



Investigating the two-dimensional diffusion-reaction behaviour of sulphate ions in cement-based systems

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HIGHLIGHTS

- A two-dimensional approach to evaluate distress in cementitious systems due to sulphate attack is described.
- The effect of embedded aggregate on the outcomes from sulphate attack is illustrated.
- Effect of curing conditions and duration of exposure to sulphate attack are shown.
- Crack growth over time is predicted using the measured sulphate ingress and constitutional properties.

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ABSTRACT

This paper describes experimental approaches to evaluate two-dimensional sulphate attack behaviour inside cement-based systems. First, the sulphate content at various depths was measured using titration. This was followed by mechanical evaluation on cylindrical samples to establish the associated stress-strain response. Three types of binder namely, Portland cement conforming to CSA Types GU and HS and, a 30:70 blend of fly ash and Portland cement Type GU, were employed. The specimens were immersed in a sulphate rich solution for up to 12 weeks. The results show that all specimens suffered more from two-dimensional sulphate attack as compared to that seen with exposure in one dimension. Also, the positive role played by an embedded aggregate emerged in that it was seen to impede the transport of sulphate ions inside the cement-based system. As well, self-healing under prolonged exposure was observed, which was more significant with both binders that contained Portland cement Type GU.

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1. Introduction

Adverse sulphate attack, is a significant durability concern, as it reduces concrete structures to rubble through the formation of expansive products such as ettringite [1–4] or gypsum [5]. Even before such absolute degradation, the external sulphate ions react with the inherent calcium aluminates, causing expansive strain, resulting in the growth and propagation of micro-cracks. Accordingly, external sulphate attack continues to be an extensively examined topic of study. For starters, the composition of the binder, the external environment and the curing conditions are known to influence. It is widely recognized that the ionic composition and concentration in the external environment affect the nature of sulphate attack inside cement-based systems [6–9]. In their study on the sulphate resistance of plain and blended cements exposed to

varying sulphate concentrations, Al-Dulaijan et al. [6] found a reduction in compressive strength with an increase in the concentration of sulphate in the environment. Further, this increase in the concentration of sulphate in the environment is seen to manifest as accelerated leaching of portlandite inside the cement-based system to cause further loss in mass and strength [7]. Due to the chemically active nature of hydrated cement paste, the properties of cement-based materials evolve with time [10]. Hence, the curing condition plays a significant role upon sulphate resistance. For instance, specimens exposed to a sulphate rich environment registered larger equivalent damage depth as well as relative expansion, at only 3 days of maturity, as compared to other specimens that were exposed at 28 days of maturity [11]. Nehdi [12] noted that moist curing is seen to alleviate surface scaling and so, lessen the damage under sulphate attack [12]. The mixture composition and especially, the water-to-binder ratio has been widely examined for influence on the behaviour after sulphate attack. Blending with fly ash is known to impart superior resistance with the Class F subtype performing better than the Class C [6,13]. While it is well

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known that reducing C_3A in the binder is helpful, by itself though, this step may not be significant - At all times, a lower water-to-binder ratio is favourable to improving sulphate resistance [14]. Also, mechanical evaluation is not sufficient to establish sulphate attack as, often the system may undergo a cycle of cracking and healing regimes [15].

In this paper, the sulphate attack in cement-based systems is examined for a two-dimensional ingress. Three different binders namely, Portland cement Type GU, Type HS and a third binder made to a 30:70 blend of fly ash and Portland cement Type GU, were employed to result in different mixtures, that were in turn subjected to three curing durations. Subsequently, these cured specimens were immersed in sulphate-rich solution with a molar concentration of 352 mol/m^3 and, were retrieved after varying durations of exposure for further testing to evaluate the sulphate content and mechanical properties. Furthermore, this study also examines how the presence of aggregates affects sulphate resistance in plain cement paste.

2. Experimental program

2.1. Materials and mix proportions

Three different binders were examined in this study, (i) Portland cement Type GU (General Use), (ii) Portland cement Type HS (High Sulphate-resistant), both conforming to ASTM C1157/C1157M-17 [16], and (iii) a 30:70 blend between Class C fly ash and the Portland cement Type GU. Their chemical composition is listed in Table 1. The fine aggregate employed was a standard concrete sand with three grading distributions (Size 3.4, Size 2.4 and Size 1.0). The associated mix proportions as designed here are shown in Table 2. The fresh mixtures so produced was cast into prismatic moulds, to determine the sulphate content later, and into cylindrical moulds, for mechanical testing. Casting was accompanied by vibration in order to ensure homogeneity as per ASTM C490 [17]. Note here that there were three batches of prismatic samples produced with aggregates embedded, which were used for further evaluation upon aggregate effect on sulphate transport inside cement-based system. And for each of them, six round aggregates (as near spherical as possible) with diameter equal to 30 mm were embedded at the centroid with equal space along length, namely at 25, 75, 125, 175, 225 and 275 mm. In addition, a few thin bars were used to fix those aggregates as close to the middle as possible. Later, upon sawing the samples, those samples where in the aggregate was seen to be away from the centre were discarded. The specimens were demolded after 24 h and transferred to a controlled environment to experience standard curing conditions (23 centigrade degrees and relative humidity 95%). They were left to cure variously for 7, 28 and 56 days. Fig. 1 presents a flowchart to illustrate better the process for producing the specimens examined in this study. Seen from that, for all 16 prismatic specimens, each of them, originally with size of $50 \text{ mm} \times 50 \text{ mm} \times 300 \text{ mm}$, was evenly cut into 6 cubic samples of side 50 mm, shown as Fig. 2 (a). These were examined to determine the sulphate content later. The samples with an embedded round aggregate were cut further along the thickness in order to result in an inhomogeneous two-dimensional diffusion-reaction region, whose size was $25 \text{ mm} \times 50 \text{ mm} \times 15 \text{ mm}$, as shown in Fig. 2(b). In the meanwhile, the paste samples serving as the reference control group were sawn from the cubical samples that were produced previously from the 300 mm long prismatic paste specimens. For each binder and duration of exposure, three cylinders were cast at $\phi 75 \text{ mm} \times 150 \text{ mm}$ in dimension.

Table 1
Chemical composition of binders (Portland cement Type GU, Type HS, Class C Fly ash).

Composition	Unit	GU Cement	HS Cement	Fly ash
SiO ₂	Mass [%]	20.4	20.6	55.5
Al ₂ O ₃	Mass [%]	4.6	3.3	23.2
Fe ₂ O ₃	Mass [%]	3.4	4.7	3.6
CaO	Mass [%]	62.7	62.3	11.0
MgO	Mass [%]	2.8	2.75	1.2
SO ₃	Mass [%]	2.7	2.3	0.2
K ₂ O	Mass [%]	0.5	0.5	0.8
Na ₂ O	Mass [%]	-	-	2.8
P ₂ O ₅	Mass [%]	-	-	15.7
TiO ₂	Mass [%]	-	-	0.7
C ₃ S	Mass [%]	57	62	-
C ₂ S	Mass [%]	15	12	-
C ₃ A	Mass [%]	6.3	1.1	-
C ₄ AF	Mass [%]	10.3	14.1	-

Table 2
Mixture component and proportion (kg/m^3).

GU/HS Samples		IC Samples	
Sand	1500 kg (total) Size 3.4 = 970 kg Size 2.4 = 300 kg Size 1.0 = 230 kg	Sand	1470 kg (total) Size 3.4 = 960 kg Size 2.4 = 290 kg Size 1.0 = 220 kg
Binder	540 kg	Binder	535 kg
Water	260 kg	Water	260 kg

2.2. Evaluating sulphate content

Each cube specimen was prepared to ensure that a two-dimensional sulphate attack regime occurs inside the cement-based system. Accordingly, they were sealed with an epoxy resin on four sides of each cube sample, leaving two adjacent orthogonal sides exposed to the sulphate solution directly, see Fig. 3(a). In those specimens, which were embedded with a round aggregate, two symmetric sides as shown in Fig. 3(b) were left uncoated to experience external sulphate attack. The underlying belief was that inhomogeneous two-dimensional diffusion-reaction of sulphate ions will take place inside the specimen due to the existence of an inhomogeneous diffusion-reaction area caused by that aggregate. These cubic specimens so prepared, were immersed in a sulphate rich environment in accordance with ASTM C1012 [18]. The sulphate bath was prepared in a vat using 50 g of anhydrous sodium sulphate per liter of water, corresponding to 352 mol per cubic meter. This produced a pH between 7.5 and 7.8, which was consistently maintained through the duration of the study. The specimens were taken out from this vat for subsequent testing after exposure durations ranging from 0 to 12 weeks. In a study on 1-dimensional sulphate attack by Bindiganavile et al. [15], it was found sufficient to sample from within 12 mm from the specimen surface, for measuring depth and concentration of penetrated sulphate. Hence, the samples used for further chemical titration in the present study were collected from 0 to 20 mm depth. Further, the process of drilling was seen to fracture the specimens and so, it was deemed necessary to drill away from the centre of the specimen but along its diagonals as shown in Fig. 4a. The drilling scheme for collecting powder samples is shown in Fig. 4 while Fig. 5 illustrates the actual operation using a press-drill machine to collect the powders from cube specimens. For prismatic specimens with a single embedded aggregate, the same machine was used and, the only difference was that the drilling direction was perpendicular to the exposed face. It should be emphasized here that, the samples drilled out were automatically in a powder form. So that, the powder samples in each case (ie. the paste and the aggregate-embedded specimens) were collected into plastic storage tubes to prevent contamination. Prior to determining their sulphate content, every one of the collected powder samples was oven-dried and weighed. They were subsequently dissolved in 20 ml distilled water under boiling temperature ($\sim 95^\circ\text{C}$). This hot solution was treated with 5 ml hydrochloric acid, followed by stirring for 60 min. Note here that another 20 ml hot distilled water ($\sim 95^\circ\text{C}$) was added into the stirring sample at 30 min to ensure the solution temperature remained close to boiling. In the process, it was recognized that the concentration of the powder dropped to half the initial concentration. After stirring for 60 min, the solutions were left undisturbed for another 30 min to enable preliminary sedimentation. At this point, the solution was centrifuged in a Thermo Scientific LYNX-4000 machine. The resultant so obtained was filtrated to produce approximately a 40 ml dilute without any insoluble substance. Conforming to ASTM C 114-13 [19], 10 ml of BaCl₂ solution was thereafter added into each such dilute sample. The reaction associated with precipitating the sulphate is per Eq. (1).



At the end of the titration process, a further 30 min were allowed to ensure that the barium sulphate precipitation was complete. A series of operations followed, whereby the empty crucibles were weighed and the precipitate underwent air-filtration, oven-drying and weighing. The total sulphate content and its distribution inside each cementitious system was obtained per Eq. (2). At this point, note that this value for the sulphate content includes not only those sulphate ions which ingressed from the external environment, but also those inherent in the constituent materials. Hence, it is essential that the inherent sulphate ions be deducted in order to obtain the sulphate that has invaded from the exterior. As such, a batch of specimens was retrieved immediately upon curing but prior to any sulphate exposure, and the corresponding data is listed in Table 3.

$$U = \frac{m_{pre} \times \rho_{cm}}{M_{BaSO_4} \times m_{sp}} \quad (2)$$

where:

- U = sulphate concentration tested in titrated sample (mol/m^3);
- m_{pre} = the weight of barium sulphate precipitates (g);
- m_{sp} = the weight of powders sample (g);
- ρ_{cm} = density of cement mortar sample (g/mm^3);
- M_{BaSO_4} = molar mass of barium sulphate (g/mol).

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