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Properties and microstructure of reactive powder concrete having a high content of phosphorous slag powder and silica fume

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HIGHLIGHTS

- Phosphorous slag powder was utilized to produce reactive powder concrete (RPC).
- Mechanical strength, freeze–thaw and sulfate resistance of RPCs are investigated.
- The content of phosphorous slag could be as high as 35% (by the weight of binder).
- RPCs prepared herein have excellent mechanical and durability properties.
- The diameter of the most probable pore in RPC samples is less than 10 nm.

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ABSTRACT

Reactive powder concrete (RPC) specimens whose content of phosphorous slag powder (PS) and silica fume was about 50% (by the weight of binder) were produced after they had been cured in 95 °C steam for a given duration. The test results of strength (compressive and flexural), freeze–thaw and sulfate resistance verified the excellent mechanical and durability properties of RPC containing a high content of PS. The investigation of selected RPC compositions by Thermogravimetric Analysis, Mercury Intrusion Porosimetry and Scanning Electronic Microscope made it possible to better understand their mechanical and durability properties depending on their microstructure. Thermogravimetric Analysis and Scanning Electronic Microscope demonstrated the sequential hydration effect of cementitious composites during heat treatment. Mercury porosimetry results showed that RPC had very low porosity and the diameter of the most probable pore was less than 10 nm. These microstructural characteristics would enable RPC to have excellent mechanical and durability properties.

1. Introduction

Reactive powder concrete (RPC) is developed through microstructural enhancement techniques and is characterized by super-high strength, extreme durability and superior toughness [1,2]. The mechanical properties that can be achieved include the compressive strength of the range between 200 MPa and 800 MPa, the flexural strength of the range between 30 MPa and 60 MPa, fracture energy of the range between 1200 J/m^2 and 40,000 J/m^2, Young's modulus of the range between 50 GPa and 60 GPa, and ultimate tensile strain at the order of 1% [1–3]. RPC with superior performance has been applied extensively in civil, petroleum, nuclear power, municipal, marine and military facilities, as well as in other projects [4,5]. However, cement dosage of conventional RPC is generally high and silica fume (SF) content is often over 25% (by the weight of cement), which not only increases the production costs, but also has negative effects on the hydration heat and may cause shrinkage problems. Replacing cement with mineral admixtures and decreasing SF content seemed to be a feasible solution to these problems [6–12].

Electric furnace phosphorus slag which is different from granulated blast furnace slag (GBFS) is a kind of industrial waste and mainly contains SiO_2, CaO and Al_2O_3. Its total content of SiO_2 and CaO is more than 85% (by weight) and the SiO_2/CaO ratio ranges from 0.8 to 1.4. Ground granulated electric furnace phosphorous slag has a glassy microstructure which is similar to that of granulated blast furnace slag and the weight percentage of the glassy structure could be as high as 90% [13,14]. Therefore, ground

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granulated electric furnace phosphorous slag may be used as reactive composition of RPC.

Previous findings demonstrated that reactive powder concrete containing high volume binary blends (SF + PS) had no significant mechanical performance loss and incorporation of phosphorous slag powder (PS) in RPC was feasible [15]. This paper aims to achieve the following objectives:

to obtain RPCs with strength grade of C200 by utilizing phosphorous slag powder;
to examine the freeze–thaw and sulfate resistance of RPCs containing high content of phosphorous slag powder; and
to reveal the relationship between the performance and microstructure of RPCs containing phosphorous slag powder through Thermogravimetry, Mercury porosimetry and Scanning Electronic Microscope.

2. Experimental programs

2.1. Materials

The RPCs considered here was prepared by the following ingredients. Cement: ordinary Portland cement P.O 52.5, which complies with Chinese Standard GB 175-2007, from Huaxin (Yidu, Hubei Province, China) Cement Co. Ltd. Phosphorous slag (PS): granulated electric furnace phosphorous slag produced by Yichang Yatai Chemical Co., Ltd. (Hubei Province, China). Silica fume (SF): undensified silica fume with average size of 0.1 μm–0.2 μm provided by China Construction Ready Mixed Concrete Co. Ltd. Superplasticizer: a polycarboxylate based superplasticizer provided by Jiangxi Building Materials Scientific Research and Design Institute. Fine aggregate: quartz sand with size of 0.16 mm–0.63 mm was used as fine aggregate. Furthermore, brass-coated steel fiber with diameter of 0.2 mm was used to improve the ductility of concrete. The tensile strength and aspect ratio (length-to-diameter ratio) of steel fiber is 2800 MPa and 65 respectively. The pertinent chemical and physical properties of the cement, PS and SF used in this study are given in Table 1.

2.2. Mix proportions of RPC and samples preparation

Table 2 shows the mix proportions of RPC produced in this study. For each mixture, all components (cement, PS, SF and quartz sand) were mixed, cast, and vibrated in a similar sequence as conventional concrete. Initially dry powders (cement, PS and SF) and quartz sand were mixed for about 3 min. The water and superplasticizer were then added and mixed for about 6 min. Subsequently, steel fibers was added (if necessary) and mixed for another 3 min. The entire mixing process took about 12 min before the concrete mixture was ready to cast.

When RPC mixture was ready, it was poured into the required molds which had been sprayed with mold oil to reduce the friction at the interface between the molds and RPC mixture. The RPC mixture was compacted using a vibrating table and hand tamping. The cast molds were covered by plastic sheets before demolded to prevent moisture in the concrete from evaporation. These specimens were demolded at least 48 h after casting because of the high PS content which required longer setting time.

Table 1
Chemical composition and physical properties of cement, phosphorous slag powder and silica fume.

Items		Cement	Phosphorous slag (PS)	Silica fume (SF)
Chemical composition (%)	SiO_2	21.05	38.84	96.90
	Al_2O_3	5.11	3.46	0.08
	Fe_2O_3	2.90	1.40	0.03
	CaO	61.46	46.09	0.12
	MgO	1.34	1.83	0.08
	SO_3	3.64	1.34	0.50
	P_2O_5	0.18	2.45	0.02
	Loss in ignition	2.36	0.24	1.95
Specific surface (m^2/kg)		379 (Blaine)	423 (Blaine)	20,000 (Nitrogen Ab.)
Compressive strength (MPa)	3 days	36.7	/	/
	28 days	53.5	/	/

Table 2
Mix proportions of RPCs (by weight ratio).

No.	Binders			W/B[a]	Quartz sand	Superplasticizer	Steel fibres[b] (%)
	PS	Cement	SF				
PS30-0	0.30	0.55	0.15	0.16	1.0	0.02	0
PS30-1	0.30	0.55	0.15	0.16	1.0	0.02	1
PS35-0	0.35	0.50	0.15	0.16	1.0	0.02	0
PS35-1	0.35	0.50	0.15	0.16	1.0	0.02	1
RPC-0	0.35	0.50	0.15	0.16	0	0.02	0

[a] Including water from superplasticizer.
[b] Volume percentage.

2.3. Test methods

For each RPC mixture, i.e., mixture PS30-0, PS30-1, PS35-0 and PS35-1, prismatic specimens (40 mm × 40 mm × 160 mm, 100 mm × 100 mm × 400 mm) and cubic specimens (100 mm × 100 mm × 100 mm) were cast to determine the strength (compressive and flexural), freeze–thaw resistance and sulfate attack resistance respectively. After demolded, these specimens were moved in a ZKY-400B Steam Curing Container for Concrete to be cured at 95 °C for 3 days. Then, they were placed in a water tank at 20 °C until the age of 7 days. The strength (compressive and flexural) was then tested according to Chinese Standard GB/T17671-1999 while sulfate attack resistance and freeze–thaw resistance were performed according to GB/T 50082-2009 (see Fig. 1).

Mixture RPC-0 shown in Table 2 was selected for Thermogravimetric Analysis. Thermogravimetry was measured on a NETZSCH STA 449 C thermogravimetric analyzer under dry air atmosphere. A temperature range between 20 °C and 1000 °C with a 10 °C per minute heating rate was selected. Samples were prepared by taking small pieces from cubic specimens (40 mm × 40 mm × 40 mm) which had been cured at 95 °C for a given duration (2 days, 3 days and 4 days respectively) and then all specimens were cured in water at 20 °C till 7-day age. Finally, they were ground to a fine powder with the particle size about 10 μm.

For Mercury Intrusion Porosimetry and Scanning Electronic Microscope analysis, cubic specimens (40 mm × 40 mm × 40 mm) were cast according to mixture PS35-0. After demolded, these specimens were initially exposed to steam curing at 95 °C for a given duration (2 days, 3 days and 4 days respectively) and then were cured in 20 °C water till 7-day age. Samples were splinters taken from these specimens and were oven-dried at 60 °C for 24 h. Porosimetric measurements were carried out on a POREMASTER 33G porosimeter, and 3 nm to 400 μm pore sizes were investigated by this technique. Microstructure of the RPC was investigated by using a ULTRA PLUS Scanning Electronic Microscope.

3. Results and discussions

3.1. Strength

RPC specimens containing a high content of PS and SF were obtained according to Table 2. The compressive and flexural strength results of these specimens are summarized in Table 3.

From Table 3, it is noted that the flexural and compressive strength of these RPCs are about 21 MPa and 150 MPa respectively. The addition of 1% (by volume) steel fiber gave 187 MPa or more compressive strength of RPC. The result implied that the hydration activity of phosphorous slag powder (PS) was intensified. On one hand, owing to the small particle of phosphorous slag, a lot of small phosphorous slag particles filled the interspaces among cement particles, which would increase the packing density of cementitious composites and then improved the strength of hardened paste. This is the so-called packing effect [16]. On the other, a small particle size meant many lattice distortions and chemical bonds breakages existing in the surface of phosphorous slag particles [17,18]. During the process of heat treatment (curing at 95 °C for 3 days), both the quantity and speed of ionic species, such as Ca^{2+}, $[SiO_4]^{4-}$ and $[AlO_4]^{5-}$ diffusing from cementitious composites particles increased greatly. The hydration reaction of reactive mineral admixtures, such as SF and PS, was activated and occurs sequentially [7], which continuously consumed portlandite $Ca(OH)_2$ produced by cement hydration. As a result, many $Ca(OH)_2$ were transformed into another type of hydration product (Calcium