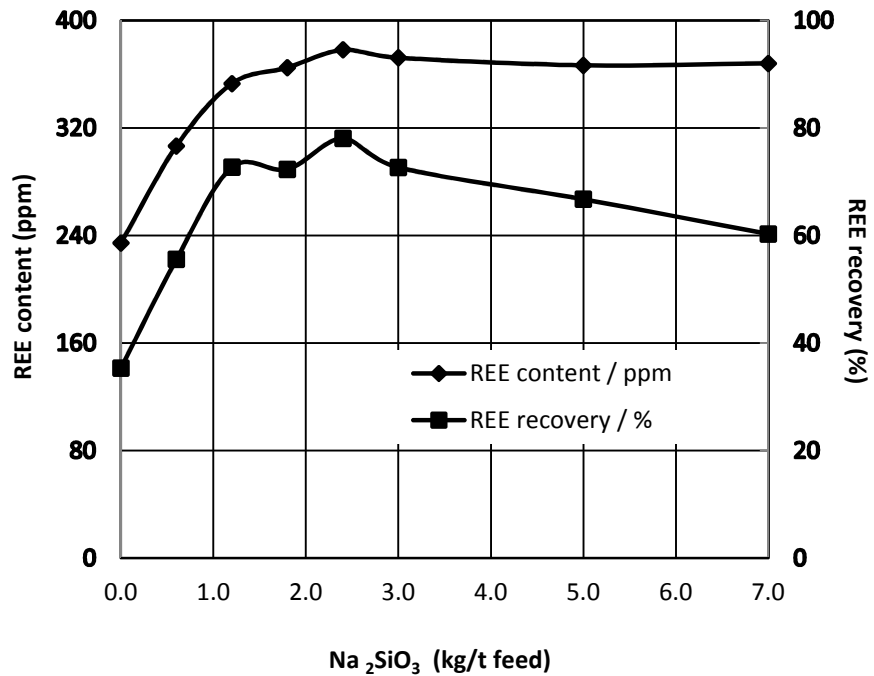


Figure 3 – Effect of depressant on REE content in concentrate and recovery at collector dosage of 0.5 kg/ton and pH modifier dosage of 8 kg/ton of feed.

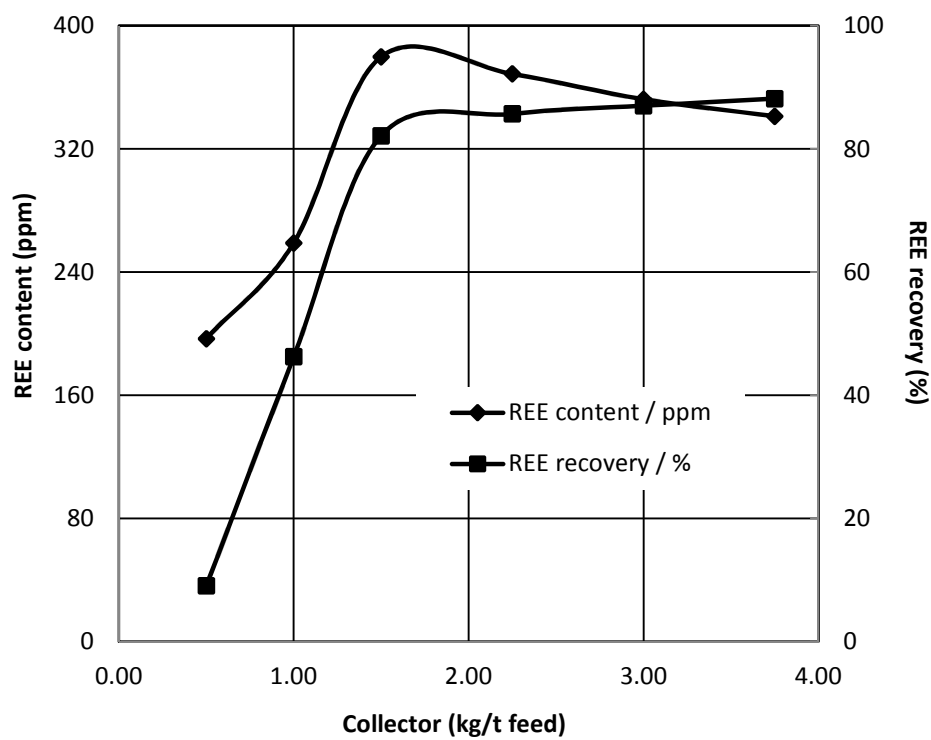


Figure 4– Effect of collector dosage on REE content in concentrate and recovery at pH modifier dosage of 8 kg/ton and depressant dosage of 2.4 kg/ton of feed.

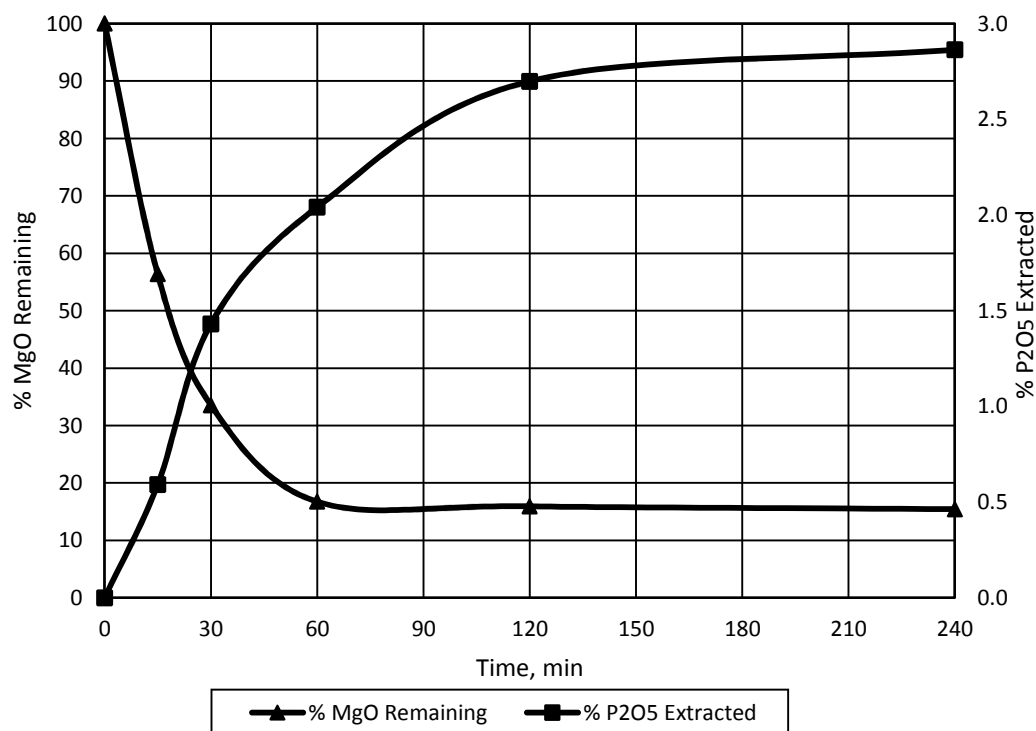


Figure 5 – Effect of leaching time on MgO removal and phosphate loss.