Conference Paper

Comprehensive Processing Technology of Slags of Phosphorus Industry To Produce Precipitated Silica and Rare-Earth Concentrate

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Abstract

The report presents a technological scheme of complex processing of man-made waste of phosphorus production - slag to produce highly dispersed silicon dioxide and a rare-earth metal concentrate. The technological parameters of each operation of the technology are chosen. Recovery of rare-earth metals from phosphorus slags during various operations was, %: leaching – 95.0; extraction - 90.2; stripping on the average ~ 93.2; the production of $\sum$ REM oxide concentrate is 93.9. The composition of some batches of "white soot" obtained during processing of the technological scheme is shown.

Keywords: Phosphorus slag, Leaching, Extraction, Stripping, Rare-earth metals, White soot, concentrate of $\sum$ REM oxide

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1. Introduction

Many studies have been devoted to the utilization of phosphorus slags. Most of them are targeted to processing phosphorus slags for use in building materials and products made of them: technologies have been developed for the production of granular slags, slag crushed stone, slag pumice, mineral wool, slag stone, cast and other building products and materials [1]. One of the applications of granular phosphorus slag is to use it to strengthen saline soils, as well as, to create strong and cheap structural layers of road toppings.

However, in spite of large effort made to dispose phosphorus slag, most of it still remains in dumps, thus worsening the ecological situation in the Southern regions of Kazakhstan. One of the reasons for the inefficient use of slags today is a low cost of products prepared from it, thus making the processing of slags economically unprofitable. Therefore, it is important to find ways to process phosphorus slag to obtain high value-added products strongly demanded by industry.

The preparation of precipitated highly dispersed silica from phosphorus slag is the direction of high priority today.

Recently an increase in demand for amorphous nanodisperse silica is observed. In addition to its traditional use as additives in rubber, plastics, paper, for making glues, liquid glass, ceramics, adsorbents, etc., the consumption of nanodispersed chemically pure amorphous silica in high-tech industries, such as the production of light guides, semiconductor silicon, new photomaterials, ceramic oxides, highly dispersed abrasives, catalysts, sorbents for chromatography, for the determination of various inorganic and organic compounds as with the help of analytical instruments (spectrophotometers, reflectometers), and with the use of indicator tubes, drugs and cosmetics.

Phosphorus slag with the content of SiO_2 35-42 % can serve as a source for production of precipitated silica [2, 3]. Developments for obtaining mineral fillers from wastes of the phosphoric industry were carried out at JSC "Institute of Metallurgy and Beneficiation" in collaboration with specialists from phosphorus plants [4].

The recovery of rare-earth metals (REM) along with the production of precipitated silicon dioxide from slag is also of great interest.

2. Results and Discussion

The object of research was slag of phosphorus production of Zhambyl branch of "Kazphosphate" Ltd. (NDPP) with the following composition, % (wt.): 36.9 SiO_2; 43.2 CaO; 5.2 Al_2O_3; 2.0 P_2O_5; 1.60 Fe_2O_3; 0.057 Ba; 2.4 MgO; 0.14 TiO_2, Sr 0.16 and so on; ∑ ∑ КУЬ – 536.48 g/t.

We report here a flow diagram of comprehensive phosphorus slag processing to obtain precipitated silica ("white soot") and RE concentrate (Figure 1).

The technology includes the following operations:

- First stage of slag leaching with the solution of nitric acid;
- REM-containing solution extraction with tributyl phosphate (TBP)
- Stripping of the REM with water;
- Sedimentation of ∑ REM salts with the solution of oxalic acid,
- Calcination of the precipitate to obtain a concentrate of ∑ REM oxides
- Second stage of leaching of the silicon-containing cake to obtain "white soot"

Regime parameters of the technology of comprehensive slag processing:

1. The first stage of leaching is carried out using 7.5 mol/dm^3 of nitric acid at temperature of – 60 °C and during 1 h under vigorous stirring with a rotating stirrer.

   To prevent an active transition of silica to a gel state, the leaching of phosphorus slag was performed under conditions that lower the decomposition rate of

Content, [% (wt.)]						pH of aqueous extract	Specific surface area, [m^2/g]
SiO_2	Al_2O_3	CaO	MgO	Fe_2O_3	Na_2O		
State standard (18307 – 78)							
87.0	0.1	0.8		0.17	1.1	8.0-9.5	120±20
”White soot”							
87.0	0.09	0.6		0.14	0.05	9.2	144
87.0	0.09	0.4		0.16	0.05	9.4	196
88.5	0.10	0.7		0.14	0.06	9.4	160

Table 1: Physicochemical parameters of ”white soot”.

calcium silicate. To meet these conditions, leaching is preceded by a preliminary preparation, i.e. the repulping of phosphorus slag that consists of mixing of the slag with water at ratio S : L = 1:1 with simultaneous heating of the pulp to a predetermined temperature. The pretreatment temperature of phosphorus slag in water was kept at 10 °C below the leaching temperature. After that the concentrated nitric acid was gradually added to the pulp under intensive stirring. In the first 0.5 h about 25 % of the total amount of nitric acid introduced, in the next 0.5 h - the rest of it. The pulp was then heated to the desired temperature and leached under stirring for 1 h. The S: L ratio was 1: 2.6 after taking into account water introduced for repulpation.

2. Extraction of the solution after leaching of slag containing 196 mg/dm^3 ∑REM, and g/dm^3: 123.93 Ca, 9.79 Al and 2.94 Fe was carried out under the following conditions: extractant, % (v/v): 50 TBP, 50 purified kerosene, the ratio of O:A = 1 : 6, the contact time of the phases is 5 min, phase separation time is 5 min, temperature is 25±5 °C, the number of stages is 3.

3. The stripping was carried out with water at ratio of O: A = 6: 1, temperature of 25±5 °C and a phase contact time of 5 min, the number of steps was 1. The concentration of ∑REM in re-extracts averaged ∼ 5.9 - 6.0 g/dm^3, the recovery from the saturated organic phase was ∼ 92 – 94 %, respectively.

4. Deposition of REM salts was carried out with 2 mol/dm^3 solution of oxalic acid, washing the precipitate with hot water, drying and calcining it at 900 °C for 2 h to obtain a concentrate of ∑ REM oxide.

5. Second stage of leaching of the silicon-containing cake obtained from the first stage, was done with 1.6 mol/dm^3 nitric acid solution, then washing of the precipitated silica to reach pH 6 -6.5, drying the precipitate at 300 °C, abrasion of the ”white soot” on the sieve 0.14.

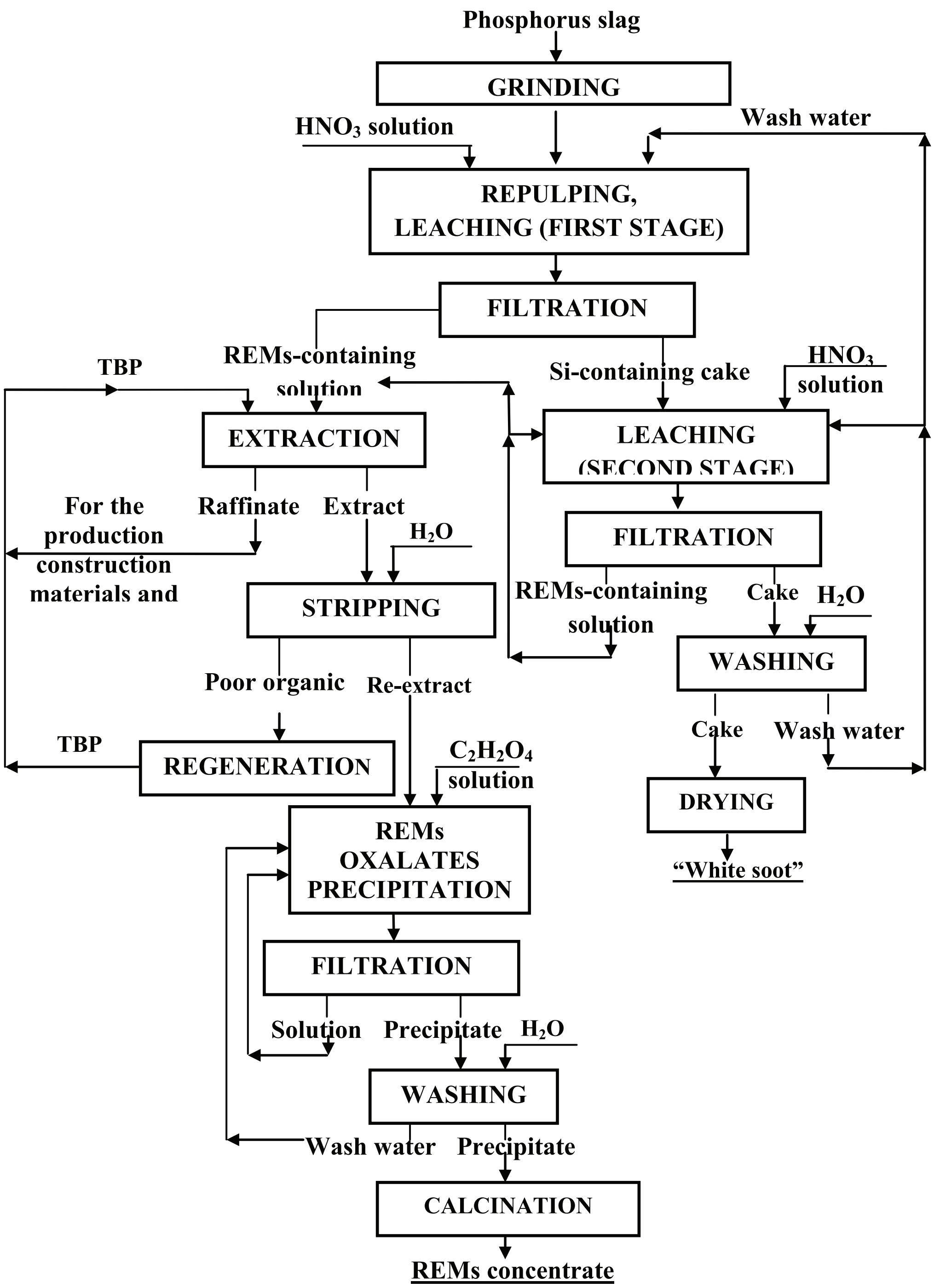

Figure 1: Technological scheme for processing phosphorus slag to obtain precipitated silicon dioxide and REM concentrate.

Recovery of ΣREM from phosphorus slags during various operations was, %: leaching – 95.0; extraction - 90.2; stripping on the average ~ 93.2; the production of ∑ REM oxide concentrate is 93.9.

Table 1 shows the composition of some batches of "white soot" obtained during the processing of the technological scheme.

The appearance and color of ”white soot” precipitates are identical to each other and meet the requirements of State standard (18307 – 78). The content of silicon dioxide in ”white soot” varies from 87.0 to 88.5 %. The specific surface area of the obtained batches of precipitated silica was in the range of 144 - 196 m^2/g. The spread of the values is due to the fact that the deposits of ”white soot” were dried in a cabinet at temperature of 300 °C. In production conditions, drying is generally carried out in spray dryers (fluidized bed furnaces) at a temperature of 750 °C. The throughout extraction rate of the silicon product and the REM oxides into the concentrate from their initial content in the slags was ~ 98.0 and 75.0 %, respectively.

3. Summary

The developed technology of comprehensive phosphorus slag processing to obtain precipitated silicon dioxide powders and rare-earth concentrate can solve the problem of complex processing of phosphorus raw materials and import substitution in Kazakhstan.

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