Pergamon

Cement and Concrete Research 31 (2001) 845–851

CEMENT AND CONCRETE RESEARCH

Review

Sulfate attack research — whither now?

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Received 29 November 2000; accepted 15 March 2001

Abstract

Sulfate attack research is at a critical stage. In spite of meaningful advances in the past few years, this problem is still not well understood. Due to its complicated mechanism, the reaction between cement hydration products and sulfate-bearing solutions manifests itself in a variety of ways. In order to provide adequate means for selection of materials for concrete exposed to such aggressive environments, additional research is necessary to further clarify the interaction between concrete and sulfate-bearing solutions. Specifically, the role of the cation in the sulfate solution, and the effects of formation of various products like gypsum, ettringite, and thaumasite, on the extent of damage need to be investigated. The available testing methods for sulfate attack have been subject to some criticism lately. Although these test methods can give an indication of the mechanisms involved in sulfate attack, prediction of field performance using lab studies is difficult. Efforts are needed to introduce appropriate changes in the tests in order to obtain field-like conditions in the laboratory. Combined with good monitoring methods, this would enable the prediction of service life of structures exposed to sulfate solutions. Recent advances in nondestructive testing techniques can be applied to the task of monitoring field structures, although there is a significant effort necessary to calibrate these methods for sulfate attack-related scenarios. In order to produce efficient concrete designs for service in aggressive environments, it is imperative to develop reliable models. Modeling can help in selecting the appropriate materials and their proportions, as well as in determining service life parameters. As a first step towards modeling, critical parameters, which serve as an indicator of deterioration, need to be recognized and established. This paper discusses these issues, and cites some interesting recent developments. Finally, some recommendations for future studies are provided.

Keywords: Sulfate attack; Expansion; Pozzolans; Ettringite; Modeling

1. Introduction

Sulfate attack is an important concrete durability and serviceability concern. The complicated nature of the attack has led to the formulation of numerous theories regarding its mechanism, some of which are conflicting. Interest in sulfate attack research has been rising in recent years owing to numerous litigations in Canada and the United States. There is an ongoing effort to develop a better understanding of the issue in order to develop more efficient solutions.

Several years ago, Cohen and Mather [1] presented the needs for a systematic approach to conducting research on sulfate attack. Their work was focused on analyzing the existing standards and test methods for sulfate attack, and their shortcomings. They proposed various criteria for evaluation of the problem, for obtaining a better understanding of the process, and for developing more reliable mechanism-based standard tests.

During the last decade, research has shifted towards analyzing sulfate attack from a materials science perspective. Scanning electron microscopy has been used successfully as an investigative tool to explain the features of the sulfate attack and its mechanism [2–4]. Although the knowledge and research has improved, there are still areas that are in need of further development.

Some critical issues regarding tests and specifications for sulfate attack were addressed by Skalny and Pierce [5] in their recent paper. It was suggested that the current testing procedures are not indicative of the field situation, and that there has to be an effort to develop failure criteria and parameters to enable prediction of sulfate attack in field structures. The authors also stated that although the current knowledge is sufficient to combat sulfate attack, there are gaps in translating this knowledge to field practice, as well as failures to apply correctly the available knowledge.

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PII: S0008-8846(01)00510-5

Hime and Mather [6] also indicated that several questions related to chemistry of sulfate attack have not been answered satisfactorily. For instance, they discussed the need to differentiate between sulfate attack, which is chemical, and internal expansion of concrete due to dehydration and rehydration of salts such as sodium sulfate, sodium carbonate, or magnesium sulfate, where the SO_4 ion does not play any active part. The authors also stressed the importance of specifying the type of cation accompanying the SO_4 ion in the sulfate solution. They stated that the distinguishing features of each of the sulfate reactions needed to be determined in order to prevent misdiagnosis and improper or unnecessary repair.

The objective of this paper is to address the critical issues and report recent developments related to sulfate attack research. This article is divided into four sections, pertaining to (1) the sulfate attack mechanism, (2) test methods, (3) field monitoring methods, and (4) development of modeling criteria. Issues involved are discussed, and suggestions for future research are provided.

2. Sulfate attack mechanism

The reactions involved in sulfate attack have been reviewed by Cohen and Bentur [7]. The attack by sulfate solutions produces diverse effects, and the associated mechanisms can be somewhat clarified by isolating these effects and studying each one in detail. It must be understood that combination of two or more effects could produce complications, which cannot be fully understood using results from individual tests.

2.1. 'Sulfate attack'?

The use of the term 'sulfate attack' has promoted most of the research efforts to focus on the effects of the SO_4 ion alone. In reality, attack by different sulfate solutions, such as the ones containing Ca, Na, Mg, and Fe as the cation, proceeds differently with respect to the mechanism and the type of distress caused. It is this confusion which leads to improper selection of materials for situations where there is a perceived danger of exposure to aggressive sulfate-bearing solutions. For example, use of low C_3A cements, which is considered to provide resistance to sodium sulfate attack, can be detrimental when the solution contains magnesium sulfate or sulfuric acid. In the latter case, the prevailing low pH conditions lead to a direct attack on the calcium silicate hydrate gel (C-S-H). It is important that researchers, engineers, and contractors understand the implications involved in order to design and construct a durable structure.

2.2. Effects of ettringite, gypsum, and thaumasite formation

Ettringite and gypsum are the primary products of the chemical reaction between a sulfate-bearing solution and cement hydration products. This fact has been recognized for a long time, although considerable controversy exists in identifying the significance of each of these products in the ultimate deterioration of concrete undergoing sulfate attack.

Failure by expansion as a result of sulfate attack has traditionally been attributed to the formation of ettringite [8–10]. Some researchers have not been able to detect a strong correlation between expansion and ettringite formation [10]. However, C_3A is known to exist in at least four different crystalline forms in cement, which differ in reaction rates but not in the total amount of ettringite formed [11]. Thus, the stage at which expansion is measured is critical.

Gypsum is the primary reaction product of sulfate attack at high sulfate-ion concentrations (>8000 ppm SO_4^{2-}) [12]. Ettringite is not stable in low-lime environments when the pH falls below 11.5–12 [12]. At this low pH range, ettringite could decompose to form gypsum. Even though the formation of gypsum is important, there is no clear documentation of the nature of disruption caused by gypsum [13,14].

The study of gypsum formation is often not emphasized enough in traditional sulfate attack research. This explains the choice of low C_3A cements universally as a protection against sulfate-bearing solutions. There are two aspects of gypsum-related deterioration that need to be addressed: (a) surface softening, and (b) expansion. Softening of the concrete surface and mass loss due to spalling are the commonly observed modes of damage in field structures subjected to sulfate attack, but these effects are not usually quantified [1]. Standards should be developed for quantification of the surface-softening phenomenon including mass loss and depth of damage. A hardness method could be used to detect damaged areas, and quantify the amount of damage. These methods are discussed briefly in a later section of this paper.

In order to study whether gypsum formation causes expansion, it is important to use a C_3A-free system to avoid the effects of ettringite. In a study of the properties of alite (C_3S) cements by Mehta et al. [15], the performance of concrete specimens prepared with alite cements under sulfate exposure was investigated. Long-term exposure caused significant spalling and loss of strength in alite concrete as compared to concrete prisms prepared with an ASTM Type V cement. This phenomenon was attributed to gypsum formation, which was detected by X-ray diffraction studies. However, expansion measurements were not made in this study.

In a study by Tian and Cohen [16], the effect of gypsum formation during sulfate attack on the resulting expansion of C_3S paste and mortar bars was investigated. The quantities of gypsum formed in various mixtures were compared by the relative heights of XRD peaks. It was found that C_3S mortars were highly susceptible to expansion, and registered a high degree of gypsum formation. This effect was reduced when the C_3S was partially substituted with silica fume.

Using thermal analysis techniques, the amounts of various phases as a mass percent of hydrated cement paste can be determined. These techniques can be used to monitor the amounts of gypsum and ettringite formed during the reaction. Studies correlating measured physical properties, such as expansion and mass change, with the amounts of various phases (gypsum, ettringite, and brucite) formed are currently underway at Purdue University. Quantifying the role of gypsum and ettringite can help in identifying the optimum cost-effective solutions to mitigate sulfate-related damage. For example, crystal growth inhibitors such as sodium citrate (which inhibits the crystal growth of gypsum) can be used as a possible additive to reduce gypsum-related damage [17].

Thaumasite ($CaCO_3 \cdot CaSO_4 \cdot CaSiO_3 \cdot 15H_2O$) is formed during sulfate attack at low temperatures (0°C to 5°C). Formation of thaumasite occurs as a result of the reaction between C-S-H and SO_4^{2-}, CO_2 or CO_3^{2-}, and water. Thaumasite could also form from ettringite with which it could enter into a solid solution [18]. Detection of thaumasite by XRD is difficult because its pattern is similar to that of ettringite [19]. Thaumasite formation, which can be accompanied by formation of brucite and secondary gypsum, consumes C-S-H, and causes a degradation of the structure of the hydrated paste.

Thaumasite-related deterioration has been identified in many historic buildings in cold climates, where hydraulic cement-based mixtures with high water to cement ratio were used for restoration purposes [20]. Numerous other field studies have also indicated the formation of thaumasite in sulfate environments. However, until recently thaumasite has been regarded as a phase that forms only under exceptional circumstances (i.e., at low temperatures — 0°C to 5°C). Thaumasite has not been studied extensively because most of the reported sulfate attack cases have been from semi-arid environments, where the prevalent warm conditions are not considered conducive for the formation of thaumasite. With the increasing use of limestone dust as filler, and also limestone aggregates in concrete, a ready source of CO_3^{2-} is available within the concrete for the formation of thaumasite, whenever low temperature conditions prevail. Recent sulfate attack cases from Southern California [4], where low temperatures are unlikely, show evidence of thaumasite formation, suggesting that it is not necessary to have a low temperature for this phase to form. Thus, there is no clear understanding of the mechanism of thaumasite formation. Identification of the dangers of thaumasite formation during sulfate attack is, therefore, important, and any research on sulfate attack should include a study of the effect of different temperatures, as well as carbonation.

2.3. Magnesium ion attack

Bonen and Cohen [21] investigated the effect of magnesium sulfate solutions on portland cement pastes. They suggested that the attack by magnesium ion primarily leads to the formation of a brucite layer at the exposed surface. Because of the low solubility of brucite, the penetration of Mg^{2+} beneath the brucite layer into the interior of the paste specimen is restricted. It should be noted, however, that brucite formation consumes a high amount of CH supplied by the hydrated paste. Once the available CH is depleted, the pH of the pore solution gets lowered. In order to maintain its stability, the C-S-H can release CH to the surrounding solution, increasing the pH. This process ultimately contributes to the decalcification of the C-S-H, and the loss of the cementitious structure. In the advanced stages of attack, the Ca ion in the C-S-H can be completely replaced by the Mg ion, leading to the formation of magnesium silicate hydrate (M-S-H), which has been reported to be non-cementitious [22].

When mineral admixtures such as silica fume are used, the pozzolanic reaction consumes calcium hydroxide. Thus, the attack by magnesium sulfate solutions can proceed quickly to the stage where decalcification of the C-S-H gel starts occurring. This leads to a poor performance of the systems containing silica fume compared to Type I cement [3,7]. However, the reduction in permeability and refinement of pore structure with the use of mineral admixtures can often overcome this negative effect. It is, therefore, essential to determine critical dosage levels of mineral admixtures to maximize their benefits, and minimize the deleterious effects of magnesium sulfate attack.

3. Development of testing methods

The reliability of the current ASTM test methods for assessing sulfate attack (ASTM C1012 [23] and ASTM C452 [24]) has been questioned for a long time. According to numerous investigators [1,5,25], the current standards tend to overlook some important issues that can be critical with respect to the performance of field structures exposed to sulfate attack. These issues are discussed in detail below.

Investigations by Mehta [26] and Brown [27] suggest that continuous immersion of test specimens, as suggested by the ASTM standards, is not a valid representation of the field situation since the pH of the attacking solution rapidly changes from neutral (~ 7) to basic (~ 12) due to a leaching of the alkalis from concrete into the surrounding sulfate solution. Furthermore, the sulfate concentration in the solution decreases during continuous immersion. This could lead to a discrepancy in using laboratory experiments to predict field behavior. In general, laboratory specimens subjected to continuous immersion are able to withstand the attack longer than the corresponding field exposure specimens. This is because the field specimens are subjected to atmospheric effects such as wetting and drying, in addition to the attack by a sulfate solution of an almost constant concentration.

Mehta [26] proposed a new test method in which the pH of a sodium sulfate solution was maintained at a constant level ($\sim$6.2) by means of a continuous titration with H_2SO_4. The pH was continuously monitored using a pH controller. Zero C_3A cements were found to be as poorly performing as cements with some C_3A. A considerable amount of gypsum was detected by X-ray diffraction. Mehta concluded that the effect of controlling pH in an acidic range was to drive the mechanism of the reaction towards gypsum corrosion. According to Mehta, the procedural details were found to be adequate enough to yield reproducible results. However, several other researchers, including Mather [11], believe that this test is not appropriate since an acid attack mechanism, rather than sulfate attack, is introduced by the use of H_2SO_4.

Brown [27] used a similar experimental set-up to study the effect of controlling the pH during sulfate attack. Three different pH levels were used (6.0, 10.0, and 11.5) and compared to the uncontrolled, continuous immersion experiment. He found that as the pH of the solution was lowered, the resistance to sulfate attack, as measured by linear expansion and cube strength of mortars, decreased. The rates of linear expansion and loss of cube strength were relatively independent of the solution pH. This test can help improve the understanding of field exposure conditions, though the reproducibility has not been tested extensively.

All the tests described above monitor the changes due to the chemical processes that may be involved in sulfate attack. Translating the effects observed in the laboratory to field situations is a challenging task because a complex interplay of physical and chemical factors occurs in field conditions. The added challenge to testing is the simulation of conditions like drying and wetting cycles, and temperature and humidity variations, among others. The US Bureau of Reclamation conducted a large-scale study lasting 20 years [28] in which the length change of concrete cylinders exposed to wetting and drying cycles (accelerated testing) was compared to that of cylinders subjected to continuous immersion in a sodium sulfate solution. As a conclusion, it was estimated that 1 year of accelerated testing equaled 8 years of continuous immersion in the solution. The researchers, however, did not identify the mechanisms causing failure.

In addition to the already discussed pH issue, two more critical considerations that the current standards do not account for are the temperature at which the test is conducted, and the concentration of the solution. The issue of thaumasite formation, as discussed in Section 2.2, outlines the need to understand the effect of changing temperature on the reaction mechanism and chemistry. Study of the attack at various temperatures could also yield valuable information about the activation energy of the reaction. This data can help in designing accelerated testing methods.

According to Biczok [12], the mechanism of reaction changes when the concentration of the solution changes. For attack by sodium sulfate solution, at low concentration of sulfates (< 1000 ppm SO_4^{2-}), the primary product deposited is ettringite, while at high concentrations (>8000 ppm SO_4^{2-}) gypsum is the main product. In the intermediate range (1000–8000 ppm SO_4^{2-}), both gypsum and ettringite are observed. In magnesium sulfate attack, ettringite production is observed at a low concentration (< 4000 ppm SO_4^{2-}), a combined ettringite and gypsum formation is observed at an intermediate concentration (between 4000 and 7500 ppm SO_4^{2-}), and magnesium corrosion dominates at high concentrations (>7500 ppm SO_4^{2-}). Most of the work on effect of sulfate concentration was done before the 1960s. The effect of varying concentrations becomes critical especially in field structures where the same concrete element may be attacked in different regions by different concentrations of sulfates, due to development of drying fronts, wetting fronts, etc. Thus, the aspect of varying concentrations requires further studies.

Efforts aimed at improving test methods and standards for sulfate attack have been made in the past few years. Clifton et al. [25] from the National Institute of Standards and Technology (NIST) presented a comprehensive review of the test methods, and provided recommendations for improving the current standards. The creation of a new rapid standard test for determining the intrinsic reactivity of cements with sulfates was proposed, which would take into account the constant pH type of test (described earlier), as well as variations in specimen shape and size, and curing regimes. The need for developing a standardized method for service life design of concrete, in which sulfate attack is one of the many degradation processes, was also recognized.

Laboratory indicators of sulfate damage can be applied to the monitoring of field structures, with some experimental modifications. The next section explores the needs for developing monitoring methods in the field.

4. Monitoring field structures damaged by sulfate attack

The methods for condition surveys of concrete structures, which are being exposed to sulfates during service, need some development. With the improvement of NDT methods, on-site determination of properties is becoming quicker and easier. Appropriate analyses using the results from these methods can be used to predict the remaining service life of concrete. Such analyses can also help in deciding whether the structure needs repair or even complete replacement.

A recent study by Ju et al. [29] outlines the use of surface hardness tests and ultrasonic measurements to study unattacked and deteriorated structures. In their study, cores were taken from affected structures for investigation by ultrasonic pulse velocity techniques. Obtaining reliable results from pulse velocity measurements requires access to the structure from two opposite sides, which necessitates the use of cores if access to both sides of the structure is not available. With experimental modification [30] and using sensitive transducers, the pulse velocity technique can be used for one-sided measurements. However, the use of this technique to quantify the depth of degradation (that results from the attack by sulfates) needs to be studied in detail, with sufficient data for calibration.

The spectral analysis of surface waves technique is used extensively to study the stiffness variations of semi-infinite layered structures such as pavements [31]. Recent advances in this area have shown that this method can probably be used on finite-sized concrete structures when the concrete quality is not consistent throughout the depth of the structure [32]. Sulfate attack produces weak zones near the surface of the concrete, and using the surface waves method may help to quantify the extent of damage.

Weakened surface zone qualities can also be detected by hardness measurements. Ju et al. [29] used the Schmidt rebound hammer to perform hardness tests. They concluded that despite all the limitations and uncertainties of using the rebound hammer test, it could be used to provide valuable information about the relative qualities of concrete in a structure. Good training and adequate calibration of the technique are necessary to obtain reliable results using surface hardness tests.

Results from laboratory tests and field investigations can be effectively combined to obtain parameters to predict the service life of structures exposed to sulfates. The efforts of various researchers to develop such modeling parameters are discussed in the next section.

5. Parameters for modeling sulfate attack

In an attempt to develop all-encompassing specifications for sulfate resistant concrete, Dunstan [33] outlined the concept of a sulfate resistance factor (F_{SR}). This quantity incorporates several factors pertaining to the cement chemistry (i.e., the amount of C_3A and C_4AF in the cement), water to cementitious materials ratio, quantity of pozzolan or blended material used, and the concentration of sulfates in the ground water or soil. F_{SR} was correlated to service life of the structure. Dunstan also identified other parameters that were not considered in the development of the factor, for instance, the C_3S content, sulfate solutions with different cations, chemical admixtures, and type of curing, among others.

In his study of sulfate resistance of blended cements, Irassar [34] described a new criterion of evaluation of sulfate resistance, called the 'crack-time.' Flexural strength development was used as the test parameter to evaluate the phases of sulfate attack: filling of pores, cracking, strength loss, and deterioration of the structure. With time, the flexural strength of mortar bars in sulfate solution was found to increase up to a maximum, and then decrease rapidly. The evolution of the flexural strength was modeled as a second-degree parabola. The crack-time was defined as the time at which the flexural strength of mortar bars was at maximum. Using this parameter, successful predictions could be made for performance of concrete containing various blended cements in sulfate solutions.

Kurtis et al. [35] analyzed data collected by the US Bureau of Reclamation over a 40-year period of nonaccelerated sulfate attack testing [28] to develop statistical models for prediction of sulfate attack. In this test, concrete cylinders for expansion measurements were submerged continuously in a 2.1% sodium sulfate solution ($\sim$14200 ppm SO_4). Analysis of the expansion data showed distinct behavior for low ($<$8%) and high (>10%) C_3A cements. Two regression-based models were developed to predict expansion by sulfate attack as a function of water to cement ratio, duration of exposure, and C_3A content. This study was particularly important because the data were derived from a long-term test under field-like conditions. Also, this study outlined the needs for statistical analyses of the available data in order to develop prediction models. However, it must be understood that expansion alone cannot be used as the basis of a prediction model regarding service life since it is only one of the manifestations of sulfate attack.

Different studies use various parameters (expansion, strength loss, etc.) to evaluate sulfate resistance. Modeling criteria can be successfully developed for sulfate attack if a unified parameter is used. The problem here is that due to the complicated mechanism involved in sulfate attack, no one parameter can be used to predict sulfate-related damage in all conditions. For example, continuous immersion studies in the laboratory often identify expansion to be a representative parameter for damage. However, in many field studies surface softening and loss of structural stability are seen to be the key damage indicators. Thus, new parameters, similar to the crack-time criterion described earlier, need to be developed if successful modeling of the impact of sulfate attack on the performance of a given structure is to be pursued.

6. Summary

Key issues related to the understanding of sulfate attack are discussed in the paper. These issues are concerned with filling in the gaps in the existing knowledge related to sulfate attack.

Different sulfate-bearing solutions produce varied effects in concrete. Any research should avoid generalization of results obtained from studying just one type of sulfate solution to all conditions.

In order to improve the understanding of the chemistry and the mechanism of sulfate-related damage, the role of attack products, in particular — gypsum and thaumasite, needs to be quantified. The role of gypsum formation should be investigated by studying C_3A-free systems as a reference, while studying the sulfate reaction at different temperatures should identify thaumasite effects.

Optimum blends of mineral admixtures with cement should be determined in order to mitigate magnesium sulfate attack, through the right combination of low permeability and product chemistry.

The current test methods and standards for sulfate attack need to be reexamined. Issues such as the changing pH of the solution, drying and wetting, as well as the effects of temperature and concentration of the sulfate solution, need to be addressed. Understanding the role of these parameters can improve the reliability of the tests by producing more field-like conditions of exposure.

Estimating the remaining service life of structures exposed to sulfate attack is important in order to develop repair and maintenance schedules for such structures. For this purpose, surface hardness methods and elastic stress wave methods (one-sided pulse velocity, spectral analysis of surface waves) need to be developed. It must be understood that although these methods are fairly simple to perform, sufficient calibration is needed to obtain reliable information from the data because of the complicated nature of the sulfate attack problem.

Modeling parameters and criteria should be developed so that efficient designs for durability can be obtained. As a first step towards modeling the sulfate attack phenomenon, a set of reliable experimental parameters needs to be determined. Sulfate attack damage does not manifest itself in the same way all the time, and thus recognizing any one parameter to quantify sulfate-related distress is quite a challenge. This is possible only with a good understanding of the mechanism and using reliable test methods.

Acknowledgments

Support from the Center for Advanced Cement Based Materials (ACBM) and the Andrews Fellowship from Purdue University are gratefully acknowledged. The authors also thank Dr. Bryant Mather for providing valuable comments and suggestions during the preparation of the manuscript.

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