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LEACHING BEHAVIOUR OF PHOSPHOGYPSUM WITH DIFFERENT ACIDS

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Abstract
This study was conducted with different experimental conditions such as acids, temperatures, and reaction times for investigating leaching behavior for rare earth element (REE) leaching. In this study, phosphogypsum (PG) sample was obtained from a real PG plant in Florida and sample characteraziton was evaluated for REE, P_2O_5, ^{238}U, and ^{232}Th contents. Then preliminary leaching experiments were conducted as kinetic experiments with 9 different experimental conditions. These leaching tests showed that leaching yield was not so high. PG sample's leaching was so difficult, complex to get into solution also the REE concentration was small.

Keywords:*Leaching, phosphogypsum, acids*

1. INTRODUCTION

Many research has recently been conducted on the end-of-life products containing high concentrations of REEs, such as lamps or magnets, there is less attention has been paid to industrial landfilled stocks [1] such as PG. However, PG seems contain lower REE concentrations but have huge volume and considered a promising source of REEs also environmental concern due to the high content of metals, metalloids and radionuclides [1] and also various impurities such as residual acid (P_2O_5), fluorine compound [2-4]. In this regard, PG considered as a significantly value added component for both environmentally and techno-economic side due to REEs and radioactive elements content.

It is quite difficult to selectively separate REEs and radionuclides from PG. Because REE and radionuclides in PG are in low concentration and both pass into solution with applied acidic leaching methods. As a second step, a selective recovery method or its ability to be selectively leaching in a more economically effective way should be considered. Inorganic acids could leach REE and some radionuclides like U. In this study preliminary test results were evaluated for leaching of REEs from PG.

2. EXPERIMENTAL

In this study, PG sample was obtained from a real PG plant in Florida and sample characteraziton was evaluated for REE, P_2O_5, ^{238}U, and ^{232}Th contents. Then preliminary leaching experiments were conducted as kinetic experiments with 9 different experimental conditions. Preliminary experiments were conducted to determine optimum leaching acids for subsequent experiments. In this purpose, 9 experimental set up was conducted with H_2SO_4, citric acid, ascorbic acid, boric acid, and combination of inorganic and organic acids.

For leaching experiments, leaching experiments at room temperatures conducted in orbital shaker in 0.7 L solution while the experiment at 60 °C was conducted in three necked temperature controlled reactor as 1.3 L. Leaching experiments were conducted with 15% S/L ratio of PG

sample. For this purpose, for experiment 7, firstly 1 M H_2SO_4 was prepared and put in the reactor and the temperature of solution acid was controlled. After obtaining 60°C in the solution, calculated citric acid and PG sample was added to solution and solution was mixed at 350 rpm. After 30 min, 1-2-3-4-6-8 h later approximately 15 mL sample were taken and centrifuged at 5000 rpm for 5 min. After liquid phase separated and REE, P_2O_5, ^{238}U, and ^{232}Th concentration was analyzed with ICP-OES. After 8 h experimental time, the solution was filtered and washed approximately 500 mL distilled water, then filtrate was dried at oven at 105 °C and then after grinding, REE, P_2O_5, ^{238}U, and ^{232}Th concentration of residues were also measured with ICP-OES. After 8 h experimental time pH of solution was also measured. Table 1 shows preliminary experimental leaching conditions.

Table 1. Preliminary experimental leaching conditions

Experiments	PG samples	Acids	Concentrations	Temperature
1	15% S/L raw	H_2SO_4	1 M	Room T.
2	15% S/L raw	H_2SO_4 Citric acid	1 M 20 g/L	Room T
3	15% S/L raw	Citric acid	20 g/L	Room T
4	15% S/L raw	H_2SO_4 Ascorbic acid	1 M 20 g/L	Room T
5	15% S/L raw	H_2SO_4 Citric acid	1 M 40 g/L	Room T
6	15% S/L raw	Ascorbic acid	20 g/L	Room T
7	15% S/L raw	H_2SO_4 Citric acid	1 M 20 g/L	60 °C
8	15% S/L raw	Oxalic acid	20 g/L	Room T
9	15% S/L raw	Boric acid	20 g/L	Room T

3. RESULTS AND DISCUSSION

Characterization results shows that raw PG sample contains 230.64 ppm REEs with 13.23 ppm U and 0.63 ppm Th. Leaching results of impurities, REEs and radionuclides with 1 M H_2SO_4 was shown in Table 2.

Table 2. Leaching results of impurities, REEs and radionuclides with 1 M H_2SO_4 (15% S/L, room temp.)

Reaction Time	P_2O_5	Insol	MgO	Al_2O_3	Fe_2O_3	CaO	Total REE	U	Th
h	%						ppm		
0.5	0,00		0,00	0,00	0,00	0,00	9,01	0,50	0,00
1	0,00		0,00	0,00	0,00	0,00	10,02	0,56	0,00
2	0,00		0,00	0,00	0,00	0,00	10,23	0,62	0,00
3	0,08		0,00	0,01	0,01	0,00	11,48	0,77	0,00
4	0,07		0,00	0,01	0,01	0,00	15,10	1,27	0,00
6	0,08		0,00	0,01	0,01	0,00	11,76	0,83	0,00
8	0,08		0,00	0,01	0,01	0,00	12,39	0,88	0,00
Residue	0,02	10,80	0,01	0,09	0,09	25,81	184,44	4,17	0,43

Leaching results with 1 M H_2SO_4 at room temperature showed that REE recovery was increased with reaction time till 4 h (Table 2). After 4 h leaching recovery was decreased. Lower REEs recovery was obtained with 1 M H_2SO_4 at room temperature. It may be due to the using lower acid concentration and working at room temperature. Leaching results with other conditions was given as only results without any tables. Leaching with 1 M H_2SO_4 and 20 g/L citric acid for REEs and U leaching were increased with increasing leaching time. However, adding 20 g/L citric acid

concentration to 1 M H_2SO_4 could not reach the recovery yields comparing to experimental results conducted with 1 M H_2SO_4. While the experiments were conducted with only 20 g/L citric acid, REEs concentrations were about 3-6 ppm for all reaction time and did not change with increasing reaction time. The recovery yield of both REEs and U were lower than leaching results obtained with 1 M H_2SO_4 and 1 M H_2SO_4 with 20 g/L citric acid. Also, the leaching results with only 20 gl/L ascorbic acid could not leach REEs from PG at studied conditions. These results show that, inorganic acids showed better removal efficiency rather than organic acid.

When the leaching temperature increased to 60°C, increasing temperature could not achieve the REE extraction however slightly increase U leaching. Also, leaching experiments were conducted with 20 g/L oxalic acid and 20 g/L boric acid. There was no REE leaching with 20 g/L oxalic acid and 20 g/L boric acid. The solution pH was 1.22 in oxalic acid solution and 2.64 in boric acid solution. In our previous experience, these organic acids could consider while the selective leaching of metals.

4. CONCLUSION

This study was conducted with different experimental conditions such as acids, temperatures, and reaction times for investigating leaching behavior for REE leaching. These leaching tests showed that leaching yield was not so high. PG sample's leaching was so difficult, complex to get into solution also the REE concentration was small.

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