



Resistance of concrete and mortar against combined attack of chloride and sodium sulphate



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ABSTRACT

Marine environments are typically aggressive to concrete structures, since sea water contains high concentrations of chlorides and sulphates. To improve predictions of concrete durability within such environments, it is important to understand the attack mechanisms of these ions in combination.

In this research, the reciprocal influence of Cl^- and SO_4^{2-} was investigated for four mixtures, namely with Ordinary Portland Cement, High Sulphate Resistant cement, and with Blast-Furnace Slag (50% and 70% cement replacement). Chloride penetration depths and diffusion coefficients were measured to investigate the influence of SO_4^{2-} on Cl^- attack. Besides, length and mass change measurements were performed to examine the influence of Cl^- on SO_4^{2-} attack. Since the formation of ettringite, gypsum and Friedel's salt plays an important role, XRD-analyses were done additionally.

It can be concluded that chloride penetration increases when the sulphate content increases at short immersion periods, except for HSR concrete. Concerning the sulphate attack, the presence of chlorides has a mitigating effect.

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1. Introduction

A lot of damage is reported for constructions in marine environments [1–8]. Marine environments are very aggressive, since sea water consists mainly of chlorides and sulphates. Both ions can be very harmful for the durability of concrete structures. However, almost no literature is found about the reciprocal influence. Chlorides affect durability by initiating corrosion of the reinforcement steel, and sulphates by deteriorating the concrete itself. Concrete structures in marine environments mostly have a high economic impact (e.g. bridges, wharfs, piers, tunnels, etc.), so it is important to know the attack mechanisms in detail in order to predict concrete's service life as exactly as possible.

Concerning concrete deterioration due to chlorides, it is important to notice that corrosion will only be initiated by the free chlorides and not by the fraction that is chemically bound to the cement hydrates or physically adsorbed at the pore walls. So, chloride binding is a significant factor related to reinforced concrete durability for three reasons [9]: reduction of the free chloride concentration in the vicinity of the reinforcing steel will reduce the risk of corrosion; chloride binding will delay the chloride penetra-

tion; formation of Friedel's salt results in a less porous structure and slows down the transport of Cl^- -ions. Friedel's salt ($3\text{CaO}\cdot\text{Al}_2\text{O}_3\cdot\text{CaCl}_2\cdot 10\text{H}_2\text{O}$) is the result of chemical binding between chlorides and C_3A . Chemical binding can also occur between chlorides and C_4AF . Besides, physical binding occurs due to interaction with CSH. Factors influencing binding are [10–13]: chloride concentration, cement type, cement replacement, cation, alkalinity, temperature, water-binder factor, etc.

Cement replacement by Blast-Furnace Slag (BFS) seems to have a positive influence on the resistance of concrete against chloride penetration. BFS concrete is already used in big marine structures because of the low hydration heat [14]. Besides, partial replacement of Ordinary Portland Cement (OPC) by BFS is considered as a promising way to improve concrete's service life when the prediction is based on chloride penetration.

In general, it is assumed that BFS concrete is able to bind more chlorides. This is attributed to increased Friedel's salt formation. Replacement of 70% of the cement by BFS is the most suitable in view of chloride binding [15]. On the other hand, former research does not always show a higher chloride binding capacity of BFS concrete compared to OPC concrete [12,16]. Besides, when cured properly, BFS concrete possesses a finer pore structure resulting in a limited penetration depth of free chlorides and consequently a reduced risk for chloride initiated corrosion in comparison with

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OPC concrete. The chloride diffusion coefficient of BFS concrete reaches lower values than the coefficient of OPC concrete [16–20].

The other main attack mechanism in marine environments is external sulphate attack. This occurs when water contaminated with sulphates penetrates into the concrete by means of diffusion or capillary suction. Sulphates are mostly found in the form of sodium sulphate (Na_2SO_4) or magnesium sulphate (MgSO_4). The cation associated with SO_4^{2-} has an influence on the attack mechanism and the resulting deterioration [21]. Sodium sulphate attack will result in expansive reaction products while magnesium sulphate attack will result in reduction in strength. In current paper, the influence of Na_2SO_4 and the combined attack of NaCl and Na_2SO_4 is examined. Because of the different attack mechanism, the influence of MgSO_4 and the combined attack of NaCl and MgSO_4 will be discussed in following papers.

Once penetrated into the concrete, sulphates react with the lime formed during the hydration process of the Portland clinker. One of the reaction products is calcium sulphate or secondary gypsum. This calcium sulphate reacts with hydrated calcium aluminates, C_3A , and forms ettringite ($3\text{CaO}\cdot\text{Al}_2\text{O}_3\cdot3\text{CaSO}_4\cdot32\text{H}_2\text{O}$). Gypsum leads to reduction of stiffness and strength, expansion and cracking and eventually to transformation of the material into a mushy and non-cohesive mass. Ettringite has the ability to swell strongly, which results in a densification of the microstructure followed by internal stresses that lead to cracking and destruction of the concrete [22]. The factors influencing the rate of external attack are: the quantity of sulphate ions, the possibility of the sulphates to penetrate into the concrete and the volume of C_3A in the cement and the type of cement used to make the concrete.

Temperature changes can have an influence on the sulphate attack mechanism as well. At low temperatures and in presence of soluble carbonate and reactive silicate, thaumasite ($\text{Ca}_3\text{Si}(\text{CO}_3)(\text{SO}_4)(\text{OH})_6\cdot12\text{H}_2\text{O}$) can be formed. This is not expansive but lowers the strength and has a negative influence on the microstructure. It is generally assumed that thaumasite is only formed at temperatures below 15°C . However, some researchers also found thaumasite at temperatures higher than 15°C [23–25]. In the current paper, the influence of temperature fluctuations on the sulphate attack mechanism and on chloride penetration was not examined; the tests were performed at 20°C . Nevertheless, in West-European marine environments sea water temperatures are often below 10°C while in the Middle East and South East Asia sea water temperatures can be higher than 30°C . According to Aköz et al. [26] raising temperatures of sodium sulphate solutions in the range of 20 – 40°C have a beneficial influence on resistance of mortar against sulphate attack. However, they are not able to determine the dominant factor affecting the performance of mortar in a sodium sulphate solution at 40°C . On the other hand they observed that raised temperatures until 40°C could have negative effects on mortar resistance in magnesium sulphate solutions. They assume that this is due to decalcification of C-S-H to M-S-H , which leads to a porous structure. Concerning the influence of fluctuating temperatures on chloride diffusion, some researchers [7,27,28] found that chloride diffusion rises with increases in temperature.

The use of cement replacement materials tends to improve the resistance against sulphate attack [29]. However, when BFS is used and the samples are exposed to magnesium sulphate, deterioration exceeds that observed in Portland cements [30]. In that case, the calcium hydroxide is consumed by the pozzolanic reaction, so the sulphates and the magnesium ions will react directly with the C-S-H due to the absence of $\text{Ca}(\text{OH})_2$, resulting in a cohesionless M-S-H [31]. Furthermore, BFS concrete which is partially immersed in a sulphate solution will show severe deterioration in the upper parts of the concrete in contact with air due to salt crystallization [32,33]. This is in contrast with the situation with

complete immersion. When the concrete is completely immersed in the sulphate solution, BFS concrete will have a higher resistance than OPC concrete.

Generally, the C_3A -content of the cement plays a major role in the attack mechanism of sulphates and in the binding behaviour of chlorides. When both ions penetrate the concrete together, C_3A -binding will definitely influence this multi-ion transport. In literature a lot of papers are found on individual chloride and sulphate attack. Nevertheless, only limited literature is available concerning combined environmental attack of chlorides and sulphates. Two groups of papers are found: firstly, the papers investigating the effect of chlorides on sulphate attack and secondly the papers investigating the effect of sulphates on chloride attack.

According to Al-Amoudi et al. [34] there are three possible schools of thought concerning the influence of chlorides on sulphate attack: (1) the sulphate attack mechanism is intensified, (2) sulphate attack is mitigated, and (3) the influence is insignificant. In their research, they examined the role of chloride ions on expansion and strength reduction due to sulphate attack by adding high volumes of sodium chloride, namely 15.7% Cl^- , to mixed sodium and magnesium sulphate solutions. The sulphate concentration amounted to 2.1% SO_4^{2-} in which the sodium and magnesium sulphate were proportioned to provide 50% of the sulphate concentration from each of them. They found that the deterioration is more severe for specimens immersed in a pure sulphate solution than in a combined sulphate–chloride solution. Concerning the influence of the cement type, they concluded that the deterioration due to sulphate attack is not very different in cements with varying C_3A contents in the range of 3.5 – 8.5% . Besides, Al-Amoudi et al. also found that replacement of OPC by BFS has only a marginal beneficial effect.

Also Abdalkader et al. [35] investigated the influence of chloride on the performance of mortars subjected to sulphate exposure. Their tests were done at 5°C with 6 g/l SO_4^{2-} magnesium sulphate solutions diluted with 5 g/l Cl^- sodium chloride. They observed that sulphate attack is more severe when the samples are immersed in a combined solution compared to those stored in a pure sulphate solution, since the presence of chlorides accelerates damage caused by thaumasite.

Based on the findings of Zuquan et al. [36] the influence of chlorides on the sulphate attack mechanism prolong the periods of the attack process, which results in a less severe deterioration. Their tests were performed at 20°C with a combined solution of 5% Na_2SO_4 and 3.5% NaCl .

Concerning the influence of sulphates on chloride attack, De Weerd and Geiker [37] concluded that chloride ions penetrate much deeper into the concrete compared to other elements originating from the seawater e.g. Mg^{2+} , S^{2-} and Na^+ which have, approximately, a constant concentration from 20 mm inwards. This should mean that the binding competition between Cl^- and SO_4^{2-} only occurs in the outermost layers. Nevertheless, due to the binding of chlorides, more free sulphate ions will be present and they are able to penetrate deeper into the concrete. However, data found by Brown and Badger [22] indicate that Friedel's salt converts to ettringite in the presence of sodium sulphate solution. Thus, ettringite is the stable phase under these conditions. This should mean that more free chlorides will be present in the concrete. Based on the findings of Zuquan et al. [36] the influence of sulphates (in the form of Na_2SO_4) on chloride diffusion is dependent on the exposure period, namely at early exposure periods the presence of sulphate decreases the concentration of chlorides and the chloride diffusion coefficient. But at later exposure periods, the presence of sulphates in combined solutions, cause an increased ingress of chloride.

In this research, the influence of the multi-ion transport on the proper attack mechanisms was examined. Accelerated tests were

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