Cement and Concrete Research xxx (xxxx) xxx–xxx

Contents lists available at ScienceDirect

Cement and Concrete Research

journal homepage: www.elsevier.com/locate/cemconres

Rheology of activated phosphorus slag with lime and alkaline salts

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ARTICLE INFO

Keywords:
Activated phosphorus slag
Rheology
Workability
Alkaline salt
Lime

ABSTRACT

The present study aimed to determine the rheological behavior of activated phosphorus slag (AAPS) with a combination of $Ca(OH)_2$ and Na_2CO_3 (CNC activator) or Na_2SO_4 (CNS activator) compared to OPC paste. The findings showed that the rheology of AAPS pastes fit with the Herschel-Bulkley model and behaved like a shear-thinning fluid. The AAPS pastes rheology is affected by concentration and nature of activator. The shear stress in AAPS pastes was lower than OPC paste, and increased with increasing the activator concentration. In early ages, reaction products of PS/CNC pastes included calcite-type compounds with the formed gel while gypsum compounds formed in the PS/CNS pastes. This resulted in higher shear stress and lower workability for PS/CNC pastes compared to PS/CNS pastes. The formed gel due to interaction between Ca^{2+} ions of CNC activator and Si^{4+} ions of Phosphorus slag caused an increase in the shear stress for PS/CNC during the time.

1. Introduction

Alkali-activated cements are three dimensional structured inorganic binders, synthesized by reaction of an aluminosilicate precursor with a concentrated alkaline solution (usually hydroxide, metal silicate, weak acid salts, or strong acid salts). Some specific properties such as high resistance to acid, salt, and sulfate environment and ecological benefits (reduction of CO_2 gas emission), put them into a competitive position against commercial construction materials as Portland cement [1,2]. However, further application of alkali-activated cements as grout or concrete also depends on the rheological properties of fresh mixture in the engineering process such as pumping, injection, spreading, molding, and compaction. From the rheological standpoint, cement pastes have a complex rheology, and behave like a non-Newtonian fluid that most researchers agree to be well-enough fitted on Bingham model [3–7].

In the literature, very few investigations on alkali-activated cement rheology can be detected. Primary researches on the rheological behavior of alkali-activated cements were conducted on alkali-activated fly ash and sodium carbonate-activated slag pastes [8,9]. Rheological behavior of alkali-activated fly ash showed Bingham type behavior same as Portland cement behavior rheology [9]. The effect of fly ash source on activation energy of alkali-activated fly ash pastes is determined, while activator concentration did not effect on Arrhenius pre-exponential factor and activation energy [9].

Palacios et al. studied the rheological properties of alkali-activated slag pastes with sodium hydroxide (4 wt% Na_2O) and water-glass [10,11]. They reported that nature of the alkali activator affected the AAS pastes rheological behavior. Their results indicated that in AAS pastes and mortars activated with NaOH, the structural breakdown behavior in the down ramp of the flow curve fitted to the Bingham model, while water glass-activated pastes and mortars, and also alkali-activated slag behaved like Herschel-Bulkley model. They also identified the effect of mixing time on mechanical strength and dry shrinkage in these pastes. Longer mixing time cause to reduce of dry shrinkage (approximately 11%) due to decrease in the percentage of pores smaller than 0.05 μm in binder microstructure [12]. In other investigation, about the effect of some process parameters such as the concentration and nature of the used activator solution, Puertas et al. showed that he rheology of AAS pasted activated with NaOH alone or Na_2CO_3 combined with NaOH, like Portland cement pastes, fit the Bingham model. These authors also confirmed that water glass-activated slag pastes fit the Herschel-Bulkley model. Moreover, increasing of Na_2O concentration leads to a raise of shear stress.

Phosphorus slag (PS) is one of industrial aluminosilicate waste that can be used as the starting raw precursors in producing alkali-activated cements. This aluminosilicate material is a non-ferrous amorphous slag, which is produced in the process of elemental phosphorus production [13–15]. Phosphorus slag has appropriate hydraulic properties due to the presence of calcium oxide and silica in its structure. However,

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https://doi.org/10.1016/j.cemconres.2018.07.010
Received 28 December 2017; Received in revised form 21 June 2018; Accepted 27 July 2018
0008-8846/ © 2018 Published by Elsevier Ltd.

Table 1
Chemical composition and Blaine fineness of phosphorus slag and OPC (wt% as oxides).

Component	SiO_2	Al_2O_3	Fe_2O_3	CaO	MgO	P_2O_5	K_2O	Na_2O	Blaine (kg/m^2)
Phosphorus slag	38.39	7.66	0.90	45.16	2.60	1.50	0.56	0.43	400 ± 0.1
Portland cement	21.99	4.31	4.15	62.88	2.86	-	0.47	0.21	405 ± 0.1

previous results have shown that phosphorus slag cannot be used as supplementary cementing materials due to the negative effect of phosphorus oxide on hydration reaction of phosphorus slag and reduction of its mechanical properties [13,15,16].

Shi and Li [16] presented a study on sodium silicate-activated phosphorus slag (AAPS). They reported that AAPS binder with low CaO/SiO_2 ratio is mainly composed of calcium silicate hydrate (C-S-H) and exhibiting 28 days compressive strength up to 120 MPa. You et al. [17] reported that properties of phosphorus slag-fly ash activated cement was dependent on the activator concentration, modules of sodium silicate solution and mix proportion of fly ash and phosphorus slag as raw aluminosilicate material. They showed that increasing of fly ash content lead to reduce the compressive, while have not effect on the setting time.

In the other study, Maghsoodloorad et al. [13] investigated the effect of concentration and type of alkali activator solutions at different silica moduli (SiO_2/Na_2O = 0.5, 1 and 1.5) on the 28 days compressive strength and. The results showed that alkaline solution with higher activity lead to achieve high strength up to 120 MPa after 28 days of curing. Maghsoodloorad and Allahverdi [18] also presented a study on the use of low-cost activators ($NaOH + Na_2CO_3$, $Na_2CO_3 + Ca(OH)_2$) for alkali-activated phosphorus slag. They have claimed that combinations of $NaOH + Na_2CO_3$ and $Ca(OH)_2 + Na_2CO_3$ can produce alkali-activated phosphorus slag binders with high 28-day compressive strength and low efflorescence. In the other investigation, Maghsoodloorad et al. [19] studied the effect of curing condition on compressive strength and microstructure morphology of mortar with phosphorus slag activated with two combined activator ($NaOH + Na_2CO_3$, $Na_2CO_3 + Ca(OH)_2$). They reported that change in the curing condition lead to reduce the porosity and increase the bonding between the aggregates and phosphorus-slag cement. Moreover, before hydrothermal curing in high temperature, a low-temperature procuring stage is effective in achieving high compressive strength. The effect of activator type and different curing conditions on efflorescence formation in the AAPS is studied by Maghsoodloo and Allahverdi [20]. Their results showed that the efflorescence formation survey in AAPS with sodium-containing activator solutions is more than potassium containing alkali activators. Also, addition of high alumina cements as additive to AAPS and hydrothermal curing of pastes (at 85 °C, 7 h and 95% of R.H.) could be used in order to reduction of efflorescence formation.

Mehdizadeh and Najafi [21] investigated the rheological behavior of phosphorus slag activated with sodium silicate solution and sodium hydroxide. Their results showed that the AAPS paste fitted on the Herschel-Bulkley model, and behaved like a shear-thinning fluid. Moreover, increasing of SiO_2/Na_2O molar ratio and activator concentration (Na_2O/Al_2O_3) leads to increase the yield stress and shear stress due to more gel formation. These authors also measured the apparent activation energy by using the rheological data based on Arrhenius viscosity model. They reported that increase of silica content of activator and decrease of activator concentration result in increase of the activation energy of AAPS paste.

Important novelty of this work is the use of non-conventional activators such as alkaline salts (Na_2CO_3 and Na_2SO_4) and investigation of rheological behavior of alkali-activated phosphorus slag. This salts in aqueous medium do not give rise to elevated pH values, but their interaction with $Ca(OH)_2$ as indicated in the Eqs. (1) and (2) can generate NaOH, which provides the level of alkalinity suitable for AASP.

$$Na_2CO_3 + Ca(OH)_2 \rightarrow 2NaOH + CaCO_3 \quad (1)$$

$$Na_2SO_4 + Ca(OH)_2 \rightarrow 2NaOH + CaSO_4 \quad (2)$$

Nevertheless, authors of this work have not found references on the rheological behavior of phosphorus slag activated with the combination of lime and alkaline salts as an activator.

Hence, in this paper, the rheological behavior, workability and the effect of parameters such as the nature and concentration of the activator on the rheological behavior (yield stress, Thinning index, and flow index) of alkali-activated phosphorus slag and comparison with OPC paste have been studied.

2. Experimental

2.1. Raw materials

Phosphorus slag is used as the starting aluminosilicate precursor and supplied from an Iranian phosphoric acid production plant in Iran. A commercial Portland cement, CEM I 42.5R (OPC), was also applied as a reference. The mineralogical, chemical composition and Blaine fineness according to ASTM C311 for the raw materials are given in Table 1.

The X-ray diffraction (XRD) pattern of phosphorus slag is shown in Fig. 1. As seen, a small amount of crystalline magnesium oxide (MgO, JCPDS 02-1207) presents in its structure, and it is almost completely amorphous.

The phosphorus slag as aluminosilicate material was grounded in a closed circuit laboratory mill (110 mm radius and 380 mm length) to a Blaine specific surface area of 400 ± 0.1 m^2/kg.

The particle size distribution of phosphorus slag and Portland cement was determined by a laser particle size analyzer and are shown in Fig. 2. The mean particle diameter of the Portland cement and phosphorus slag were 11.38 μm and 13.18 μm, respectively.

Analytical grade of calcium hydroxide ($Ca(OH)_2$, 99% purity, Maojallai), sodium carbonate (Na_2CO_3, 99.5%, Maojallai) and sodium sulfate (Na_2SO_4, 99.5% purity, Maojallai) were used for preparation of alkaline activator solutions.

2.2. Sample preparation

According to Table 2, different alkaline solutions consist of $Ca(OH)_2 + Na_2CO_3$ (activator No.1) and $Ca(OH)_2 + Na_2SO_4$ (activator No. 2) were used to activate the phosphorus slag.

To prepare activators, the stoichiometric amounts of Na_2CO_3, Na_2SO_4, and $Ca(OH)_2$ according to Eqs. (1) and (2) was used to produce two equivalent quantity of sodium hydroxide (3% and 5% by mass of solid materials) in cementitious mixtures.

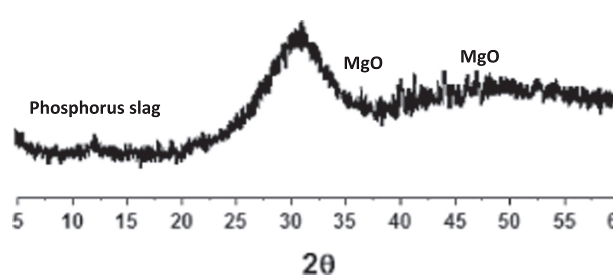

Fig. 1. X-ray diffraction data for phosphorus slag.

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