



The effect of sea water on the phase assemblage of hydrated cement paste



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ABSTRACT

When concrete is exposed to sea water, it has been observed that the composition of the outer most millimeters of the concrete is considerably altered compared to the composition of the bulk concrete. The limited size of this zone complicates the investigation of the phases formed. This paper presents a new experimental set-up in which hydrated cement paste is exposed to sea water allowing a detailed investigation of the phase changes observed in that zone on a bulk material. The paste was characterized before and after sea water exposure by XRD, DTA/TG, ICP-MS and SEM-EDS. In the exposed sample, calcium carbonate and calcium sulfate had formed and calcium hydroxide was depleted. Two types of agglomerates of hydrated cement paste were observed. One type consisted of decalcified C-S-H and a combination of ettringite and Cl-AFm phases. The other type consisted mainly of M-S-H. These findings are in line with the observations on long term marine exposed samples in which the formation of a magnesium rich phase at the concrete surface is detected, followed by a sulfate and chloride enriched zone. The knowledge of these phase changes are important to obtain a better understanding of concrete deterioration in marine environment.

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1. Introduction

The phase changes in concrete exposed to sea water are complex due to the presence of a variety of ions in the sea water such as sodium, chloride, magnesium, sulfate, calcium and carbonate ions. These ions can affect the phase assemblage of the concrete in different ways [1–3].

Most studies on the effect of sea water on concrete focus on the ingress of chloride ions in concrete as chlorides pose a threat to the reinforcement by causing pitting corrosion. The chloride ingress from sea water can, however, be influenced by the other ions present in the sea water either by changing the chemistry and/or porosity or even by causing scaling.

In a previous study [4,5] the phase changes in a concrete wall element exposed in the tidal zone of the Trondheim fjord for

10 years were investigated. Fig. 1 shows the elemental profiles¹ inwards from the exposed concrete surface. The chloride level near the exposed surface is low as the chloride peak is preceded by a magnesium enriched zone followed by a sulfate enriched zone. In addition, these zones showed signs of carbonation. This zoned attack has also been observed by Marchand et al. [6], Chabreliet et al. [7], and Jakobsen [8] on marine exposed concrete.

The aim of this paper is to investigate the phase changes in the cement paste in the outermost zone of the concrete exposed to sea water. The limited size of this zone and possible scaling of the concrete surface [8] complicate the investigation of the phases formed. Therefore a novel experimental set-up was designed in an attempt to simulate the conditions in this zone, where a relatively small amount of concrete is in contact with a vast ocean. An excessive amount of sea water was dripped through a well-hydrated powdered cement paste sample during 6 weeks. This test set-up differs in many aspects from real exposure such as for example temperature fluctuations and none the least time. It does however allow detailed investigation of specific phases forming at the concrete surface on a bulk material with a range of techniques. The spatial resolution of the phase changes i.e. the depth from the exposed surface at which these changes form is however not covered in this paper.

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¹ The elemental composition is calculated based on the EDS analysis of an area of 780 × 580 μm. This area comprises both cement paste and aggregates. The Ca content is a measure for the amount of cement paste present as the aggregates in the concrete do not contain Ca. Thus, the elemental changes are plotted relative to the Ca content to take in account the variable presence of aggregates in the analyzed area.

2. Experimental

2.1. Materials

The cement used in the study is a Portland cement type CEM I 42.5 R according to EN197 [9]. The oxide composition of the cement obtained by X-ray fluorescence (XRF) is given in Table 1. The mineral composition calculated according to the Bogue calculation is given in Table 2 together with the Blaine fineness and the specific density of the cement.

The sea water was obtained from the Trondheim fjord. The composition of the sea water determined by inductively-coupled plasma mass spectrometry (ICP-MS) is given in Table 3, as well as the very similar composition of Atlantic sea water [10].

2.2. Preparation procedure for granulated cement paste

Fine ground well-hydrated cement paste was produced according to the following procedure: About 5 liters of cement paste with a w/c ratio of 0.4, without admixtures was mixed with a Hobart mixer. The paste was sealed cured in plastic bags in a water bath at +5 °C for the first 3 days and during the subsequent 4 days it was cured in a water bath at 20 °C. After this the paste was crushed with a jaw crusher and subsequently ground with a rotating disc mill to a maximum particle size of 1 mm. An additional 40% of water by weight of powder mass was added prior to curing the paste for another week at 20 °C. The resulting moist hydrated cement paste was crushed again with a jaw crusher and subsequently manually crushed by a hammer as the high moisture content would clog the disc mill. Thereafter the powdered batches, now looking like “moist sand”, were homogenized and stored for an additional two weeks at 20 °C.

The procedure above was followed in order to: avoid coarsening of the CSH by high curing temperatures; avoid collapsing of the CSH due to drying; minimize carbonation by short transfer times in air; maximize degree of hydration of low w/c paste in a relatively short time.

The paste was characterized by thermogravimetric analysis (TGA). Approx. 400 mg of the hydrated paste was weighed into a 900 µl alumina crucible. The paste was dried at 105 °C while purging with N₂ for 4 h until approx. constant weight. The weight loss measured relative to the initial mass was 38%, which represents the amount of evaporable water present in the paste. The sample

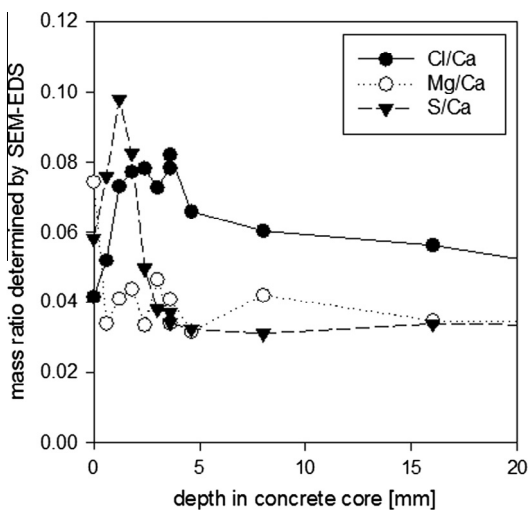


Fig. 1. Elemental profiles inwards from the exposed concrete surface measured by SEM-EDS on a polished sample of a 10 year old concrete wall element exposed in the tidal zone of the Trondheim fjord from [4].

Table 1
Oxide composition of the cement.

Oxide	Content (%)
CaO	61.0
SiO ₂	19.6
Al ₂ O ₃	4.87
Fe ₂ O ₃	3.48
MgO	2.83
SO ₃	3.81
P ₂ O ₅	0.15
K ₂ O	1.11
Na ₂ O	0.51
Free CaO	1.43
Limestone	4.1

Table 2
Mineral composition according to Bogue and physical properties of the cement.

Mineral or property	
C ₃ S	54%
C ₂ S	16%
C ₃ A	7%
C ₄ AF	11%
Blaine	552 m ² /kg
Density	3.15 g/cm ³

Table 3
Concentration of the major elements in the sea water in the Trondheim fjord and the Atlantic Ocean [10].

Concentration (g/l)	Cl	Na	Mg	S	K	Ca
Trondheim fjord	19.4	9.5	1.1	0.86	0.35	0.35
Atlantic Ocean	19	10.5	1.35	0.88	0.38	0.40

was then further heated from 105 °C to 1000 °C with a rate of 10 °C/min while purging with N₂. No considerable carbonation was detected and the amount of evaporable water determined between 105 and 1000 °C was approx. measured to be 21% indicating a degree of reaction of approx. 90%.

2.3. Sea water exposure of cement paste

The dripping test set-up is shown in Fig. 2. Sea water was dripped onto a bed of 50 g hydrated cement paste packed in a cellulose extraction thimble (Whatman™ 603). The time needed

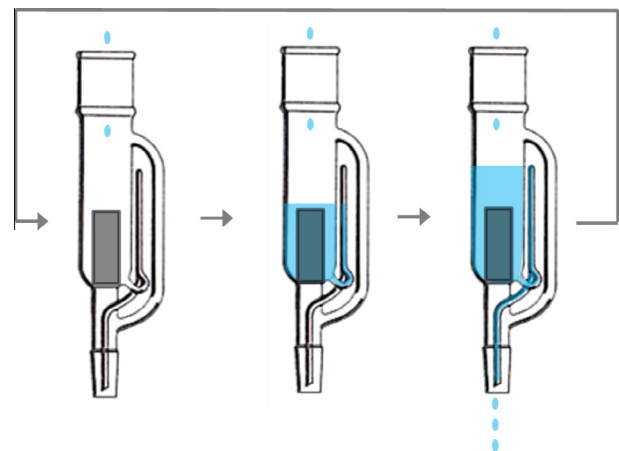


Fig. 2. The set-up of the dripping test with the cement paste sample in the soxhlet extraction chamber.

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