



Effect of calcinated and activated perlite on improving efficiency of dihydrate process for phosphoric acid



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ABSTRACT

Wet process phosphoric acid (WPA) manufacturing is mainly via the dihydrate process. Phosphogypsum is the by-product. The size distribution of phosphogypsum is a major factor for the economics of the process as it greatly affects filtration efficiency of the acid. The goal of this study is to enhance slurry filtration by adding treated perlite during the phosphate rock digestion stage. This study was carried out using a semi-continuous laboratory scale unit simulating the dihydrate process conditions. Results showed that there was an optimum dose which depended on the type of perlite and it was 86 for raw perlite, 17.2 for calcinated perlite and 8.6 g/kg P₂O₅ for the activated perlite. The addition of activated perlite ensured high filtration and washing flows and high P₂O₅ yields through reducing the P₂O₅ losses especially the unattacked form which consequently improved plant productivity. Perlite proved to be a very effective additive for the growth of gypsum crystals it altered the morphology of the gypsum particles. Additional results indicated that the activated perlite led to decreasing the amount of fluorine (18.8%), aluminum (18.9%), iron (8.2%) and soluble organic matter (16.6%). However, it involved an undesirable increase of magnesium (14.6%) and silica (16.4%) contents.

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1. Introduction

Phosphoric acid is an important intermediate product for the production of fertilizers. The manufacture of wet process phosphoric acid has often been described as the art of producing an acceptable by-product which is phosphogypsum. Both the size and the shape of the phosphogypsum crystals are critical to achieve optimum filtration rates which dictate both production rates and yields (Moudgil, 1995). It is known that the filtration rate depends on the characteristics of filter cake such as the crystal size, the size distribution and the morphology of the crystals. In other words, large spherical and narrow size distribution crystals result in a better filtration rate (Becker, 1983; Slack, 1968). Using the same filter area, the production capacity with lower operation (running) costs can be achieved if the filtration rate is increased (Abdel-Aal et al., 2006).

It has long been recognized that the size and/or shape of the phosphogypsum crystals can be modified by adding trace quantities of certain chemicals. Much time and effort have been expended to find an economical crystal modifier that would guarantee optimum filtration rates under industrial operating conditions. Improved production rates and yields would offer financial benefits to the industry and the improved yields would help reduce the potential for ground water

contamination by lowering the acid content of the pond water (Moudgil, 1995).

The most traditional procedure is the one in which a chemical agent is added after the crystals are formed to flocculate the smaller sized crystals that impede the filtration separation of the phosphoric acid from the phosphogypsum (Moudgil, 1995). The crystal modification method is more interesting in that a chemical agent is added to the phosphoric acid reactor to modify the phosphogypsum crystals as they are formed from the reaction of phosphate rock and sulfuric acid. Modifying the crystals during the formation stage has the potential to decidedly produce superior crystals that can be readily separated from the phosphoric acid at high production rates with higher yields (Moudgil, 1995).

While the flocculation method improved the crystals (Becker, 1983) the new method changing the way the crystals grow was considered superior. If this second technique can be proved to be successful enough on a widely adopted plant scale, it will make a significant environmental and economical contribution to the phosphate industry (Moudgil, 1995). Consequently, in this work we look for a new method of modifying the phosphogypsum crystals.

Slurry filtration is an important step in the production of phosphoric acid by the dihydrate process. Many studies have been conducted to ameliorate the filtration rate. Different materials were tested as crystal modifiers for the crystallization of calcium sulfate dihydrate e.g., clays (Kaolinite or bentonite) (Lin and Schorr, 1993), silica (diatomite, opal silica, porcelainite) (Lin and Schorr, 1996), aluminum sulfate, polymers (Moudgil, 1995), surfactant (Abdel-Aal et al., 2006), and other organic

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additives such as EDTA and gelatin (Franklin et al., 1982). In fact, some of them, in particular aluminosilicate, are added to phosphate rocks to modify their mineral and chemical composition to alter their behavior and overcome problems in Wet process Phosphoric Acid (WPA) production (Lin and Schorr, 1993). Indeed, gypsum crystals formed during the reaction are separated by filtration whose rate is influenced by the crystal shape and size distribution. Silica and aluminum minerals present in the aluminum–silicate adjuvant have a positive effect on the crystal morphology and the filtration rate (Becker, 1983; Theys, 2003). Depending upon its content and under which form it is found in the filtration section silica can have a positive or negative impact on the filtration. If the silica source is soluble, by reacting with the free fluorine it will impede the production of gypsum needles and promote formation of gypsum crystals, which are easy to filter (ratio length on width of the crystals close to 1, cluster) (Theys, 2003). The presence of aluminum in phosphoric acid has a positive influence on the crystal habits and size consequently influencing filterability (Becker, 1983; Chari, 1989). Aluminum is said to promote regular crystal growth in all directions by yielding much thicker crystals. This reduces the surface to the volume ratio of the crystals and hence improves the filtration rate (Gilbert, 1966; Li et al., 1997). Magnesium in phosphoric acid has a dual effect on the crystallization, initially combining with the fluorine it improves crystallization but above a certain threshold its effect in increasing the viscosity of the acid phase causes a rapid deterioration of the filtration rate (Theys, 2003).

The use of perlite as a mineral modifier and an aluminum–silicate adjuvant has improved the wet process phosphoric acid productivity (Lin and Schorr, 1996). Therefore the use of treated perlite with Tunisian phosphate rock supplied from Mdhilla in the south of Tunisia is studied in this work. Detailed laboratory studies are necessary to test perlite with known chemical formulas to promote research in this area, which would have a scientific and economical impact. Utilization of perlite would avoid any effect of co-components on gypsum crystallization. To our knowledge, limited data are reported about the testing of aluminum–silicate adjuvant for phosphoric acid production on the industrial-scale.

The present research aims to study the effect of calcinated and activated perlite introduced with phosphate rock on slurry filtration rate and on phosphoric acid productivity and quality as well as on gypsum morphology under conditions simulating the industrial dihydrate process production of phosphoric acid. The calcination of perlite is performed at the same conditions given by Stecker Glenroy (1950) whereas its activation was done by sulfuric acid according to the method described by Veera M. Boddu et al. (2009).

2. Experimental procedure

The main steps in the dihydrate process are the reaction, the crystallization, the filtration, and the washing. The crystallization of calcium sulfate dihydrate (phosphogypsum) occurs as the reaction is taking place. Indeed, it is necessary to assure sufficient time for gypsum crystal growth. In fact, the reaction time was fast (2 to 10 min) whereas the crystallization of the gypsum particles ($\text{CaSO}_4 \cdot 2\text{H}_2\text{O}$) is very long and extends from 2 to 8 h (Becker, 1983). After the reaction and the crystallization steps, the slurry is filtered in order to separate the strong acid (28–32% P_2O_5) from the phosphogypsum cake. Phosphogypsum is counter-currently washed by hot water. The main objective of the washing operation is to extract, with wash liquor, the phosphoric acid held by the capillary forces in the interstices of the gypsum cake as much as possible. Wash liquor displaces the impregnating phosphoric acid (Abdel-Aal et al., 2006). The washed phosphogypsum is collected and analyzed in order to determine the P_2O_5 yields and losses under its various forms: co-crystallized (Coc), soluble (Sol), and unattached (Unat).

In this work the activation step is performed by 2.0 M sulfuric acid (H_2SO_4). In fact, expanded perlite is suspended in sulfuric acid and

boiled for about two hours at 100–110 °C. The product is filtered, washed with distilled water until the filtrate is neutralized, and dried in an oven at 110 °C.

Adsorption capacity, particularly when using sulfuric acid treated perlite, is very high in comparison to conventional sorbents employed at similar temperatures. Modified perlite is capable of adsorbing metal in aqueous solutions. Thus, coating the surface of perlite with appropriate chemicals enhances its adsorption capacity at high temperature (Boddu et al., 2009).

2.1. Material characterization

Experiments are carried out in a laboratory scale unit at conditions simulating the industrial dihydrate process production of phosphoric acid. The temperature in the reactor is maintained at 80 °C during the phosphate rock digestion and gypsum crystal growth.

The phosphate samples used in the study are Metlaoui's phosphate rocks supplied by the Tunisian Chemical Group (TCG). Chemical analyses of these samples are given in Table 1. Whereas perlite samples which contained 77.16% SiO_2 and 14.68% Al_2O_3 are provided from Europerlita. The applied doses of treated perlite vary from 0.0 to 25.8 g/kg P_2O_5 .

Pure (98%) sulfuric acid of 1.793 g/ml specific gravity and weak phosphoric acid of 26.02% P_2O_5 content and 1.243 g/ml specific gravity are used for the digestion.

2.2. Apparatus and procedure

The laboratory scale unit for Tunisian phosphate rock processing has nine elements with regulated power: (1) a hot plate, (2) a stirring motor (Speed Range: 40–1200 rpm), (3) a propeller stirrer with two types of blades, (4) blades for breaking foam ($L = 7.8$ cm; $W = 2.4$ cm), (5) blades for agitation ($L = 2$ cm; $W = 3.1$ cm), (6) a thermometer, (7) a 5 L beaker, (8) separatory funnels for sulfuric acid supply, and (9) a stand. Also, the distance between both types of blades is equal to 8.5 cm.

The adopted system for the filtration and washing step of slurry is composed of:

- o graduated measuring cylinder of 2 L,
- o mercury board for measuring the applied pressure,
- o vacuum pump to assure a pressure of 400 mm Hg equipped with regulated valve,
- o and a removable Buchner funnels.

Detailed experimental procedure including reaction and filtration techniques are described elsewhere. In fact, the reaction is carried out in a cylindrical 5 L reactor of 17.31 cm diameter. It is fitted with a stainless steel-coated stirrer and placed in a water bath adjusted at 80 °C. The

Table 1
Chemical composition of Metlaoui's phosphate.

Chemical compound	Weight. (Wt.%)	Weight (Wt.%) P_2O_5 (Wt.%)
P_2O_5	29.03	1.00
CaO	49.3	1.69
K_2O	0.08	0.00
Na_2O	1.18	0.04
OC ^a	0.49	0.02
SiO_2	2.22	0.08
SO_3	3.22	0.11
F	3.36	0.12
Fe_2O_3	0.32	0.01
Al_2O_3	0.57	0.02
MgO	0.71	0.02

^a Organic carbon

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