



Corrosion behaviour of steel rebar in mortars subjected to magnesium sulfate and sodium chloride mixtures at 5 and 20 °C



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HIGHLIGHTS

- Depending on concentration, chloride can accelerate or mitigate TSA.
- 0.5% chloride in DS4 magnesium sulfate solution accelerates TSA and rebar corrosion.
- 2% chloride in DS4 solution mitigates TSA and versus 0.5% Cl⁻ reduces corrosion risk.
- TSA and consequent corrosion risk increase with rise in the cement's lime content.

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ABSTRACT

Although exposure to sulfate and chloride ions separately is known to result in deterioration of concrete and corrosion of steel rebars, the effects of both ions being simultaneously present are ambiguous, with some studies showing the presence of chloride to be beneficial, while in others attack was accelerated. This paper describes an investigation into the corrosion activity of steel rebars embedded in cement mortars made with CEMI and CEMI blended with 10% limestone filler subjected to combined sulfate (0.6% SO₄²⁻ and 0.152% Mg⁺² as Epsom salt) and chloride (0.5 and 2.0% Cl⁻) at both 5 and 20 °C. Evaluation was made by means of linear polarization resistance (LPR) and visual inspection of the mortar specimens and the rebar surfaces. X-ray diffraction (XRD), scanning electron microscopy (SEM) and energy dispersive X-ray (EDX) analysis were also carried out on selective materials collected from steel-mortar interfaces. Mortar specimens stored in composite sulfate and 0.5% chloride solution at 5 °C showed deterioration due to thaumasite formation and the associated rebars suffered greater corrosion than for other mixtures. It is concluded that thaumasite formation resulting in the loss of chloride binding in the cement mortars is a crucial factor in accelerating steel corrosion.

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1. Introduction

Several field cases, for example Slater et al. [1] and Eden [2] have reported the presence of chloride in pore fluids where concrete has suffered thaumasite sulfate attack (TSA). Such sulfate attack, with chloride also present, may occur particularly in concrete structures in or near the sea, where ground water becomes mixed with seawater or is contaminated with sea spray. In addition, significant amounts of chloride ions may be present as a result of the use of road de-icing salts during winter time, where concentration will vary according to run-off, and also in arid climates

where increases in sulfate and chloride ions occur as a result of evaporation.

Although according to the UK Government's Thaumasite Expert Group report [3] and studies by Zuquan et al. [4] and Eklou et al. [5], the presence of chloride mitigates or reduces the vulnerability of concrete to conventional sulfate attack, in which ettringite is the main deterioration product, Torres [6] and Sotiriadis et al. [7] note that there is very little information available about vulnerability to the thaumasite form of sulfate attack of concrete/mortar simultaneously exposed to both chloride and sulfate solutions at low temperature. According to a recent experimental study carried out at the University of Sheffield [8], the extent of deterioration due to thaumasite formation in 50 mm mortar cubes depended on chloride concentration in solutions. Whereas 0.5% chloride in solution resulted in accelerated attack, higher chloride concentrations

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reduced thaumasite formation so that at 2.0% chloride, attack was mitigated in the 30 month long study. Research by Sotiriadis et al. [7], reported mitigation of sulfate attack when a 2.1% chloride concentration was present in solution.

An additional potential serious impact associated with the presence of chloride ions is a higher risk of corrosion of steel reinforcement. Based on the observations made during inspections and analyses of concrete structures by Wimpenny and Slater [9] for the Highways Agency (now Highways England), TSA development may result in an increased risk of chloride penetration to the depth of the reinforcement. The chloride binding capacity of cement matrix was reported [8] to be affected by TSA, however it would appear that the published literature is lacking reports of any investigations into the effect of thaumasite related damage on the mortar/concrete cover and impact on steel reinforcement. Accordingly, it would be of great interest to investigate the impact of TSA on the corrosion resistance of steel reinforcement in concretes of different types, which is the focus of this paper.

The results of an experimental study into the evaluation of corrosion of steel rebars embedded in cement mortars made with CEMI and CEMI blended with 10% ground limestone filler, which have contrasting vulnerability to TSA, exposed to the combined action of sulfate and chloride solutions at 5 and 20 °C are reported and discussed. Although a binder with added limestone would not normally be specified for situations in which exposure to sulfate is expected, it was chosen in this study to provide a binder of contrasting TSA susceptibility compared with CEMI, in which up to 5% undeclared limestone may be present. Corrosion activity was monitored using the linear polarization resistance (LPR) method during the experiment and the state and extent of corrosion was determined by visual inspection of the state of rebar surfaces afterwards. Representative samples of both sound and degraded mortars from steel-mortar interfaces were subjected to X-ray diffraction (XRD), scanning electron microscopy (SEM) and energy dispersive X-ray (EDX) analysis to determine the extent and nature of deterioration of the mortars.

2. Experimental programme

The mortar mixtures shown in Table 1 were used to prepare 50 mm diameter by 100 mm high cylinders with mild steel bars centrally cast in samples. The bars had been coated with bitumen at the mortar-air interface and at their ends to prevent crevice corrosion.

A fixed high w/b ratio (0.6) was selected to accelerate chemical diffusion and interaction, thus allowing identification of reactions within a shorter time frame. Commercial grade CEMI complying with BS EN197-1 [10] and containing approximately 3.5% limestone filler, as determined by thermogravimetric analysis, was used. The chemical and mineralogical composition of the cement and limestone are given in Table 2. After casting, the specimens were covered with polythene sheet and left for 24 h, de-moulded and then placed in curing water for 6 days at 20 °C. They were then air-cured at room temperature (20 ± 1 °C) for 21 days prior to being transferred to their designated exposure solutions in small tanks maintained at 5 ± 0.5 °C and room temperature (~20 °C). The volume of specimens to volume of solution ratio was 1:3. The composite exposure solutions contained 0.6% sulfate and 0.152% Mg⁺² with either 0.5% (SC5) or 2.0% chloride (SC20). These were made by dissolving the requisite amounts of 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 [11] and BS 8500 [12]. In practice it would not be advisable for unprotected concrete containing limestone filler to be utilised in these conditions, but in this series of tests it provided a means of assessing the impact of susceptibility to TSA on the extent

Table 2

Chemical and mineralogical composition [weight %] of cement and limestone. (LOI = Loss on ignition).

	Cement	Limestone
SiO ₂	19.85	0.63
CaO	64.61	55.2
Al ₂ O ₃	4.67	0.28
Fe ₂ O ₃	2.74	0.15
Na ₂ O	0.23	<0.003
K ₂ O	0.45	0.054
MgO	1.09	0.47
SO ₃	3.015	<0.002
LOI	2.52	42.89
C ₃ S	68.28	–
C ₂ S	5.4	–
C ₃ A	7.74	–
C ₄ AF	8.34	–

of steel corrosion as well as to shorten the duration of the experiments. Furthermore as CEMI may contain limestone filler, it was important to study the effects of varying the amount of limestone present in the binder. In order to compensate for the reduction in ion concentration at the early stages of the experiment, the solutions were renewed every three months up to one year. After this period 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.

After 720 days of exposure, mortar specimens were transferred from covered boxes to open large containers with similar solutions at room temperature (~20 °C). The samples were left to equilibrate in temperature to 20 °C for one day before the first LPR measurements were taken. After this, a positive 100 mV DC voltage was applied to the steel bar (anode), where the negative terminal consisted of stainless steel mesh (cathode) immersed in the solution. This was to accelerate the rate of corrosion for the remaining 180 days of the test. LPR measurements were taken again at 900 days of exposure, after which the specimens were broken open to facilitate visual examination of the steel surfaces and for the removal of samples of the inter-facial mortar. Because the solutions were subject to evaporation, the levels in the containers were regularly checked and topped-up with fresh solution. In the case of the 900 days LPR measurements, to ensure the system readings was stable, the readings were taken 3 days after disconnecting the +100 mV applied voltage. Some samples were repeatedly tested in order to examine the accuracy of the test, but these measurements did not significantly vary from each other.

The electrochemical measurements were made using a VersaSTAT 3F potentiostat manufactured by Princeton Applied Research, where the experimental setup is shown in Fig. 1. A carbon rod placed outside the specimen was used as a counter electrode, while a saturated calomel electrode was used as the reference electrode. The polarization resistance (R_p) determinations were performed at a scan rate of 0.167 mV/s over a range of ±10 mV. A computer program, VersaStudio model, developed by EG&G Princeton Applied Research, was used for applying the potential scan, analysing the data, and calculating the polarization resistance (R_p) and corrosion current (I_{corr}) parameters. Since, the purpose of the tests in this study was to provide comparative data rather than absolute values of the parameters, no corrections were made to the results, which would probably have resulted in values being over-estimated [13].

For XRD testing, material collected from steel-mortar interfaces was dried at room temperature, gently ground by hand using a cleaned agate pestle and porcelain mortar to a fine <63 µm powder. This was examined using a Philips PW1830 diffractometer using monochromatic CuK α radiation operating at a voltage of 40 kV and current of 30 mA.

Scanning electron microscopy (SEM) and energy dispersive X-ray (EDX) analysis were also performed on selected sound- and deteriorated steel-mortar interface mortar samples. Samples were dried at room temperature before being pressure impregnated with epoxy resin to form discs which were then ground, polished and coated with carbon. They were 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.

Table 1
Mortar mixtures.

Mix	Binder	Per weight cement			
		Cement	Limestone	Water	Siliceous sand
A	CEMI	1	0	0.6	2.50
B	CEMI-LF	0.9	0.1	0.6	2.50

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