

Kurdistan Engineering Union

Sulfate Attack

Research

By

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CHAPTER ONE

1.0 INTRODUCTION

Sulfate attack is the term used to describe a series of chemical reactions between sulfate ions and the components of hardened concrete, principally the cement paste, caused by

exposure of concrete to sulfates and moisture. As is the case with other aggressive chemicals, sulfates are potentially most deleterious to concrete when present in gaseous or liquid form, the latter situation being the most common; attack by solid sulfate-containing chemicals is rare. [1]

The differences in the consequences of any sulfate attack are caused by the environmental and physical conditions under which these reactions proceed. The chemistry of sulfate attack is complex and involves numerous overlapping reactions.

1.1. Background

Sulfate in soil and/or in water surrounding the concrete can interact with concrete in a number of ways that result in the loss of strength and the production of cracking in the concrete. [2] The rate of sulfate attack on concrete increases with an increase in the strength of the solution concrete attacked by sulfate has a characteristic whitish appearance the damage usually starts at edges and is followed by progressive cracking and spalling which reduce the concrete to friable state or even soft state.

Although some scientists have extensively debated the source of sulfate, the scientific community recognizes the associated deterioration of concrete. Conventional portland cement concrete can deteriorate when exposed to alkaline caused by alkali sulfate solutions. The major mineral formed by the sulfate-concrete interaction is ettringite ($C_6AS_3H_{32}$). Ettringite is one of the materials that normally forms during the early setting

of portland cement from a reaction between calcium aluminate and gypsum in the curing cement paste. The formation of ettringite from its constituent materials involves an increase in volume of 9.37% [3]. In the case where ettringite forms before the cement paste has gained strength, the increase in the volume can be accommodated without producing cracking. If ettringite forms after the paste has gained strength, the crystallization can cause cracking [4].

Some investigators point out that the sulfate compounds can simply form crystals in the pore spaces of the concrete—which produces cracking due to crystalline sulfate compounds growing or ripening in voids and develops stress that causes cracks in the surrounding concrete [3,5,6] . Most investigators agree that there is a “physical salt-weathering mechanism” but that chemical interaction of sulfate with the compounds present in concrete is part of the damaging interaction. In the chemical interaction, it is the transformation of the compounds in the concrete and sometimes the loss of reacted soluble components that causes the loss of strength in the concrete.

Additionally, there are well-documented cases of concrete deterioration due to acid sulfate attack resulting from acidic soil, water, or groundwater. Acidic soils can be produced by the oxidation of sulfides in the soil, and this condition can be extremely detrimental in that it results in dissolution of constituents in the concrete and in the formation of expansive crystalline compounds that cause cracking [7] .

CHAPTER TWO

2.0 MECHANISM OF SULFATE ATTACK IN CONCRETE

2.1 Sulfate Sources

There are two main types of sulfate sources in concrete, they are internal and external sources.

2.1.1. Internal Sources:

It is due to a soluble source being incorporated into the concrete at the time of mixing, gypsum in the aggregate, etc. It occurs when there are excess sulfates from the constituents' materials (concrete).

Examples include the use of sulfate-rich aggregate, excess of added gypsum in the cement or contamination, admixtures, hydraulic cements and fly ash. Proper screening and testing procedures should generally avoid internal sulfate attack. Internal sulfate attack is rarer compared to the external sources of sulfate.

Delayed ettringite formation (DEF) is a special case of internal sulfate attack. It occurs in concrete which has been cured at elevated temperatures, for example, where steam curing has been used. It was originally identified in steam - cured concrete railway sleepers (railroad ties). It can also occur in large concrete pours where the heat of hydration has resulted in high temperatures within the concrete.

DEF causes expansion of the concrete due to ettringite formation within the paste and can cause serious damage to concrete structures. DEF is not usually due to excess sulfate in the cement, or from sources other than the cement in the concrete. Although excess sulfate in the cement would be likely to increase expansion due to DEF, it can occur at normal levels of cement sulfate.

A key point in understanding DEF is that ettringite is destroyed by heating above about 70 C. Damage to the concrete occurs when the ettringite crystals exert an expansive force within the concrete as they grow. In normal concrete, the total amount of ettringite which

forms is evidently limited by the sulfate contributed by the cement initially. It follows that the quantity of ettringite which forms is relatively small. [8]

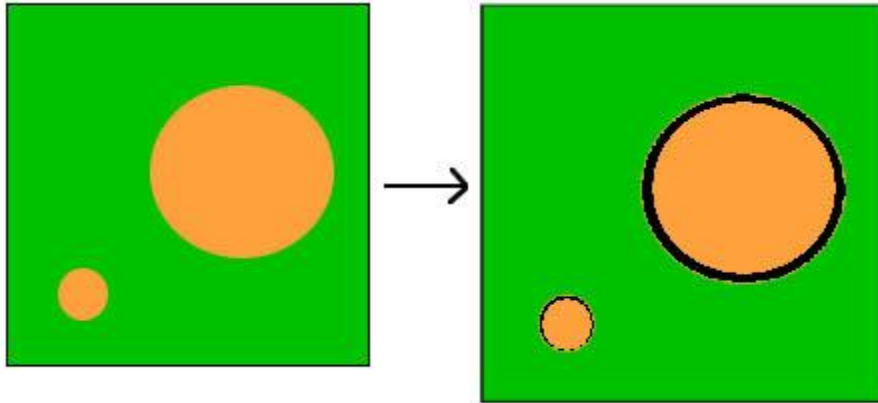


Figure 1 Diagram showing how paste expansion produces a small gap around small aggregate particles and a bigger gap around larger particles.

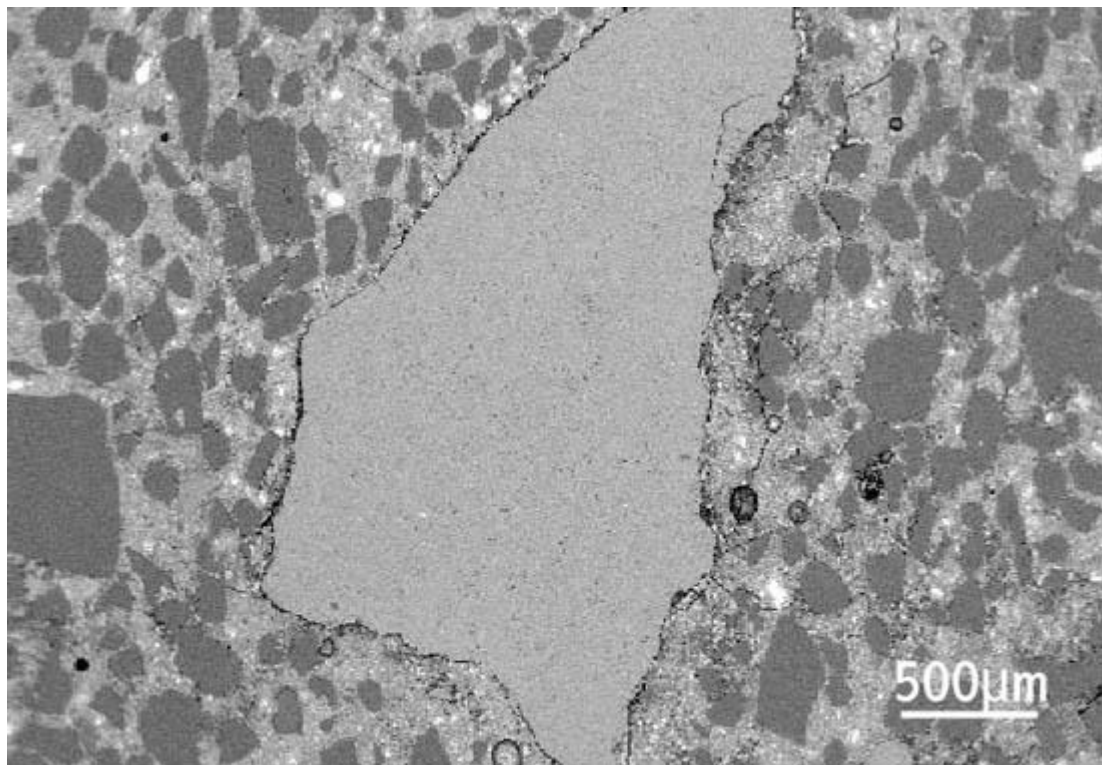


Figure 2 Scanning electron microscope image of limestone aggregate particle. The cement paste has expanded and a gap has formed between the aggregate and the cement

paste. This is characteristic of damage to concrete due to DEF. The aggregate is no longer contributing to concrete strength, since it is effectively detached from the cement paste. Often, these gaps become filled with ettringite. [8]

2.1.2 External Sources:

External sources of sulfate are more common and usually are a result of high-sulfate soils and ground waters, or can be the result of atmospheric or industrial water pollution. A fairly well-defined reaction front can often be seen in polished sections; ahead of the front the concrete is normal, or near normal. Behind the reaction front, the composition and microstructure of the concrete will have changed. These changes may vary in type or severity but commonly include:

- Extensive cracking
- Expansion
- Loss of bond between the cement paste and aggregate
- Alteration of paste composition, with monosulfate phase converting to ettringite and, in later stages, gypsum formation. The necessary additional calcium is provided by the calcium hydroxide and calcium silicate hydrate in the cement paste

The effect of these changes is an overall loss of concrete strength. The above effects are typical of attack by solutions of sodium sulfate or potassium sulfate. Solutions containing magnesium sulfate are generally more aggressive, for the same concentration. This is because magnesium also takes part in the reactions, replacing calcium in the solid phases with the formation of brucite (magnesium hydroxide) and magnesium silicate hydrates. The displaced calcium precipitates mainly as gypsum. [8]

Other sources of sulfate which can cause sulfate attack include:

1. Seawater
2. Oxidation of sulfide minerals in clay adjacent to the concrete - this can produce sulfuric acid which reacts with the concrete

3. Bacterial action in sewers - anaerobic bacterial produce sulfur dioxide which dissolves in water and then oxidizes to form sulfuric acid
4. In masonry, sulfates present in bricks and can be gradually released over a long period of time, causing sulfate attack of mortar, especially where sulfates are concentrated due to moisture movement

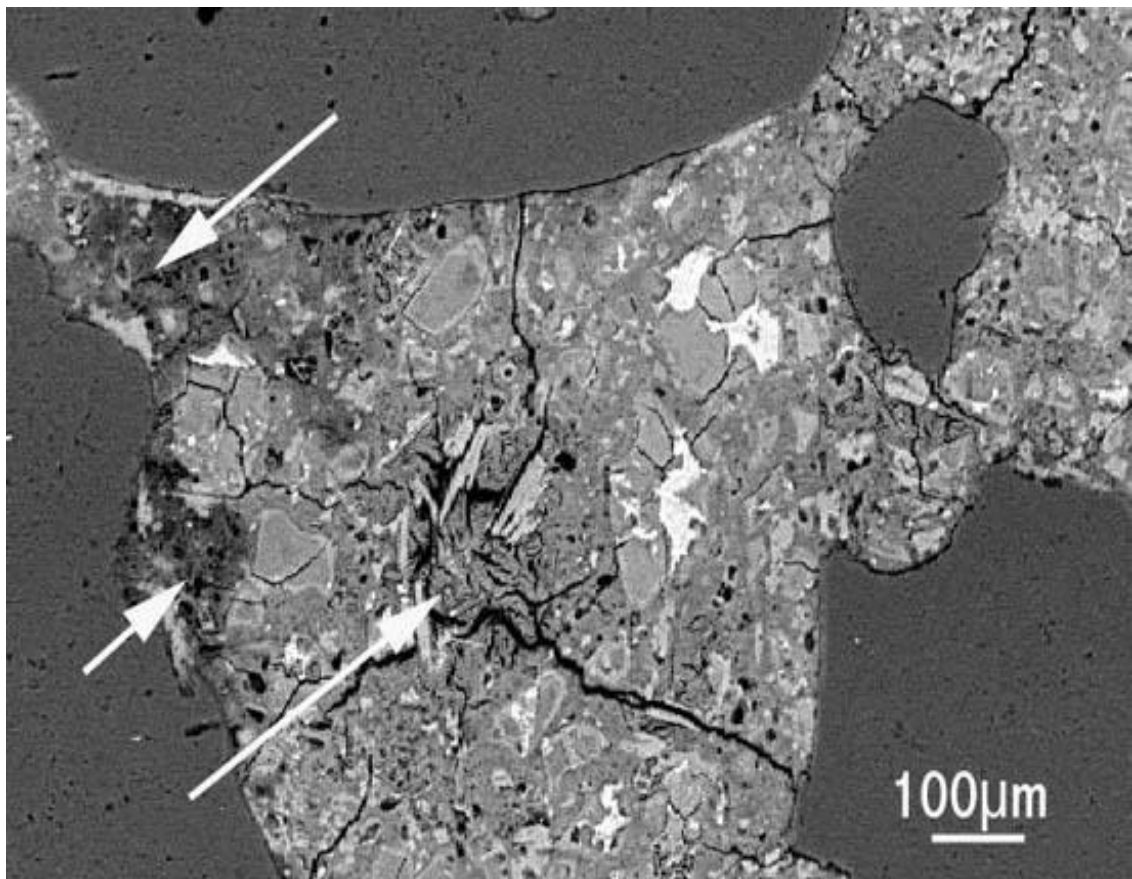


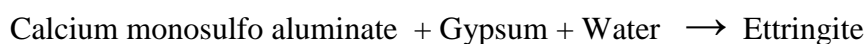
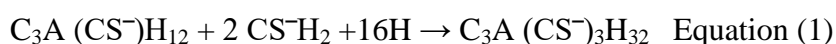
Figure 3 Scanning electron microscope image of sulfate attack in concrete. Ettringite (arrowed) has replaced some of the calcium silicate hydrate in the cement paste; the darker areas of paste have been partly decalcified. As a consequence of these alterations, the paste will be weakened. Although much of the cement paste here remains apparently unaltered (eg: top right), if widespread within the concrete (which in this instance it was) sulfate attack can significantly weaken the concrete. [8]

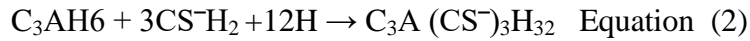
2.2 Types of Sulfates:

The complexity of sulfate attack begins first with the variety of sulfates that can damage concrete. The most common sulfates that interact with concrete are calcium, sodium, and magnesium sulfate. Each of these sulfate forms are discussed separately next, but one should bear in mind that it is common to find mixed sulfates present in the same groundwater or soil sample. [9]

2.2.1 Calcium Sulfate

Calcium sulfate (gypsum) is generally believed to be the least aggressive of the three sulfates, mainly due to its lower solubility. The solubility of gypsum is approximately 1440 ppm, which is significantly less than that of sodium sulfate and magnesium sulfate. There are essentially two different schools of thought on the issue of gypsum solubility and its impact on sulfate attack. One school of thought is that the inherently low solubility of gypsum limits its concentration in groundwater (or in the pore water of soil), and this low concentration limits the potential damage to concrete [10]. The other school of thought is that once calcium sulfate ions enter concrete, its solubility increases as it encounters the highly-alkaline pore solution within the concrete, and the higher sulfate concentrations can then more aggressively attack the hydrated cement paste. Specifically, as shown in equation (1), calcium sulfate will react with monosulfoaluminate and water to form ettringite, which can result in expansion and cracking. Calcium sulfate also reacts with tricalcium aluminate to form ettringite as shown in Equation (2). [9]



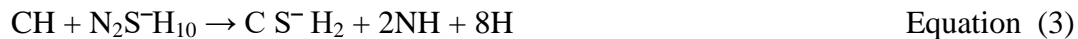


Calcium aluminate hydrate+ Gypsum + Water \rightarrow Ettringite

2.2.2 Sodium Sulfate

Attack from sodium sulfate is more complex than attack from calcium sulfate because more phases are affected. Sodium sulfate may attack concrete in two different ways. The first form of attack (Equation 3) involves sodium sulfate reacting with calcium hydroxide (portlandite) to form gypsum. Gypsum can then react with monosulfoaluminate (Equation 4) to form ettringite. Once the calcium hydroxide is depleted, gypsum formation will discontinue.

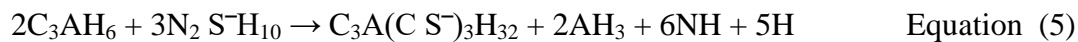
Once the monosulfoaluminate becomes depleted, excess gypsum will form in the system and ettringite formation will cease [11]. The second form of attack (Equation 5) involves sodium sulfate reacting with tricalcium aluminate to form ettringite.



calcium hydroxide + sodium sulfate \rightarrow Gypsum



Monosulfoaluminate + Gypsum \rightarrow Ettringite

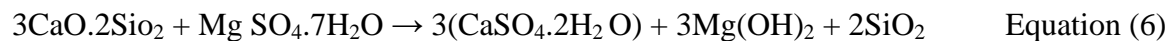


tricalcium aluminate + sodium sulfate \rightarrow Ettringite

studied the expansion of alite (C3S) caused by the formation of gypsum. In the case of a pure alite paste, no aluminum should be available to form ettringite. Cement pastes made with and without silica fume were placed in a 5% sodium sulfate solution. After 360 days in sodium sulfate, the alite paste, without silica fume, began to expand, and x-ray diffraction analyses showed that this expansion was due to gypsum formation [12]. These findings suggest that gypsum formation can, in fact, lead to expansion, in addition to the loss of mass or cohesion that is typically observed when gypsum forms.

2.2.3 Magnesium Sulfate

Magnesium sulfate is the most complex of the three types of sulfates. It can react with all hydrated cement products and is generally considered to be the most damaging form of sulfate. Magnesium sulfate will react with calcium silicate to form gypsum plus magnesium hydroxide and a silica gel, as shown in Equation (6). This formation of magnesium hydroxide (brucite) is known to form a barrier which may provide protection to the concrete and it also tends to internally affect pore solution pH. Brucite formation does have its downfall in that it needs a high amount of calcium hydroxide to form. Once the CH is depleted, the magnesium sulfate will seek more calcium. In this case, decalcification of the C-S-H will occur, due to a removal of calcium [11].



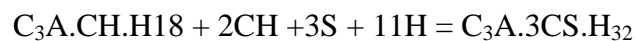
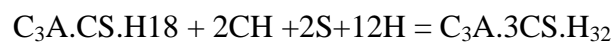
Calcium silicate + Magnesium sulfate → Gypsum + Magnesium hydroxide + silica gel

2.3 Nature of reaction:

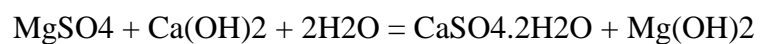
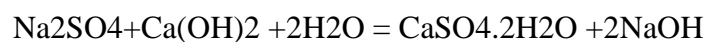
SULFATE ATTACK processes decrease the durability of concrete by changing the chemical nature of the cement paste, and of the mechanical properties of the concrete. There are chemical and physical processes involved in sulfate attack.

2.3.1. Chemical process:

The sulphate ion + hydrated calcium aluminate and/or the calcium hydroxide components of hardened cement paste + water = ettringite (calcium sulphoaluminate hydrate)



The sulphate ion + hydrated calcium aluminate and/or the calcium hydroxide components of hardened cement paste + water = gypsum (calcium sulphate hydrate)



Two forms of Chemical reaction depending on

- Concentration and source of sulfate ions.
- Composition of cement paste in concrete.

2.3.2. Physical process:

- The complex physico-chemical processes of "sulfate attack" are interdependent as is the resulting damage.
- physical sulfate attack, often evidenced by bloom (the presence of sodium sulfates Na_2SO_4 and/or $\text{Na}_2\text{SO}_4 \cdot 10\text{H}_2\text{O}$) at exposed concrete surfaces.
- It is not only a cosmetic problem, but it is the visible displaying of possible chemical and microstructural problems within the concrete matrix.

Both chemical and physical phenomena observed as sulfate attack, and their separation is inappropriate. [13]

2.4.Result of reaction:

1. Gypsum formation leads to reduction of stiffness and strength then by expansion and spalling of concrete.
2. Ettringite formation causes expansion results stresses and cracking in concrete .
3. Thaumasite formation a calcium-carbonate-silicate which attacks the C-S-H and causes Softening and loss of integrity of the concrete.



Figure 4: Cracking due to sulfate attack [13].



Figure 5 spalling due to sulfate attack [13] .

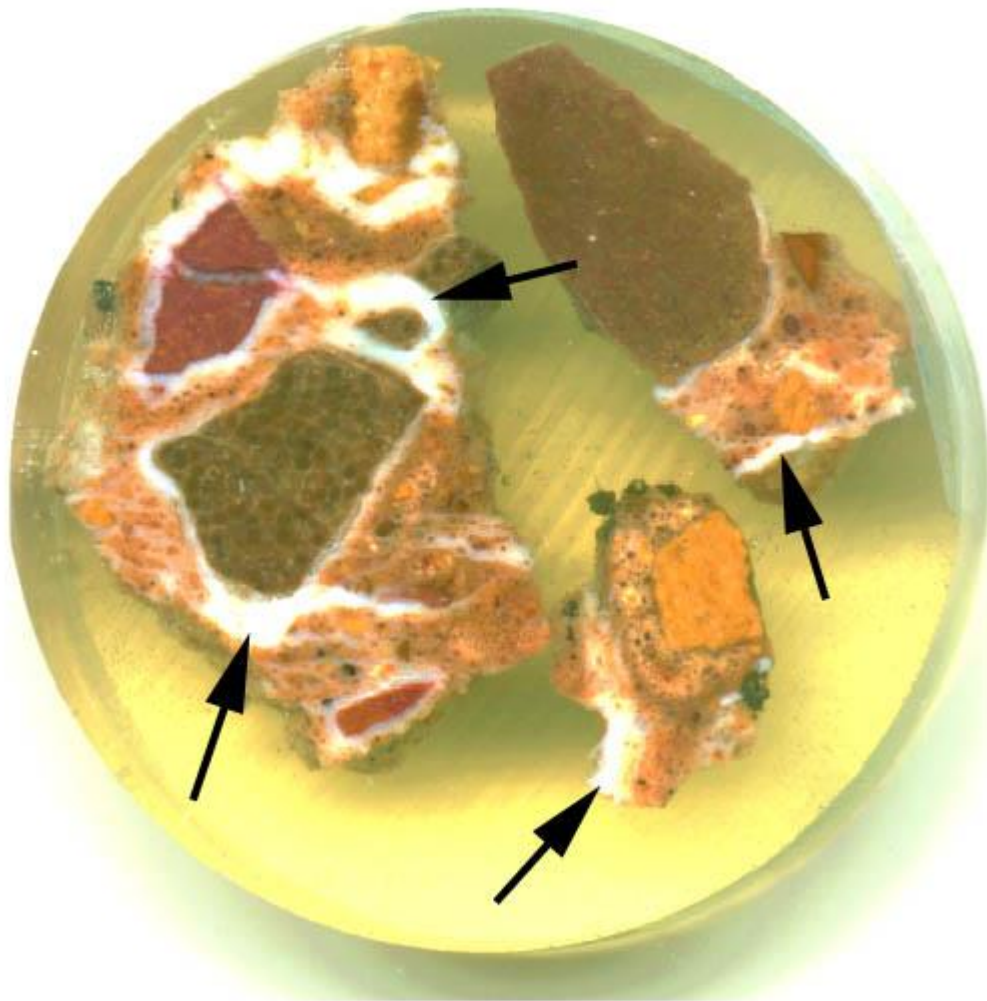


Figure 6. Thaumasite has formed around coarse limestone aggregate (large dark particles) and in cracks. Examples of thaumasite are arrowed. This polished section was 40mm in diameter but thaumasite formation is so extensive that it requires little magnification to be clearly visible.

Chapter three

3.1FACTORS AFFECTING SULFATE RESISTANCE OF CONCRETE

Sulfate attack on concrete will take place when the sulfate solution penetrates the concrete, chemically reacts with its constituents, mainly the cement matrix. Thus, factors affecting sulfate resistance of concrete are not only those influencing the chemical reaction with the cement matrix, but also those influencing the permeability and the overall quality of the concrete. Some of these factors include;

1. Cement type and content:

- The most important mineralogical phases of cement that affect the intensity of sulfate attack are: C_3A , C_3S/C_2S ratio and C_4AF [5]
- Cements with low C_3A content are less vulnerable to sulfate attack. However, cements with low C_3A have a higher C_3S/C_2S . An increase in the C_3S content of cement generates a significantly higher amount of calcium hydroxide. The calcium hydroxide can also react directly with the sulfate ions leading to the formation of gypsum, which is harmful
- Higher cement content significantly reduces the rate of sulfate attack

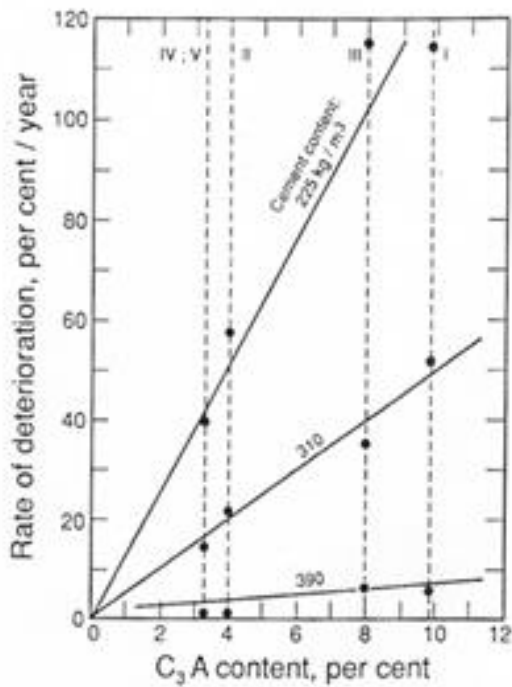
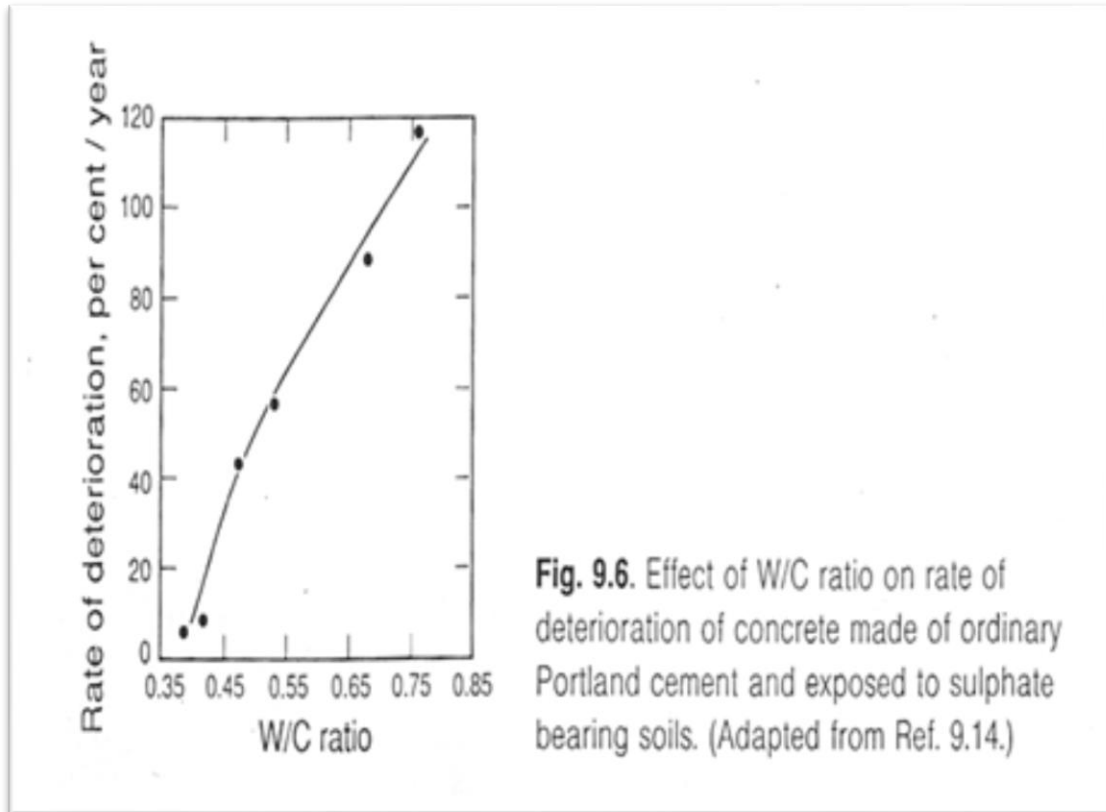


Fig. 9.5. Effect of the C_3A content in Portland cement on the rate of deterioration of concrete exposed to sulphate bearing soils. (Adapted from Ref. 9.14.)

2. Water-cement ratio

Low permeability of the concrete is a significant factor influencing its resistance to sulfate attack. Given good quality materials, satisfactory proportioning and good concrete practice, the permeability of the concrete is a direct function of its water-cement ratio and the curing time. In other words, all other factors being equal sulfate resistance of concrete increases with decreases in its water-cement ratio. [14]



3. Admixtures

There are many types of admixtures available for incorporation in concrete to enhance certain properties, or to achieve economy, or both. Generally, the effect of admixtures on concrete properties depends not only on their formulation and their interaction with the cement, but also on the variations or adjustments they bring about in the proportions of the concrete mix. Thus, admixtures that effect reduction in water-cement ratio and/or increased workability can enhance the sulfate resistance of concrete provided they are not used to reduce its cement content.

4.Design and detailing

Properly designed and detailed structures and elements should provide for adequate reinforcement, correctly located to minimize cracking. Detailing to minimize ponding and or areas of turbulence is important in reducing the intensity of the sulfate attack, thus enhancing the sulfate resistance of the concrete.

5.Construction practice

Concrete placing, compaction and curing are important factors in producing low-permeability concrete. Adding further water on site during placing of concrete to restore slump or to aid with the final finishing will impair the sulfate resistance of concrete. Adequate compaction and proper curing are required to produce dense concrete with discontinuous capillaries (low permeability). Finishing of the concrete to provide a dense smooth surface, free of holes and defects can enhance the concrete resistance to sulfate.
[14]

3.2 PREVENTION AND CONTROL OF SULFATE ATTACK

Certain measures can be taken to prevent sulfate attack:

1. protect the concrete against composition - induced internal sulfate attack.

cement itself may be a source of sulfate attack. Due to this, standards and requirements of ASTM (see ASTM C150; ASTM C1157; BS 5328) and other standards on cement and clinker compounds should be followed very strictly. If this is done, then proper

concentrations and ratios of the clinker materials to give sulfate levels that will not lead to excessive expansion.

Apart from clinker materials, aggregates and mineral additives are other potential sources of excessive sulfate. Therefore, aggregates and intermixed mineral admixtures should not contain sulfate-bearing compounds that may later become available for reaction with cement components of concrete mixture.

Quality control is the best method to use in preventing composition-induced internal sulfate attack. Continuous monitoring and proper records of the clinker material and other sources of sulfates should be kept in order to allow Quality control to work in its most efficient manner[1].

2. protection of concrete against heat-induced internal sulfate attack.

Proper mixture design is an efficient way of protecting concrete from degradation by heat-induced internal sulfate attack. The materials used in designing concrete mixtures must pass specifications and have a history of satisfactory performance. An important note is that the lowest possible water to cementitious material (w/cm) ratio is recommended.

A very important time during the production of concrete that should be taken special care of is during casting and curing. The formwork material and the thickness of it can affect the heat transfer of the concrete. This must be taken into consideration when designing for homogeneous heat and humidity distribution within the concrete. Exposed concrete surfaces should be kept wet. An even distribution of heat and humidity should be maintained when inside the curing chamber. Another important consideration that should be taken note of is the preset time. A preset time should be accurate to allow the cement to set properly. [1]

The heating rate can effect to concrete. The heating rate should be kept steady at approximately 15-20 C (25-35 F) per hour. The temperature rise should be evenly distributed within the concrete specimen and within the curing chamber. When heating the specimen, it should not be heated to the extent that the external surfaces are dried out. While heating the specimen, the difference in temperature between the inside and outside

surfaces should be monitored. The difference between the external and maximum internal temperature of a specimen should never exceed 20 C (35 F). The maximum temperature of the specimen should not rise above 65 C (150 F).

Once again, quality control must be followed to produce effective results. The control of the time-temperature regime is crucial to heat-cured process and must be followed closely.

3. Protection of concrete against external sulfate attack.

This measure is extremely important because the external surfaces of a structure are in contact with their surrounding environment. Therefore a good understanding of this environment should be taken note of, especially the sulfate-containing species.

An important procedure for this measure involves the variation of sulfate concentration. If the sulfate concentration varies, the concrete should be designed for the highest observed sulfate level. The environmental and atmospheric conditions should also be taken into consideration when properties such as temperature and humidity vary. [15]

“The three main strategies for improving resistance to sulfate solutions are:

1. Making a high quality, impermeable concrete
2. Using a sulfate resistant binder
3. Making sure that concrete is properly placed and cured on the site.

It is always important to remember that sulfate resistant cements are not a substitute for proper concrete making. The use of sulfate resistant cements is to be used in addition to

protection provided from a low w/cm ratio, adequate cement content, good mix design, etc[15] .

Problems can be experienced if sulfate or other aggressive chemicals penetrate hardened concrete. This is the reason why the concrete used should be dense and have a low porosity. As shown in the table below, the maximum w/cm ratio should be 0.5 and the minimum should be 0.4.

Proposed requirements to protect against damage to concrete by sulfate attack by external sources of sulfate:

Severity of Potential Exposure	Water-soluble sulfate (SO ₄) in soil (by mass)	Sulfate (SO ₄) in water (in ppm)	Maximum water-to-cementitious material ratio (by mass)	Cementitious Materials Requirements
Class 0 Exposure	0.00 to 0.10	0 to 150	No special requirement for sulfate resistance	No special requirement for sulfate resistance
Class 1 Exposure	More than 0.10 to less than 0.20	More than 150 to less than 1500	0.5	C 150 Type II or equivalent
Class 2 Exposure	0.20 to less than 2.0	1500 to less than 10000	0.45	C 150 Type V or equivalent
Class 3 Exposure	2.0 or greater	10000 or greater	0.4	C 150 Type V plus pozzolan or slag
Sea Water Exposure	-----	-----	0.45	C 150 Type II with maximum 10% C ₃ A or equivalent

3.3 Test methods:

The resistance of concrete to sulfate attack can be tested in the laboratory by storing specimens in a solution of sodium or magnesium sulfate ,or in a mixture of the two. Alternate wetting and drying accelerates the damage due to the crystallization of salts in the pores of the concrete. The effects of exposure can be estimated by the loss in strength

of the specimen, by changes in its dynamic modulus of elasticity, by its expansion, by its loss of mass, or can even be assessed visually. [16]

Two current ASTM test methods for assessing sulfate attack are ASTM C 1012 and ASTM C 452.

" ASTM C 452 is a test method for the potential expansion of portland cement mortars exposed to sulfate. In this standard, gypsum is added to the cement prior to mixing to produce 7% SO₃ by mass of cement and gypsum. The additional gypsum allows for ettringite formation to occur internally without the need for external sulfates to enter the specimens. Mortars are then placed in limewater and measured for expansion. This test method is not very common because it is intended only to be used as a test for portland cement, and thus has limited applicability to more varied systems, such as mixtures containing SCMs". [9]

"ASTM C 1012 is one of the most common tests used in practice and is often included in specifications and guidelines. The test involves casting mortar bars, typically containing a high-C₃A cement (with or without SCMs) and standard Ottawa sand, allowing the mixture to reach a minimum strength of 2850 PSI (20 MPa), and then immersing the specimens in 5% sodium sulfate solution for up to 18 months. This is the most common test method for determining sulfate resistance and is the test method chosen for equivalent testing in ACI 201.2R. One concern with this test is that the pH of the sulfate solution will change with time, as will the pH of the pore solution (due to leaching). To minimize this pH effect, the sulfate solution is required to be replaced each time a measurement is conducted. However, at intervals of longer measurement periods, the pH may change considerably, thereby affecting the progression of sulfate attack. [17] has proposed a test set-up that automatically adjusts the pH of the soak solution to minimize this effect, but the complexity of this approach has hindered its use in standard test methods". [9]

Chapter Four

4.0.CONCLUSION AND RECCOMENDATIONS:

Sulfate attack has long been recognized as responsible for concrete deterioration in a wide variety of structures , sulfate attack processes decrease the durability of concrete by

changing the chemical nature of the cement paste, and of the mechanical properties of the concrete. Sulfate in soil or in water surrounding the concrete can interact with concrete in a number of ways that result in the loss of strength and the production of cracking in the concrete. The rate of sulfate attack on concrete increases with an increase in the strength of the solution concrete.

Sulfate attack can be **external**: due to penetration of sulfates in solution, in groundwater or sea into the concrete from outside or **internal**: due to a soluble source being incorporated into the concrete at the time of mixing, or the presence gypsum in the aggregate.

Ettringite is one of the materials that normally forms during the early setting of portland cement from a reaction between calcium aluminate and gypsum in the curing cement paste, In the case where ettringite forms before the cement paste has gained strength, the increase in the volume can be accommodated without producing cracking. If ettringite forms after the paste has gained strength, the crystallization can cause cracking.

Most investigators agree that there is a “physical salt-weathering mechanism” but that chemical interaction of sulfate with the compounds present in concrete is part of the damaging interaction. The complexity of sulfate attack begins first with the variety of sulfates that can damage concrete. The most common sulfates that interact with concrete are calcium, sodium, and magnesium sulfate.

Sulfate attack on concrete will take place when the sulfate solution penetrates the concrete, chemically reacts with its constituents, mainly the cement matrix. Thus, factors affecting sulfate resistance of concrete are not only those influencing the chemical reaction with the cement matrix, but also those influencing the permeability and the overall quality of the concrete such as ; Cement type and content, Water-cement ratio, Admixtures, Design and detailing , Construction practice .

Apart from clinker materials, aggregates and mineral additives are other potential sources of excessive sulfate, Continuous monitoring and proper records of the clinker material and other sources of sulfates should be kept in order to allow Quality control to work in its most efficient manner, the Quality control is the best method to use in preventing composition-induced internal sulfate attack.. The materials used in designing concrete mixtures must pass specifications and have a history of satisfactory performance, A very

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