

# Durability of ternary blends containing high calcium fly ash and slag against sodium sulphate attack



Seyon Kandasamy<sup>a</sup>, Medhat H. Shehata<sup>b,\*</sup>

<sup>a</sup> *Inspec-sol Inc., Mississauga, ON, Canada*

<sup>b</sup> *Ryerson University, Toronto, ON, Canada*

## HIGHLIGHTS

- The addition of slag was found to enhance sodium sulphate resistance of cementing blends containing high-calcium fly ash.
- Blends with 60% Slag and high-calcium fly ash showed superior performance regardless of the individual level of slag or ash.
- Enhanced resistance to sulphate attack could not be explained based solely on the reduced ion diffusion through the mix.
- The resistance to sulphate increased with reduced calcium hydroxide and dilution of Portland cement.

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## ABSTRACT

Cementing blends containing high-calcium fly ash (HCFA) is known to have low resistance to sulphate attack. This paper presents the results of an experimental program that focuses on attempts to enhance the performance of HCFA through the incorporation of granulated blast furnace slag in the mix. The expansions of various blends were evaluated using the mortar bar test according to ASTM C1012. Mortar bars were made with 100% Portland cement (control mix), binary blends of either HCFA or slag, and ternary blends of HCFA/slag. The expansions were measured up to two years. The addition of slag was found to improve the resistance to sulphate. When the level of combined HCFA and slag reached 60% of the total cementing materials, there was a significant reduction in the expansion regardless of the level of each of the individual material. In addition to expansion results, the paper evaluates the role of chemical and mineralogical composition of the cementing blend, the ion diffusion of the mixtures, and the calcium hydroxide consumption on the sulphate resistance of the different blends.

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## 1. Introduction

High calcium fly ash (HCFA) is less effective in comparison to low calcium fly ash in controlling sulphate attack in concrete [1–4]. In fact, high calcium fly ash has shown adverse effect under sulphate exposure [3]. High calcium and high aluminasilicate glass contents of ASTM Class C fly ash are generally referred to as the dominant factors causing adverse effect in terms of resistance to sulphate [2,4,5]. Broadly, the resistivity of fly ash depends on its chemistry, reactive crystalline phases, and the amorphous phases [6]. Increased calcium oxide, high SO<sub>3</sub>, decreased SiO<sub>2</sub>, and Fe<sub>2</sub>O<sub>3</sub> are reasons for low sulphate resistance of concrete with HCFA. In addition, increase in reactive crystalline phases like C<sub>3</sub>A, periclase, or free lime are responsible for the poor performance of HCFA. Another explanation for the low sulphate resistance of HCFA is that the ash provides more available aluminate Al<sub>2</sub>O<sub>3</sub> and less sulphate

SO<sub>4</sub>, resulting in reduced ratio of SO<sub>4</sub>/Al<sub>2</sub>O<sub>3</sub>. Reduced SO<sub>4</sub>/Al<sub>2</sub>O<sub>3</sub> favours the formation of monosulphate [7], which later turns into expansive ettringite when the sample is exposed to sulphate attack.

As a way to enhance the performance of HCFA against sulphate attack, few researchers added gypsum to the mix to optimise the SO<sub>4</sub>/Al<sub>2</sub>O<sub>3</sub> and hence increase the resistance [8,9]. The additional sulphate ions in the form of gypsum consume calcium aluminates during early hydration of the cementing materials and stabilize the aluminates into ettringite leaving less aluminate for the late formation of ettringite after the concrete is hardened [8].

Silica fume has also been used with HCFA as a way to enhance the performance of the latter with respect to sulphate attack [8,10]. Both research works showed that adding small amount of silica fume (3–5%) along with high calcium fly ash (20–30%) produces significant reduction in expansion of mortar bars exposed to sulphate solution.

Granulated blast furnace slag of alumina content <11.0% has been found effective in mitigating alkali (sodium or potassium)

\* Corresponding author.

E-mail address: [mshehata@ryerson.ca](mailto:mshehata@ryerson.ca) (M.H. Shehata).

sulphate attack, specifically sodium sulphate, in samples submerged in the sulphate solution [8,11]. However, the case is not the same for samples partially or cyclically submerged in sulphate solution where the presence of slag accelerated the deterioration [12]. This is attributable to the formation of salt crystals at the part of the concrete not submerged in solution [12]. Yet, Gollop and Taylor [11,13] found that the alumina from slag is incorporated into the C–S–H or hydrotalcite-like phase  $[Mg_{1-x}Al_x(OH)_2]^{x-}$  [11,13,14]. Richerdson and Grove [14] showed that C–S–H of low Ca/Si ratio incorporate more alumina. Gollop and Taylor [11] found that one third to half the alumina in slag is incorporated in the Hydrotalcite-like phase. After 20 years of hydration, Taylor et al. [15] showed that the Ca/Si ratio of C–S–H of Portland cement/slag paste decreases and the Mg/Al of the hydrotalcite, referred to in the 2010 paper [15] as Mg–Al layered double hydroxide phase (LDH), also decreases. This later finding suggests that with hydration of slag, more alumina is incorporated in the hydration products. Gollop and Taylor [11] reported that alumina incorporated in C–S–H or hydrotalcite-like phases are not available to form ettringite. Another positive effect of the slag is the reduction in calcium hydroxide as well as refining of pore structure. The latter is claimed by Gollop and Taylor [11] to accommodate more ettringite without excessive expansion due to the large number of fine pores. The fine pores promote the formation of short ettringite crystals with less expansive pressure.

In this paper, slag was used with HCFA to investigate whether the addition of slag can counteract the negative effect of HCFA on sulphate resistance. One way by which slag is expected to reduce the sulphate expansion is by binding the  $Al^{3+}$  in its low-Ca/Si C–S–H [11,13,16]. Hence, the ability of hydration products of slag to bind  $Al^{3+}$  could bind the reactive  $Al^{3+}$  from the HCFA and thus help in reducing the alumina available for the formation of ettringite.

## 2. Materials

Silica (Ottawa) sand, General Use Portland cement (GUPC), high calcium fly ash (HCFA), and granulated blast furnace slag (slag) were used in this experimental program. Table 1 lists the chemical composition of the cementing materials used in this study. In addition to chemical composition, the X-ray diffraction patterns of the HCFA and slag samples are shown in Fig. 1. The diffraction patterns were collected using Scintag XDS 2000 X-ray diffractometer with  $Cu K\alpha$  radiation ( $\lambda = 1.540562 \text{ \AA}$ ).

## 3. Experimental details

### 3.1. Mortar bars

ASTM C 1012 test standard was followed to measure the expansion of mortar bars exposed to 5%  $Na_2SO_4$  solution up to 2 years. Mortar is prepared as per ASTM C 305. Stainless steel studs were inserted on both ends of the mortar bars to enable the measurements. The expansion of the mortar bars was taken at 1, 2, 3, 4, 8, 12, 13, 15, 26, 39, 52, 78, and 104 weeks. Table 2 lists the proportions of the tested cementing materials.

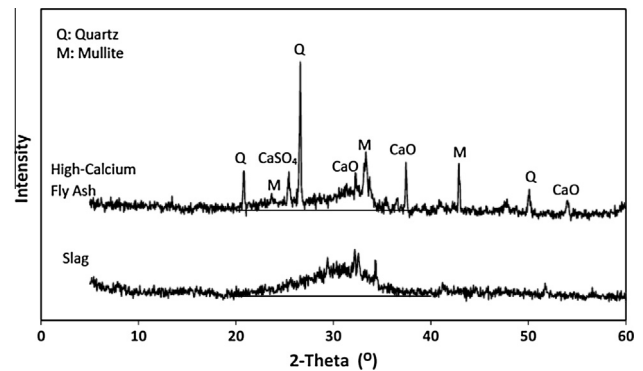
### 3.2. Thermal gravimetric analysis

Paste samples were prepared at the same cementing materials combinations shown in Table 2 using a high-speed, high-shear

**Table 1**

Chemical composition of cementing materials.

	SiO <sub>2</sub>	Al <sub>2</sub> O <sub>3</sub>	Fe <sub>2</sub> O <sub>3</sub>	CaO	MgO	SO <sub>3</sub>	K <sub>2</sub> O	Na <sub>2</sub> O	TiO <sub>2</sub>	P <sub>2</sub> O <sub>5</sub>	S.G.
PC	19.58	5.35	2.29	62.84	2.43	4.1	1.13	0.21	0.31	0.11%	3.15
HCFA	33.72	18.47	7.04	27.16	5.08	2.84	0.33	1.72	1.41	0.91%	2.65
Slag	34.40	7.40	0.94	43.20	9.30	0.83	0.58	0.57	0.44	175 ppm	2.94



**Fig. 1.** X-ray diffraction of raw HCFA and Slag.

**Table 2**

Blends prepared to evaluate sulphate susceptibility.

Mix ID	PC (%)	HCFA (%)	Slag (%)
Control	100	–	–
20%HCFA	80	20	–
40%HCFA	60	40	–
20%Slag	80	–	20
30%Slag	70	–	30
40%Slag	60	–	40
15%HCFA+15%Slag	70	15	15
20%HCFA+20%Slag	60	20	20
30%HCFA+30%Slag	40	30	30
20%HCFA+30%Slag	50	20	30
20%HCFA+40%Slag	40	20	40
40%HCFA+20%Slag	40	40	20

food blender. The w/c or w/cm was kept 0.5 for all the samples. The prepared paste blends were poured into plastic moulds of 50 mm diameter and 100 mm height and cured at 100% relative humidity and 35 °C for the first 24 h. Then, the samples were demoulded and soaked in limewater at 23 °C for another 27 days. After that, the samples were taken out of limewater, broken into pieces and soaked in alcohol for 1 week. Then, the samples were stored at 80 °C for one more week. On the day of running TGA, the fragments were taken out of the oven and ground into fine powder. Analysis was carried using AutoTGA 2950HR V6.1A on the powder.

### 3.3. Rapid chloride permeability test

ASTM C1202 – Rapid Chloride Permeability Test was conducted on the corresponding mortar mixes of the sulphate attack mortar bars. Though ASTM C1202 is intended for testing concrete specimens, it is adapted in this study for mortar mixes. Mortar mixes were prepared according to ASTM C 305 and cast into the standard cylinders 100 × 200 mm in three layers. The samples were cured in saturated lime water until the time of testing, 56 days, when the cylinders were cut into 50 mm thick specimens, and tested following the procedures of ASTM C1012. The average RCPT values obtained from two specimens of the same mortar cylinder (bottom portion and the middle portions) are reported here.

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