



Effects of fibers on mechanical properties and freeze-thaw resistance of phosphogypsum-slag based cementitious materials



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HIGHLIGHTS

- After 15 cycles of F-T cycling, PGS with fibers had a weight loss less than those without fibers.
- The losses in flexural strength and anti-impact work of PGS are due to water softening reduced.
- Major hydration products in PGS are C-S-H and Aft. They may be a major cause during PGS expansion.
- At optimal fiber content, performance of PGS with PP fiber is better than mineral and glass fibers.

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ABSTRACT

Cementitious materials made primarily with phosphogypsum (PG) and ground-granulated blast-furnace slag (GGBFS, or slag) are studied. Mineral, glass and polypropylene (PP) fibers with various dosages are incorporated into this PG-slag (PGS) system. The anti-impact work, flexural strength, and freeze-thaw (F-T) resistance of these fiber-reinforced PGS mixes are evaluated, and the microstructures of these fiber-reinforced PGS mixes are also examined. The results show that addition of any of these fibers can significantly increase the flexural strength, anti-impact work, water resistance, and freezing-thawing resistance of PGS. Among the three fibers studied, the PP fiber is the easiest to disperse, and it also has a good bond with the PGS matrix. Addition of 1.35% (by volume) of the PP fiber has increased the flexural strength of the PGS twice and increased the anti-impact work up to 7 times. At the optimal fiber content, the PP fiber provides a better overall performance than mineral and glass fibers.

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1. Introduction

During a phosphate fertilizer manufacturing process, rock phosphate is either heated in an electronic furnace (the dry process) or treated with a sulfuric acid (the wet process) to produce a phosphoric acid, the latter of which is more economical but produces a by-product, phosphogypsum (PG). PG contains mainly $\text{CaSO}_4 \cdot 2\text{H}_2\text{O}$ as well as some adverse ingredients, such as P_2O_5 , F^- , organic substances, and alkali metals.

The discharge quantity of PG is large: about 5 tons of PG is produced for each ton phosphate acid. The annual production of phosphogypsum has been in the range of 40–47 million metric tons per year since the mid-eighties and it is 50 million tons at present in China, but only 10% of which is utilized. The deposit of PG and its environmental effect have become an urgent concern [1–5].

In addition to being used for agriculture [6] and soil stabilization [7], PG can also be utilized for construction materials. Akin et al. used PG as a concrete retarder [8]. Lima, Zhou and Shen et al. produced SO_2 and CaO from PG decomposition, and then further made H_2SO_4 and anhydrite cement [9–11]. Hu and Shao et al. generated the calcined gypsum from PG [12,13]. Zhang, Camarini, and Colak made plasterboard together with fiber, water-proofing additive and phenolic resin [14–20]. Many others made construction materials using PG, such as bricks [21,22], roadbed materials [23,24], and wall materials [25,26], using PG together with slag, fly ash and/or lime.

PG has similar proprieties to natural gypsum, including rapid setting, fire resistance, thermal insulation and acoustic properties, but its strength, water resistance and cracking resistance are generally low. Xia et al. successfully developed a PG, slag and bauxite mix, which had compressive strength of 10 MPa and water absorption of 9.7% [27]. Deðirmenci et al. evaluated the compressive strength, flexural strength and water resistance properties of a PG-slag (PGS) system, and they found that the specimens cured

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in water showed noticeably lower strength than those cured in air, and water absorption increased with increasing phosphogypsum content [28]. Research has indicated that PGS often rupture easily in the influence of cold and humid climate. Therefore, use of PG products in the field of construction material is limited.

Fibers have been widely used to improve the performance of cement-based cementitious material so as to improve concrete toughening, reinforcing and anti-cracking [29–32]. However, very little study has been reported in the literature on the use of fibers in PGS. The primary objective of the present study is to evaluate effects of fibers on crack and freezing-thawing (F-T) resistance of PGS.

2. Experimental

2.1. Materials

The materials used in the present study include PG, ground-granulated blast-furnace slag (GGBFS, or slag), lime, activators, retarder and fibers. Table 1 shows the main physical property of PG and slag. Table 2 gives the main chemical compositions of PG and slag as determined by X-ray fluorescence (XRF). The lime used had CaO content >90%. The alkali activator was prepared using water glass (modulus of 3.0) and sodium hydroxide (concentration of 36%), and the ratio between water glass and sodium hydroxide is 10:1. The retarder was a citrate. Table 3 lists the properties of three fibers used, and Fig. 1 shows the photos of these three fibers.

Fig. 2 displays the micromorphology of the phosphogypsum and slag, observed under a scanning electron microscope (SEM). The PG was ground to pass 125 μm sieve and then calcined at 140 $^{\circ}\text{C}$ for 4 h (h) to obtain hemihydrate calcium sulfate. Fig. 3 displays the X-ray diffraction (XRD) pattern of the PG, hemihydrate calcium sulfate, and slag. Fig. 4 presents the particle size distributing of PG and slag.

2.2. Material proportion

PGS used in the present study was a blended powder material with a proportion of calcined phosphogypsum:slag:lime = 60:40:4 (by weight). PGS pastes were made with a water-to-powder ratio (w/p) of 0.6, 0.2 % of retarder, and 3 % of alkali activator (by weight).

To determine the optimal maximum fiber content for the designed PGS mixtures, a trial test was conducted. In the test, a given fiber was added into a given amount of mixing water in a beaker incrementally, and it was stirred by hand. At a small dosage ($\leq 1.0\%$ by volume of the PGS mixture), all fibers were generally well dispersed, while at a high dosage ($>1.0\%$), not all fibers could be well dispersed. Fig. 5 shows that at a given fiber dosage, PP fiber was dispersed very well while mineral and glass fibers were not. The maximum fiber content used in the present study was determined based on the dispersion of the fiber observed during the test.

According to the test results, the fiber contents selected for the present study were 0.00, 0.35, 0.70, and 1.00% (by volume) for the PGS mixes with mineral and glass fibers, while it was 0.00, 1.00, 1.35 and 1.70% (by volume) for the mixes with PP fiber. As discussed later (Section 3.1.3), the flexural strength and anti-impacting work of the PGS mixes decreases when the fiber content exceeds their optimal content for dispersion. Table 4 lists the proportions of PGS mixes studied.

2.3. Sample preparation

All powder materials were firstly blended in a plastic bag and then mixed with fibers by hands. These dry materials were then mixed with water and admixtures (activators and retarder) using a standard Hobart mixer.

For each PGS mix, two sets of paste samples were cast:

- (1) Twelve 40 mm \times 40 mm \times 160 mm samples, three used for flexural strength tests before F-T at 7 days and three at 28 days; three used for flexural strength tests after F-T; and three for water softening tests.
- (2) Fifteen 10 mm \times 10 mm \times 60 mm samples, three for anti-impacting work tests before F-T at 7 days and three at 28 days; three for weight loss after F-T; three anti-impacting work tests after F-T; three for water softening tests.

Table 1
Physical properties of raw materials used (%).

Raw material	Appearance	Color	Specific gravity/g/cm ³	Specific area m ² /kg
PG	Power	Grey	2.35	350
GGBFS/slag	Power	Grey	2.92	410

Table 2
Chemical composition of raw materials used (%).

Raw material	CaO	SO ₃	SiO ₂	Al ₂ O ₃	P ₂ O ₅	K ₂ O	TiO ₂	MgO
PG	31.05	30.95	4.86	4.38	3.57	0.41	0.2	0.26
Slag	33.38	–	38.90	20.90	–	0.91	1.15	2.63

Note: The loss of ignition of PG is 22.91%.

Right after casting, all samples were dry-cured under a temperature of 20 \pm 3 $^{\circ}\text{C}$ and the relative humidity of 50% according GB 175-2007 and GB 1943-2007 (Chinese standards) until their testing.

After 7 days of dry-curing, three 40 mm \times 40 mm \times 160 mm samples and six 10 mm \times 10 mm \times 60 mm samples of each PGS mix were taken out and placed under the F-T condition as described in Chinese standard GB 50574-2010. Each F-T cycle is comprised of cooling the samples in air for 2 h at $-20^{\circ}\text{C} \pm 2^{\circ}\text{C}$ followed by immersing the samples in water for 3 h at $20^{\circ}\text{C} \pm 2^{\circ}\text{C}$. After 15 cycles of F-T, the 40 mm \times 40 mm \times 160 mm samples from each of the PGS mixes were used for flexural strength tests, and the 10 mm \times 10 mm \times 60 mm samples from each of the PGS mixes were used for weight loss and anti-impacting work tests.

After 28 days of dry-curing, three 40 mm \times 40 mm \times 160 mm samples and three 10 mm \times 10 mm \times 60 mm samples from each PGS mix were submerged in water for 24 h, and then they were tested for water softening coefficients of flexural strength and anti-impacting work, respectively.

In addition, four PGS mixes (PGS00, PGS1.0G, PGS1.0M and PGS1.35P) were selected for length change measurements. Three 10 mm \times 10 mm \times 60 mm samples, with an embedded nail at the ends, were cast for each of these four mixes. The samples were demolded at 2 h after casting, and the initial lengths of the samples were measured right after the demolding. The samples were then placed in the dry curing condition as described above (20 \pm 3 $^{\circ}\text{C}$ and 50% RH), and the length change of the samples were measured periodically up to 60 days. Table 5 shows the list of tests performed.

2.4. Test methods

Flexural strength of PGS mixes was tested using an omnipotence electronic pressure gauge according to the GB 175-2007 (Chinese standard), and the loading rate is 50 N/s.

Anti-impacting work tests were conducted using impact testing machine (XJJ-5) according to the GB 1943-2007. In the test, as shown in Fig. 6, a sample was placed in the pedestal with a gap. The hammer energy is 1 J and it was pre-set to 160 $^{\circ}$. The distance of hammer axle center and sample center is 221 mm. As the test started, the hammer fell at an impacting speed of 2.9 m/s. The impact load was recorded by the dial as the sample failed, and the breaking area of the tested sample was measured. The anti-impacting work (A_K) of the tested sample was then calculated as follows:

$$A_K = W/F \quad (1)$$

where A_K is anti-impacting work (N S/m²), W is the impact load after sample break (N S) and read from the dial of impact testing machine, F —Sectional area of sample breaking.

The water softening coefficients of PGS were tested according to the Chinese standard JC/T 698-2010. It is expressed as a ratio of the flexural strength or anti-impacting work of dry-cured samples – to – the flexural strength or anti-impacting work of water-cured samples (see Eqs. (2a) and (2b)). The higher the ratio, the higher the water softening resistance of the sample is.

$$S_{fl} = (\text{flexural strength})_{\text{dry-cured sample}} / (\text{flexural strength})_{\text{water-cured sample}} \quad (2a)$$

$$S_{AIW} = (\text{anti-impacting work})_{\text{dry-cured sample}} / (\text{anti-impacting work})_{\text{water-cured sample}} \quad (2b)$$

The linear expansion of samples made with various PGS mixes was measured according to the method described by Yao. Using a Helix device (Fig. 7), the initial length (l_0) of the samples was measured right after the samples were taken out of the molds, then the samples were cured under a condition with a temperature of 20 $^{\circ}\text{C}$ and relative humidity ≥ 90 . The lengths (l_n) of the samples were measured periodically up to 60 days. The linear expansion ratio (K) of the samples was calculated according to the following equation [33]:

$$K = [(l_n - l_0)/55] \times 100\% \quad (3)$$

The fracture surfaces of selected samples were also examined using scanning electron microscope (SEM). Pore size distribution of the samples before and after being subjected to F-T is tested using Poremaster 60GT automated mercury porosimeters.

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