

# Improved durability of cement mortars exposed to external sulfate attack: The role of nano & micro additives



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## ABSTRACT

External sulfate attack (ESA) is a critical issue which may turn into a big concern about the durability and service life of structural elements get into contact with high sulfate environments. Especially when improper concrete mixture materials and design parameters are selected, structural elements exposed to sulfate attack might be significantly deteriorated. In this experimental work, the effects of various micro and nano cementitious materials on the long-term durability of mortars against ESA were studied. For this purpose, nano (colloidal) silica (NS), micro silica (MS), fly ash (FA) and ground granulated blast-furnace slag (GGBS) were selected with various replacement ratios. All the samples were kept in 5% sodium sulfate solution for approximately 3 years and the expansions caused by sulfate attack were monitored. Additionally, at the end of 3 years of exposure, residual flexural strength, compression strength and ultrasonic pulse velocity (UPV) of mortars were determined and their results were compared with the reference samples which do not contain mineral additives and cured in water. For the micro-structural evaluations, XRD analysis was performed. Results have shown that significant improvement against sulfate attack was achieved by using the additives mentioned above by properly selecting its dosage. Within the limits of this work, mineralogical analysis via XRD tests have proved that decomposition of the sulfate exposed reference samples which do not contain any mineral additives was not caused by ettringite formation, but gypsum formation.

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## 1. Introduction

It is well-known that, one of the ways of enhancing concrete durability against any deleterious external attacks is to improve its permeability properties. For this purpose, besides lowering water to cement ratio of concrete mixture, the use of pozzolanic mineral additives is another effective intervention which improves micro-structural properties of cement based materials by means of physical and chemical remediation. Moreover, the use of such pozzolanic “by-product” mineral additives in concrete allows engineers to replace “Portland cement” with these materials. Two important outcomes of such replacement are the reduced cement consumption and depletion of “by-product” materials. It is well-known that the cement industry contributes approximately 5% to global CO<sub>2</sub> emission (Worrel, Price, Martin, Hendriks, & Meida, 2001). Thus, the use of such materials is important from the point view of both environmental concerns and sustainable structures.

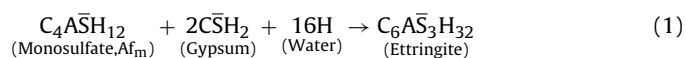
Among so many durability concerns, “sulfate attack” is one of the important durability problems for concrete structures. This problem, accordingly, has become a critical focal point for researchers working on science of concrete technology (Atahan & Dikme, 2011; Collepardi, 2003; Santhanam, Cohen, & Olek, 2002; Santhanam, Cohen, & Olek, 2003; Skalny et al., 2002). Although it is not a new issue, in the last 20 years, several sulfate attack related deteriorations and damages on structural elements has been reported in all over the world (Drimalas, Clement, Folliard, Dhole, & Thomas, 2011; Lee, Cody, Cody, & Spry, 2005; Ma, Gao, Byars, & Zhou, 2006; Mingyu, Fumei, & Mingshu, 2006; Rahman & Bassuoni, 2014; Sahu & Thaulow, 2004; Thomas, Folliard, Drimalas, & Ramlochan, 2008). Such structures faced with sulfate related damages are dams, railroad ties, concrete roads, bridge columns, RC column foundations, concrete piles, highway/railway tunnels, floor slabs and more. Depending on the type of sulfate attack and related mechanisms, the common damage types observed on these cases are spalling, swelling, cracking, expansion, softening and surface scaling of concrete elements.

Sulfate attack can be described as sequentially growing complicated chemical reactions between the hydration products of cement and sulfate ions available in the environment. These

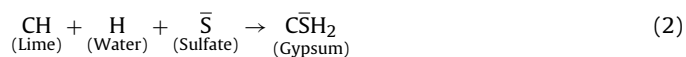
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reactions, ultimately, may yield to the decomposition of hardened cement chemistry. As a well-known information, there are different ways for sulfate ions to enter into concrete and trigger the undesirable chemical reactions. Depending on the way by which the sulfate ions are supplied, the type of sulfate attack can be classified as “External” or “Internal”. There are so many forms of sulfate attack having different deterioration mechanisms that are known of (Skalny et al., 2002). For example, in case of “magnesium sulfate” attack, disintegration of concrete is due to the exchange between the calcium ions in tobermorite gel and  $Mg^{+2}$  ions in the solution to form M–S–H gel which has weak properties. On the other hand, in case of “sodium sulfate attack”, deterioration of concrete is caused by the reactions of sulfate ions with hydration products of cement. Namely, in such a case, ettringite ( $3CaO \cdot Al_2O_3 \cdot 3CaSO_4 \cdot 32H_2O$ ) formation is considered to be the main reason for damage through the following reaction:



Formation of ettringite crystals in hardened media results an increase in solid volume (expansion) which leads concrete to be damaged. In this reaction, the amount of Af<sub>m</sub> is related to the amount of C<sub>3</sub>A content in cement. On the other hand, some researchers have not been able to reconcile the relationship between the amount of expansion and ettringite formation (Odler & Gasser, 1988; Santhanam et al., 2002, 2003). It is also mentioned in the literature that due to its low solubility, ettringite can form at relatively “low” sulfate concentrations (Skalny et al., 2002). Gypsum formation, on the other hand, is believed to be the primary reaction product of sulfate attack at “high” sulfate ion concentrations (Santhanam, Cohen, & Olek, 2001). In practice, sulfate concentration may not reach at higher values as we use at “accelerated” laboratory tests. It can be concluded that, because of this reality in application, ettringite-formation related expansion is more experienced than gypsum-formation related expansion. Gypsum related deterioration would result in the surface softening, mass loss, strength loss and expansion of concrete element. Mehta et al. (Mehta, Pirtz, & Polivka, 1979) investigated the performance of concrete specimens exposed to sulfate attack prepared with C<sub>3</sub>S (alite) cements. Although the cement they used was C<sub>3</sub>A free, after a long period of sulfate exposure, they observed significant spalling and strength loss of samples. In the light of X-ray diffraction studies, however, the damage was attributed to the gypsum formation. As long as sulfate ions in the pore solution are available, the amount of gypsum formation is closely related to the amount of portlandite ( $Ca(OH)_2$ ), which is the main reaction products of C<sub>2</sub>S and C<sub>3</sub>S.



It is possible to find recommendations for improving the resistance of concrete to sulfate attack in the structural codes and standards (ACI 201.2R-08, 2008; CSA A23-1/A23-2, 2014; EN 206-1, 2013). In their recent publication, these codes and standards are well elaborated by Rahman et al. (Rahman & Bassuoni, 2014). In summary, it is possible to improve the resistance of concrete against sulfate attack by enhancing its physical and chemical resistance by lowering water to cementitious material ratio ( $\leq 0.40$ ), using sulfate resistance Portland cements with low C<sub>3</sub>A content ( $< 5\%$ ), replacing cement with pozzolanic cementitious materials such as fly ash, GGBS, metakaloin and silica fume and, using adequate cement content in the mixtures. Besides these precautions, of course, adequate compaction and curing of concrete must be perfectly performed.

For many years, mineral additives such as fly ash (FA), ground granulated blast furnace slag (GGBS) and micro silica (MS) are being utilized worldwide in concrete mixture designs. Their positive contribution to the hardened concrete properties such as strength and

durability has been proved (Akoz, Turker, Koral, & Yuzer, 1995; Lee, Moon, & Swamy, 2005; Torii, Taniguchi, & Kawamura, 1995). Nano silica (NS), as a nano-technological product, has relatively new applications in concrete technology (Bergna & Roberts, 2006; Otterstedt & Greenwood, 2006; Querica, Hüsken, & Brouwers, 2012; Said, Zeidan, Bassuoni, & Tian, 2012; Senff, Labrincha, Ferreira, Hotza, & Repette, 2009). It consists of amorphous silicon dioxide with a particle size at nano scale. Notably high purity and ultra-high specific surface area ( $> 80,000 \text{ m}^2/\text{kg}$ ) are the most important characteristics of this nano technological material. Since the effectiveness of a pozzolanic reaction is proportional to the amount of surface area of amorphous silica particles, its efficiency is obvious. Moreover, its filling effect should also be considered as an additional benefit. In this experimental work, the effects of “external” sodium sulfate attack on the hardened mortar samples designed with various mineral admixtures such as NS, MS, FA and GGBS were investigated.

## 2. Experimental study

For the production of mortar samples, a natural sand sample having a density of 2.65 and maximum size of 4 mm was used. Chemical analysis has revealed that the sand contains ignorable amount of water soluble chloride (0.004% by wt) and sulfate (0.003% by wt). In preparation of mortar samples, Type I Portland cement with C<sub>3</sub>A content of 7.6% was used. Chemical and physical properties, as well as the mineralogical composition of the cement used are given in Table 1. The mineral additives used in the mortar mixtures were MS, NS, FA and GGBS. Some of the physical and chemical properties of them are given in Table 2.

Within the limits of this experimental work, for each mineral additive, three different replacement ratios by weight of cement were selected: 2%, 4% and 6% for NS, 6%, 9% and 12% for MS, 15%, 30% and 45% for FA and, 20%, 40% and 60% for GGBS. Additionally, in order to display the physical, mechanical and micro structural changes caused by sulfate attack, “reference” samples which do not contain any mineral additives were also produced. It should be noted here that the nano silica sol used has a solid content of 50% (50% SiO<sub>2</sub> and 50% water by weight). Declared replacement ratios for NS denote net nano silica amounts. Mixture proportions of the mortars were selected as water/sand/cementitious material ratios of 0.5/2.75/1, respectively. Flow tests were performed as described in ASTM C1437 (2007) and flow of mortars were kept constant at  $15 \pm 1$  cm. The mineral additives used have different replacement ratios and physical characteristics, thus, in order to keep the workability at a constant value, a high range water reducer was used if necessary.  $40 \times 40 \times 160 \text{ mm}^3$  molds were used and the mixtures were compacted on a vibration table. Samples were kept in  $22 \pm 2^\circ \text{C}$  and 60% RH laboratory environment for the first 24 h and then cured in  $22 \pm 2^\circ \text{C}$  lime saturated water for an additional period of 2 weeks.

Most common methods for monitoring the physical and mechanical changes on samples exposed to sulfate environment are the length change and strength loss caused by secondary mineral formations inside the samples. After the initial water curing described above, first comparator readings were recorded as reference readings using a length comparator with a precision of 0.005 mm. In order to monitor the length changes caused by sulfate attack, the procedure described in ASTM C1012 (2015) has been followed. For this purpose, all the samples, including the reference samples, were immersed in 5% Na<sub>2</sub>SO<sub>4</sub> solution. Sulfate solution in which the samples were stored has been changed every 2–3 weeks. In parallel with the samples stored in sulfate solution, one group of reference samples which do not contain any mineral additives were stored in water as well.

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