



The effect of chloride on cement mortar subjected to sulfate exposure at low temperature



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HIGHLIGHTS

- Depending on concentration, chloride accelerates or mitigates thaumasite attack.
- Accelerated attack due to 0.5% chloride in 0.6% magnesium sulfate is first reported.
- 2% chloride in the same solution mitigates TSA, but long-term vulnerability remains.
- The onset of TSA is earlier and extent greater, for higher limestone content binders.

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ABSTRACT

The effect of chloride on the performance of cement mortars made with CEMI and CEMI blended with 10% limestone filler and subjected to sulfate attack was investigated. The specimens were exposed to the combined action of chloride (0%, 0.5%, 1.0% and 2% of Cl^-) and sulfate (0.6% SO_4^{2-}) for 630 days at $5 \pm 0.5^\circ\text{C}$. The performance of the mortar cubes was assessed by means of visual inspection and loss of mass. The mineralogy of the deterioration products was determined by infra-red spectroscopy and X-ray diffraction, and scanning electron microscopy was used to examine the microstructure of the specimens. Chemical analysis of the test solutions was also carried out at 540 days of immersion in order to assess the end concentrations of calcium, sulfate and magnesium ions. All the specimens suffered from the thaumasite form of sulfate attack, except those stored in the combined sulfate and 2.0% chloride solution, which showed better performance within the 630-day study. The greatest deterioration occurred in mortars made with CEMI blended with 10% limestone filler. Based on visual and mass assessments, it was found that the effect of chloride on thaumasite degradation depended on chloride concentration, accelerating the damage at 0.5% and mitigating it at 2% in the conditions investigated. This is probably related to the reduction in calcite solubility and the increase in Friedel's salt deposition observed as the chloride level increased in solution.

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1. Introduction

Exposure of concrete structural components to external sulfates can lead to deterioration owing to the formation of expansive phases, including ettringite, gypsum and thaumasite, depending on the exposure conditions. The availability of sulfate, calcium, carbonate and silicate in the presence of water at low temperatures promotes the formation of thaumasite [1]. Two different mechanisms are proposed for thaumasite formation: the direct route and the woodfordite route. In the direct route, thaumasite results from reaction between C–S–H, calcium sulfate, calcium carbonate and water [2], whereas in the woodfordite route, it is proposed

[3] that thaumasite forms as a result of reaction between ettringite, C–S–H and carbonate ions in the presence of excess water. It has also been proposed that thaumasite can form due to the topochemical replacement of ettringite by a through solution mechanism [4].

It is generally accepted that for extensive thaumasite formation to occur, low temperature conditions are necessary, however, various researchers [5–10] point out the possibility of its formation at higher temperatures. The deterioration of mortar and concrete made with Portland-limestone cement due to the formation of thaumasite during exposure to sulfates at low temperatures is well documented [11–14].

Concrete structures can also be exposed to chloride ions in addition to sulfate, particularly in structures near the sea, in arid areas or where sodium chloride is used as a de-icing salt during winter

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time. Previous studies [15–19] have shown that the presence of chloride ions in conjunction with sulfate ions delays or mitigates the conventional form of sulfate attack, where ettringite is the main deterioration product; however, there is very little information available on sulfate attack where chloride ions are also present at low temperatures such that the conditions are conducive to thaumasite formation. In an experimental study carried out at Sheffield University [20] it was found that the extent of deterioration due to thaumasite formation in 20 mm mortar cubes depended on the chloride level in solution, deterioration was greater as chloride levels increased from 0.5% to 2.0%. On the other hand, in recent studies [21–22] in which 100 mm concrete cubes were exposed to 2.1% chloride concentration, it was reported that thaumasite sulfate attack was mitigated in limestone concrete by the presence of chloride ions. In order to shed more light on the influence of chloride ions on thaumasite formation, this paper reports and discusses the results of an experimental study into the effect of chloride concentration on the performance of CEMI and CEMI blended with 10% limestone filler cement mortars subjected to conditions favorable to thaumasite formation. The samples were exposed to the combined action of sulfate and chloride solutions for 630 days at low (5 °C) temperature during which they were monitored using visual observation, mass change, chemical analysis of test solutions, X-ray diffraction, infra-red spectroscopy and scanning electron microscopy.

2. Experimental programme

The mortar mixtures shown in Table 1 were used to prepare 50 mm cubes and 40 mm square by 160 mm long prisms. A fixed water to binder ratio of 0.6, cement to sand ratio of 1:2.5 and siliceous sand were used to prepare the specimens. This high *w/b* ratio was selected to accelerate chemical diffusion and interaction, thus allowing identification of any potential reactions within a shorter time frame. Commercial grade CEMI complying with BS EN197-1: 2011 [23] and containing approximately 3.5% limestone filler as determined by thermogravimetric analysis was used in this study. The chemical and mineralogical composition of the cement and limestone are given in Table 2.

After casting, the specimens were covered with plastic sheets for the first 24 h, de-moulded and then placed in water for 6 days at 20 °C. They were then air-cured at room temperature (20 ± 1 °C) for 21 days, after which all the cubes were weighed and then transferred with the prisms to their designated exposure solutions (Table 3) in small tanks maintained at 5 ± 0.5 °C. Exposure solutions were made by dissolving sodium chloride (NaCl) and Epsom salt (MgSO₄·7H₂O) in deionised water. The concentration of sulfate was equivalent to Design Sulfate Class DS4 according to BRE Special Digest 1 [24]. In order to compensate for the reduction in ion concentrations at the early stages of the experiment, the solutions were renewed every three months up to one year. After this period of rapid changes in ion concentration had ceased and due to extensive deterioration shown by some samples, the solutions were not changed. The tanks were kept covered for the entire duration of the experiment to prevent solution evaporation.

A visual assessment of the specimens was made regularly by removing the cubes from the solutions. Particular note was made of any changes in color, spalling and precipitation of any materials. The specimens were weighed so that changes in specimen mass could also be recorded. Before weighing, specimen surfaces were wiped dry and any loose parts were removed by hand.

X-ray diffraction was performed on samples of any degraded deposits scraped off specimen surfaces. The material was dried at room temperature to avoid any change in crystallization of the degraded samples, and then intermittently crushed in pulses of about 10 s to lessen heat generation using a porcelain mortar and pestle to pass 63 µm sieve, which would have reduced the proportion of sand particles in the powder. The powder was then examined by a Philips PW 1830 X-ray generator using a copper electrode operating at 40 kV and 30 mA with a scanning speed of

2θ/min with step size 0.02 over a 2θ range of 5–55. The database of the Joint Committee for Powder Diffraction Files (JCPDF) built into WinXPOW software was used to identify the different phases in the patterns.

The samples for IR spectroscopy were made using 2 mg of sample to 200 mg of potassium bromide which were ground simultaneously until a fine homogeneous material was achieved. A 12 mm diameter disc of the sample was then prepared and analyzed using a Perkin-Elmer FT-IR 2000 spectrometer.

Examination by scanning electron microscopy was used to identify the nature of the deteriorated products after 360 days of immersion. Fractured samples from the surfaces of prisms were collected and dried at 20 °C in desiccators and then fixed with carbon tape on a metal disc, exposed to a vacuum to remove any moisture and dust and immediately examined using an Electron Microscope (Carl Zeiss Merlin Field Emission SEM). After 450 days of immersion, 10 mm cubes were cut from the corners of the prisms. Sample preparation involved drying, pressure impregnation with epoxy resin, grinding, polishing and coating with carbon. The microstructure of each sample was then examined using an Inspect F scanning electron microscope (SEM) with an accelerating voltage of 20 kV. Chemical analysis using an energy dispersive X-ray (EDX) system and the appropriate link software were employed to identify the phases present.

The concentrations of sulfate, calcium and magnesium ions in the solutions were measured every 3 months up to 540 days after immersion, using ion chromatography on samples that had been diluted, due to very high ion concentrations of the solutions. The samples remained in solutions until the end of the experiment at 630 days.

The effect of chloride level on the solubility of calcite and gypsum was also investigated. Calcite and gypsum were dissolved in prepared solutions in concentration similar to those used in this study. The solutions were manually agitated on daily basis. After a week, the solutions were filtered and samples were analyzed using ion chromatography. The amount of calcium ions in the solutions was used as indication for both calcite and gypsum solubility.

3. Results and discussions

3.1. Visual observations

The first signs of deterioration were detected at the corners and edges of the specimens after 100 days of immersion in all exposure solutions except the combined sulfate and 2.0% chloride solution (SC20). This was more evident in the CEMI-LF specimens. The visual appearance of mortar specimens after 360 days of exposure to sulfate only (S) and 0.5% Cl (SC5) solutions is reported elsewhere [25]. As time progressed the degree of deterioration depended on the presence of chloride in the test solutions and the carbonate content of the cement. After 630 days of immersion, as can be seen in Fig. 1, the (SC5) specimens suffered greater damage than those in sulfate only (S) and SC10 solutions, where the damage sustained by CEMI-LF specimens was more serious; but those in combined sulfate and 2.0% chloride (SC20) solution showed no deterioration at this age. It seems that the presence of 0.5% chloride in the sulfate solution accelerated the deterioration due to sulfate attack, while the sulfate solution containing 2.0% chloride resulted in mitigation of attack.

3.2. Mass change

Figs. 2 and 3 show changes in mass with time for mortar specimens during exposure to the test solutions. From Fig. 2 it is clear that after immersion all CEMI specimens continued to gain weight in all test solutions. However, whereas this increase in weight continued in specimens stored in combined sulfate and 2.0% Cl⁻ (SC20) for the whole exposure period, specimens stored in combined sulfate and 0.5% Cl⁻ (SC5) solution started to show loss in mass after 160 days. Specimens stored in sulfate only (S) solution began to lose mass after 270 days. Mass loss was greater for CEMI-LF mortar (Fig. 3) than for CEMI mortar; at 630 days of exposure the mass of the CEMI-LF mortar in SC5 had dropped by 9.4%, in contrast to only 3.7% for CEM1 mortar. The increased vulnerability of concrete containing limestone to damage due to thaumasite formation is well documented [11–14,26–28] so this result is not unexpected. As can be seen in Fig. 3, mass reduction for CEMI-LF mortar in SC5 was much higher than that found for those in S

Table 1
Mortar mixtures.

Mix	Binder	Per weight of binder			
		Cement	Limestone	Water	Sand
CEMI	CEMI	1	0	0.6	2.50
CEMI-LF	CEMI + 10% LF	0.9	0.1	0.6	2.50

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