



# Influence of curing conditions on the mechanical and physical properties of chemically-activated phosphorous slag cement



Ali Allahverdi<sup>a,b,\*</sup>, Shima Pilehvar<sup>a</sup>, Mostafa Mahinroosta<sup>a</sup>

<sup>a</sup> Cement Research Center, School of Chemical Engineering, Iran University of Science and Technology, Narmak, 1684613114 Tehran, Iran

<sup>b</sup> Research Laboratory of Inorganic Chemical Process Technologies, School of Chemical Engineering, Iran University of Science and Technology, Narmak, 1684613114 Tehran, Iran

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## ABSTRACT

In this paper, the influence of curing time and temperature on the compressive strength development of chemically-activated high phosphorous slag content cement composed of phosphorous slag (80 wt.%), Portland cement (14 wt.%), and a compound chemical activator consisting of sodium sulphate and anhydrite (6 wt.%) is investigated. For this purpose, mortar specimens were prepared and cured in lime-saturated water at different temperatures of 25, 45, 65, 85, and 100 °C in an oven and at temperatures of 165 and 200 °C in autoclave. Results showed that the ultimate compressive strength decreases at curing temperatures higher than 45 °C. Investigations by XRD and FTIR show that curing at temperatures higher than 45 °C results in the formation of  $\alpha$ -C<sub>2</sub>SH.

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## 1. Introduction

Recently, the trend in using supplementary cementing materials has gained more attention [1,2]. Industrial wastes such as granulated blast furnace slag (GBFS), phosphorous slag (PHS), and fly ash (FA) have been applied to produce high strength and good performance blended cements [1,3]. In addition, blended cements can also be used to achieve economic and ecological benefits in the form of energy-saving and resource conservation [3,4].

Moreover, if part of the Portland cement (PC) can be replaced by industrial wastes, the amount of clinker needed and consequently the amount of CO<sub>2</sub> released into the atmosphere can be reduced [5]. Therefore, the important method of replacing clinker by suitable industrial wastes, especially unused wastes (such as slags) for producing blended cements of low clinker content has gained more interest [6].

PHS is an industrial by-product of yellow phosphorus production via electric furnace method. It has been reported that about seven tons of slag is generated for every ton of phosphorus production [7]. To get a sense of the amount of PHS to be used as a cementing material, here are some examples. More than 8 million tons of PHS is produced annually in China [8]. The amount of PHS production in the United States and India is about 3.6 and 0.75 million metric tons each year, respectively [9,10].

PHS is similar to the blast furnace slag (BFS) and is mainly composed of calcium oxide and silicon dioxide but less reactive than BFS [6,11]. Since the residual phosphorus of PHS in the form of P<sub>2</sub>O<sub>5</sub> has a strong retarding effect on setting time of PC and also, insufficient content of Al<sub>2</sub>O<sub>3</sub> affects early strength properties of PC, it has not been widely used at high replacement level [11]. For these reasons and also in order to enhance the amount of PHS, it must be activated to properly react and to provide the desirable early-age mechanical properties. Some of the most common activation techniques that have been applied included thermal, chemical and mechanical activations [12–14]. Chemical activation refers to using some chemicals to impart and/or activate cementing properties [13,15].

Today, attentions to do research about strength of the slag cements from academic and industrial environments have been significantly increased [16]. The development of strength in the slag-based blended cements is affected by many factors such as cement composition, fineness, water-to-cement ratio, aggregate, age and temperature of curing, and some other ones [17]. The rate and the degree of hydration, and the resulting strength and the other properties, depend on the curing process [18,19]. The objective of curing is to provide appropriate temperature and humidity conditions to ensure the progress of hydration reactions causing the filling and segmentation of capillary voids by hydration products [20]. Several researches have shown that high temperature improves strength at early ages. However, at later ages, a majority of the formed hydrates are not suitably arranged and this means a uniform structure cannot be created causing a loss of ultimate strength [21–24]. Hence, the impact of temperature and aging on the mechanical properties should be studied and quantified comprehensively [12]. Table 1 presents a historical background about some important events

\* Corresponding author at: Cement Research Center, School of Chemical Engineering, Iran University of Science and Technology, Narmak, 1684613114 Tehran, Iran.

E-mail addresses: [ali.allahverdi@iust.ac.ir](mailto:ali.allahverdi@iust.ac.ir) (A. Allahverdi), [Pilehvar.shima@gmail.com](mailto:Pilehvar.shima@gmail.com) (S. Pilehvar), [mahinroosta2010@gmail.com](mailto:mahinroosta2010@gmail.com) (M. Mahinroosta).

**Table 1**  
Some important events in the field of the use of PHS as a cementing material.

Author (s)	Year	Significance	Replacement level (wt.%)	Reference
Shi and Li	1989	Alkali-PHS cement	Not important	[25]
Singh and Bhattacharjee	1996	Manufacture of cements with phosphorous furnace slag	2.5	[7]
Dongxu et al.	2000	Effect of admixtures on PHS cement	30–40	[11]
Dongxu et al.	2001	Effect of fast-setting and early strength agent on PHS content cement	30–70	[26]
Dongxu et al.	2002	Cement containing BFS and PHS	25–70	[27]
Peiwei et al.	2008	Microstructure of concrete containing super fine PHS	40	[28]
Xia et al.	2009	Anti-crack performance of PHS concrete	20–60	[6]
Xia et al.	2011	Hydration of cement containing PHS	35	[29]
Xu et al.	2012	Effect of PHS on the performance of Portland cement	10, 30, and 50	[30]
Xiu-wei et al.	2013	Use of PHS and fly ash	10	[8]
Peng et al.	2013	Ultra-high performance concrete containing PHS	35	[31]
Jinhui et al.	2015	Effect of cement fineness on strengths of cement containing PHS	40	[32]

in the field of the use of PHS as a cementing material. Through doing a literature survey, we found out that despite the existence of some studies in the field of the use of GBFS at high replacement level, there is yet a lack of investigation about the use of PHS with high amount and also about the effects of curing conditions on properties of PHS content cements and this is an initial spark for our study. Our previous work [33] studied the effect of mechanical activation on the properties of chemically activated high PHS content cement (CAPHSC) such as sieve residue, water-to-cement ratios for paste and mortar, initial and final setting times, compressive strength, specific gravity, water absorption, and open pore volume. The new aspect of the present work is to investigate the effect of curing conditions including hydrothermal and autoclave curings on the most important physical and mechanical properties of CAPHSC. This paper aims at studying the mechanical and physical properties of CAPHSC under different curing temperatures and ages.

## 2. Experimental procedure

### 2.1. Materials

The properties of the materials utilized in this study are described as follows:

#### 2.1.1. Phosphorous slag

PHS was provided from a phosphoric acid plant located in Tehran province, Iran. The chemical composition of the PHS (in wt.%) determined according to ASTM standard C311 is given in Table 2. The density of ground PHS, 2940 kg/m<sup>3</sup>, was determined according to ASTM standard C188 using a Le Chatelier flask. Fig. 1 depicts the X-ray diffraction pattern of PHS demonstrating the presence of periclase (MgO) as the only crystalline phase.

#### 2.1.2. Portland cement

The chemical composition of Type II PC (in wt.%) and its Bogue's potential phase composition (in wt.%) are presented in Table 3. The density and Blaine fineness of this cement were 3120 kg/m<sup>3</sup> and 302 m<sup>2</sup>/kg, respectively.

#### 2.1.3. Compound activator

Two types of chemical activation including activation with strong alkaline activators and activation with mild alkaline activators are usually used. The first type is suitable for imparting cementing properties to materials with no cementing properties such as aluminosilicate and also activating industrial slags with latent hydraulic properties. The second type is proper and applicable only for materials some cementing

properties such as various kinds of industrial slags [34,35]. In this work, a compound chemical activator of the type mild alkaline was utilized in accordance with recent findings [36,37]. It is composed of Na<sub>2</sub>SO<sub>4</sub> (2 wt.%) and anhydrite (4 wt.%). The chemical composition of anhydrite (in wt.%) was as follows: CaO–36.00, SO<sub>3</sub>–54.38, and SiO<sub>2</sub>–5.88. Sodium sulphate of analytical grade was purchased from Merck (Darmstadt, Germany).

#### 2.1.4. Water

The potable water from pipeline of the laboratory was used for all mixes. The specific gravity of the used water was assumed about 1000 kg/m<sup>3</sup>.

## 2.2. Methods

### 2.2.1. Grinding

The machine used for the grinding of the materials was a laboratory ball mill with the length and diameter of 0.30 and 0.26 m, respectively and it had one cylinder box containing spherical steel balls. The proportioned mixes of PHS (80 wt.%), PC (14 wt.%) and compound chemical activator (6 wt.%) were inter-ground for a period of 7 h until they achieved the desired Blaine fineness of 303 m<sup>2</sup>/kg.

### 2.2.2. Blaine fineness

The value of Blaine specific surface area (Blaine fineness) was determined according to ASTM standard C204 using Blaine air-permeability apparatus.

### 2.2.3. Water-to-cement ratio

To achieve standard normal consistency in fresh paste and mortar, water-to-cement ratios were determined according to ASTM standard C230 using flow table test. For these tests, PC paste and mortar of

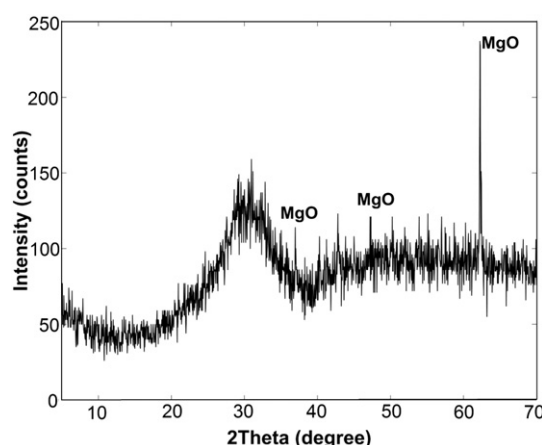


Fig. 1. X-ray diffraction pattern of phosphorous slag used in this study.

**Table 2**  
Chemical composition of phosphorous slag (wt.%).

SiO <sub>2</sub>	Al <sub>2</sub> O <sub>3</sub>	Fe <sub>2</sub> O <sub>3</sub>	CaO	MgO	P <sub>2</sub> O <sub>5</sub>	K <sub>2</sub> O	Na <sub>2</sub> O	SO <sub>3</sub>	Cl	LOI
38.42	7.65	0.90	45.14	2.60	1.50	0.56	0.43	Trace	Trace	1.87

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