



Fixation of treated phosphate waste and its use in concrete

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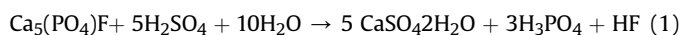
ABSTRACT

Currently, in Israel, phosphate acidic wastes formed during the production of phosphoric acid (H₃PO₄) using hydrochloric acid (HCl) are stored in large ponds. At present, this waste is only neutralized using lime (CaO) or calcite (CaCO₃). This paper investigated the potential use of Class F (<20% CaO) coal fly ash (FA) as a fixation reagent of the neutralized hazardous phosphate industry waste. Studies have shown that FA can be an efficient scrubber and fixation reagent for various kinds of acidic toxic waste. Several analytical techniques including ICP-AES, ICP-MS, SEM-EDX, and XRD, ion penetration and compression/flexure strength tests (according to ASTM C1202, IS26/4.1, and 4.2 protocols), as well as leaching experiments (EN12457-2) were used to study the fixation quality and potential utilization of the fixated product as partial substitute to sand in concrete. Scrubbing phosphate waste with the FA was found to effective in reducing the leaching rate of toxic trace elements. Moreover, the use of aggregates as a partial substitute for sand in concrete improves the mechanical and chemical properties and is environmental safe. Thus, these procedures would eliminate costly treatment and storage of these toxic wastes relatively inexpensively as well as enhance industrial concrete for civil engineering projects.

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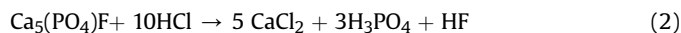
1. Introduction

The phosphate industry is essential in the production of fertilizers as well as other industries (such as medicines and food). Phosphate production requires the generation of phosphoric acid (H₃PO₄) from phosphate rock. Currently, wet acid treatments using sulfuric acid (H₂SO₄) to dissolve phosphorite and release the phosphoric acid from the calcium phosphate salts are widely used to produce the H₃PO₄. However, the process also produces very large amounts of the potentially hazardous phosphogypsum (PG) as a by-product (Equation (1)):



Furthermore, hydrochloric acid (HCl) can also be used as a wet acid treatment (as shown in Equation (2)). With this process, there

is no PG by-product, and even a higher recovery of available P₂O₅ is often achieved (98–99% as opposed to 94–95% using sulfuric acid). However, this process also produces toxic acidic solutions containing many hazardous trace elements often stored in large ponds.



In 2014, the worldwide production of phosphorite was 225 Mt (primarily in China, Morocco and the Western Sahara, and the United States (100 Mt, 30 Mt, and 27.6 Mt, respectively).

In Israel, there are 130 Mt of phosphate rock from the late Cretaceous-early Eocene Mediterranean phosphate belt (Turkey to Morocco). In 2016, the annual production was 3.5 Mt of phosphorite treated in two H₃PO₄ production sites in southern Israel: (Rotem Amfert using H₂SO₄, and Haifa Chemicals Ltd. using HCl) (GSI, 2017).

The present study focused on the phosphate waste from the Haifa Chemicals Ltd. production facility in southern Israel which uses HCl for dissolution of the phosphorite as described in Fig. 1. The present treatment of these acidic wastes in large settling ponds, using either lime (CaO) or CaCO₃, only neutralizes the wastes. This

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expensive procedure (~430\$/ton) requires large land areas and storage for the fixated wastes (ESC, 2017).

Since 1982 and for almost 33 years (1982–2015), most of the electric power (~63% in 2013) in Israel was produced by the combustion of bituminous coal (Israel-Electric-Corp.). In 2014, when large undersea reserves of natural gas were found near the coast of the Mediterranean Sea, the Israel Electric Company (IEC) began using natural gas as its main energy source for electricity. However, Israel's geopolitical status requires that at least 45% of its electric energy supply rely on coal combustion (NCSC, 2016). In 2016, ~7 Mt of bituminous coals (primarily from South Africa and Colombia) were fired in the four coal power plants (NCSC, 2016), yielding 90 kt bottom ash (BA), and 758 kt fly ash (FA). (COFA –Columbian FA ~41%, Russia -RFA- 35% and South Africa- SAFA- 24% coals (National-Coal-Ash-Board-Israel, 2013).

Due to strict environmental regulations, coals imported to Israel (marine transport) undergo beneficiation (by washing with water) to reduce S, P, and volatile toxic element stack emissions (Protection, 1992). Thus, the imported coals contain high alkaline contents and alkaline earth metals resulting in Class F FA production.

Worldwide, most FA is used in the construction industries as cement additive or for concrete production (Kesharwani et al., 2017; Lieberman et al., 2015b; Rafeizonooz et al., 2016; Zobal et al., 2016). FA is also used in agriculture (Arivazhagan et al., 2011; Basu et al., 2009; OTPC, 2010), road construction (Ishai, 2011; Kumar and Patil, 2006; Singh and Shah, 2017; Yao et al., 2015), and as a chemical scrubber (Cohen, 2003; Dermatas and Meng, 2003; Lederman, 2008; Lieberman et al., 2014a, 2015a, 2015b, 2015c). Moreover, bricks produced using the sand-like FA

waste aggregate product (AP) as partial substitute to sand indicated improved mechanical and chemical properties. Furthermore, neither the AP nor the concrete showed hazardous trace elements and both were in accordance with the European Directive (EN 12457-2) (CEN, 2003), the CAL-WET, and the new TCLP1311 tests (DTSC, 2005; EPA, 1992)

The high lime content (2–11%) of the FA, the relatively large surface area of non-porous material (0.97–1.2 m²/g), the high aluminosilicate (SiO₂ + Al₂O₃) content (>70 wt%), and pozzolanic properties make an effective scrubber for various wastes. For example, studies (Cohen, 2003; Lieberman et al., 2015c, 2016, 2014b) have reported the successful use of FA as a scrubber for the acidic wastes of regenerated of motor oil. The fixation mechanisms in these studies demonstrated, that after the neutralization, increase of exposed Al-Si anionic groups on the FA surface enables the fixation of cations on the FA surface by three types of interactions: (1) cation exchange, (2) coordination bonding, and (3) electrostatic interactions with fine precipitates. Also, fixating by-products from calcite quarries (Ca-carbonate materials) from the Palestinian Authority and quarries in Israel (Lieberman et al., 2015b, 2017), have demonstrated an adhesion of finer particles from one material to coarser particles of another - probably by electrostatic interactions forming physical barriers that prevent excessive leaching from the by-products (Lieberman et al., 2015b).

Based upon our previous experience and these promising findings, we attempted to use FA as a fixation reagent for phosphate waste and to use the scrubbed AP as a potential partial substitute for sand or aggregates in industrial concrete. Furthermore, using FA in cement or concrete appreciably reduces the CO₂ emission (1t of FA reduces 1t of CO₂) (ACAA, 2016). Thus, using the FA as a fixation reagent for phosphate waste could end the present costly process of treatment and storage of the phosphate industry wastes (~430\$/ton) and concurrently produce an environmentally safe product for civil engineering.

2. Materials and methods

2.1. Starting materials

The FA samples studied were from two pulverized bituminous coals (Colombian and South African) combusted in Israeli coal power plant facilities. The coals underwent beneficiation (washing with water) to reduce pollutants (Table S1), especially the S, Cl, and F content (0.5%, 0.005%, and 0.015%, respectively). Previous studies have determined that the compositions of the Class F FAs produced (Colombian fly ash - COFA and South African fly ash - SAFA), have a high content of aluminosilicate (>70 wt%), CaO (3.4–9.5 wt%), Fe (3–6.5%), as well as alkali metals and alkaline earth elements such as Na, Mg, and K (0.4–2.3 wt%, 1.7–2.1 wt%, and 0.8–1.6 wt %, respectively) (Lieberman et al., 2014b, 2016). As reported in previous studies, SAFA and COFA are composed of round Al-Si glassy particles. In our previous study, using XRD, quartz and mullite (3Al₂O₃-2SiO₂) were detected in the main mineral phases (Lieberman et al., 2015b). Moreover, in SAFA phases of calcite and hematite (Fe₂O₃) were also detected and in COFA, magnetite (Fe₃O₄), hematite, and anhydrite (CaSO₄) were also detected. These results are consistent with the average chemical composition of the FAs (Table 1). Furthermore, for this study, a ~35 kg sample of FA was collected from the electrostatic precipitators and transferred to sealed polyethylene containers until further analysis. Morphology and surface characterization (SEM-EDX) showed that the FA primarily consisted of small glassy particles either cenospheres (hollow particles) or pleurospheres. Other particle types, including spinel and unburnt carbon, were also present in relatively minor proportions.

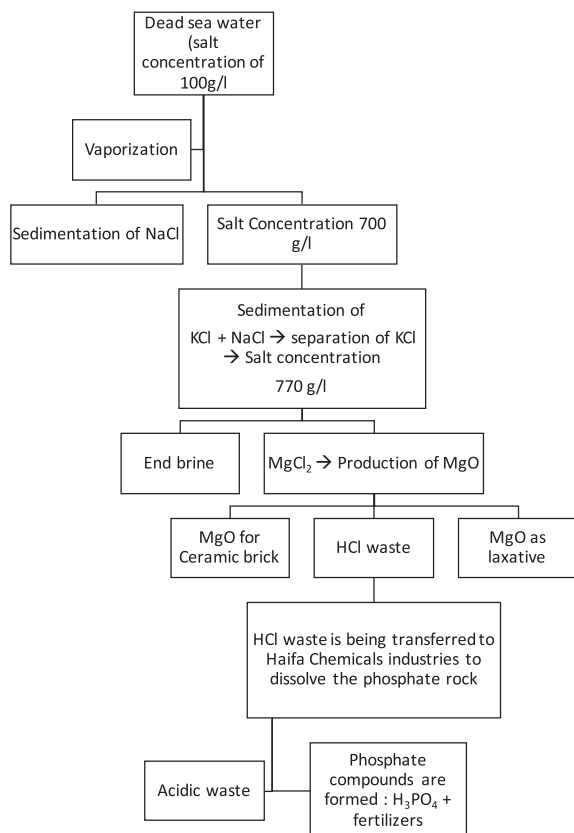


Fig. 1. Process scheme of the formation of phosphate compounds and phosphate acidic waste in Israel.

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