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Thermodynamic Study of Cement Paste under Sulfate Attack: A Review

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ABSTRACT

Sulfate attack reduces the service life of concrete structures, which is not possible to repair simply. Implementation of many factors influencing the sulfate resistance, sample scaling, and results in the least possible time is some problems of experimental studies. Therefore, numerical simulations, as well as the new methods along with experimental studies, have been led to better evaluation of sulfate attack within a shorter time and at a lower cost. In the present study, the effective factors, causes, and mechanisms of sulfate attack, examples of this phenomenon in real projects, and the previous studies in this regard, in particular, the development of the thermodynamic study of cement under sulfate attack as a fast and inexpensive solution have been reviewed. The present investigation is divided into three parts, first the sulfate attack mechanism, second the factors influencing phases containing sulfate formation, and third a review of the methods and results of the studies, focusing on the development of thermodynamic models in cement sulfate attack especially. Finally, the results of the studies show that common experimental methods related to concrete sulfate resistance evaluation can't always simulate what is actually happening; subsequently, the results of experimental studies and real cases are sometimes different. So, numerical models, in particular, thermodynamic simulation, either alone or in combination with experimental studies, can be a desirable solution for enhancing the ability to predict engineering behavior of concrete structures in the sulfate environment. Subsequently, it results in making better decisions to tackle and prevent deterioration caused by sulfate attack.

1. Introduction

Deterioration of concrete structures due to sulfate attack is one of the most important concerns in the field of concrete durability. The formulation of numerous theories regarding the sulfate attack process is formed because of its complicated nature [1]. Several theories about sulfate attack expansion mechanisms are developed. Some of the most popular theories are swelling [2], topochemical reactions [3], increment of the solids volume [4], and increase in the pressure due to crystal growth [5,6]. Some researchers, such as Hime and Mather [7] indicated that the mechanism of the sulfate attack isn't yet explicated clearly and several questions related to its chemistry aren't answered satisfactorily. They investigated the causes of concrete internal expansion during sulfate attack. Expansion can be caused due to chemical reactions or when the SO_4 ion isn't an active factor, such as rehydration and dehydration of some salts (e.g., sodium carbonate and sodium sulfate). So far, experimental methods have been widely used to study the various aspects of sulfate attack phenomenon from different types of point of view. Although, the experimental methods related to concrete sulfate resistance evaluation are efficient in the understanding of the sulfate attack mechanisms, however, they aren't suitable enough for concrete performance prediction under the real environmental conditions. It is difficult to simulate the real environmental conditions experimentally [8-11]. In some cases, the same redox conditions in an experimental simulation as in the natural system are impossible. On the other hand, experimental methods are costly, time-consuming, and difficult to schedule [12-17]. Therefore, always attempts to introduce a more suitable method or improve the available common experimental methods to take into account what a concrete structure actually

experiences in a sulfate-rich environment. The simulations needed to be more reliable and accurate to design high- durability concrete, which is suitable for corrosive environments. Proper simulation of a concrete sample under the sulfate-rich environment leads to the proper selection of the concrete components and its mixing ratio [1, 18]. The chemical reactions that occurred during the concrete sulfate attack were studied by Cohen and Bentur. The sulfate attack resulted in a lot of undesirable effects on the concrete structures. The mechanism related to each of these effects individually can be studied experimentally. But it is important to note that the combination of two or more these effects makes complexities which can't be assessed by the conventional experimental methods related to concrete sulfate resistance evaluation because these conventional methods can consider one of these effects [7]. Cohen and Mather in 1991, expressed a need for a systematic approach to a proper studies framework creation about sulfate attack. They proposed several criteria for the sulfate attacks assessment. Their studies were conducted with the aim of a more desirable process creation for understanding the mechanism of sulfate attacks and creation of a more reliable method [19]. Some criticism is pointed to the current testing methods for evaluating sulfate attack lately. In this regard, it can be mentioned in the article by Skalny and Pierce published in 1999. They suggested that the existing testing methods for evaluating sulfate attack doesn't express properly the real field situation. Improving the tests are needed to obtain field-like conditions in the experimental [20]. So, it is necessary to find solutions to boost our ability for the understanding of sulfate attack mechanism and proper prediction of concrete structures performance exposed to sulfate attack. Thermodynamic model to simulate the concrete performance under unfavorable

conditions is one of the suitable solutions which is taken into consideration recently. Thermodynamic models have been succeeded in the content of hydration and durability of concrete. In this paper, a review of concrete sulfate attack studies is conducted, and the necessities of using thermodynamic studies alone or along with experimental studies are mentioned.

2. Sulfate attack mechanism

2.1. Sulfate source

Sulfate attack in mortar, cement, and concrete can be external or internal. The sulfate ions source of internal sulfate attack comes from the concrete itself related to cement, aggregate, and water. The sulfate ions flow in the surrounding environment and afterward, the requirements are prepared for external sulfate attack occurrence. Soil, groundwater, industrial waste, and chemical industry are some of the sulfate ions source of external sulfate attack which penetrate into the porous structure of cement matrix [21,22]. Sulfur can be considered as one of the first nine abundant elements on the earth [23]. Therefore, the probability of its existence as diverse forms inside or around of structural elements is very high. The surrounding soil and ground water are main external source. Sulfates and sulfides can be converted to sulfate-ions during oxidation mechanism. Number of compounds contained sulfates are sodium sulfate (Na_2SO_4 , Glauber's salt), magnesium sulfate (MgSO_4 , epsomite), potassium sulfate (K_2SO_4) and calcium sulfate (CaSO_4) which are different in solubility level [24,25]. Two main sources of sulfate ions are alkali metal and calcium sulfates which are converted to sulfate ions and they enter the water and soil. In the presence of water, sulfate ions easily penetrate into the concrete and disturb the

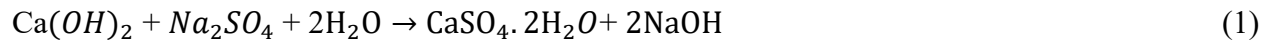
balance of the system. Chemical reactions occur until the unbalance system reaches equilibrium again. Eventually, many physical and chemical changes occur [26]. Pyrite (FeS_2), marcasite (FeS_2) and pyrrhotite (FeS to $\text{FeS}_{1.8}$) are some sulfide-containing compounds, natural ground such as clay soil is usual source of sulfides [27,28].

2.2. Cement-sulfate reactions

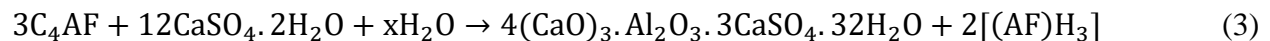
Ettringite expansion phenomenon isn't the only factor explained the concrete deteriorations due to sulfate ingress into the cement concrete system and it is needed to investigate the possible physical-chemical effects of sulfate ions on all the phases of the hydrated cement paste to understand cement deterioration process during sulfate attack [26].

2.2.1. Ettringite formation

Chemical reactions between sulfate ions and hardened concrete are known as sulfate attack on concrete. Ettringite formation and, at high sulfate concentrations, gypsum formation is generally defined as the result of these reactions [29,30]. Penetrating sulfates reaction with AFm forms ettringite. Also, aluminium existence due to C-A-S-H phases in hardened cement pastes composed of Portland cement and fly ash is another progressive factor helps to form ettringite [31]. In fact, ettringite ($3\text{CaOAl}_2\text{O}_3 \cdot 3\text{CaSO}_4 \cdot 32\text{H}_2\text{O}$) or gypsum ($\text{CaSO}_4 \cdot 2\text{H}_2\text{O}$) are produced when calcium hydroxide (portlandite) and calcium aluminate phases react with sulfate ions in hard concrete called conventional sulfate attack. According to equation 1 and 2, in the presence of moisture, sulfate salts such as sulfate and sodium sulfate magnesium, penetrate into the concrete and react with portlandite (CH). This reaction generates gypsum, sodium hydroxide and magnesium hydroxide (brucite).



Gypsum is responsible for softening and decreasing of the concrete compressive strength. In addition to reducing the compressive strength, Santhanam et al. in 2003, investigated the effect of gypsum formation on the concrete expansion during external sulfate attack [32]. Gypsum produced according to equation 1 and 2, can react with calcium aluminate and monosulfate, and caused to form secondary ettringite [33,34]. Expansion, cracking, and spalling are the latter consequence of the expansive phases production [35-39]. The



It is concluded that the mechanism of sulfate attack can be summarized in several steps generally. Somehow, sulfate salt migrates into a concrete structure. The arrival of the aggressive species leads to destroy the chemical balance of the system. So, ion sulfate chemically reacts with the hydrated cement, after that the dissolution of portlandite occurs, the solution pH is decreased, then the C-S-H gel is decomposed, and finally the new phases are produced during the chemical reaction of sulfate attack. The production and the growth of the new sulfate-containing phases change the concrete microstructure. The microstructure of the concrete made with GGBS and fly ash under the sulfate attack is shown in figure 1 and 2, respectively. Sodium sulfate nucleation and crystallization, and delayed ettringite production which is the cause of a large amount of rod-like ettringite existence in hardened cement pastes are caused the considerable volume increment of the concrete exposed to the sulfate attack [42]. The concrete expansion mechanism due to the formation of ettringite is not exactly well understood, and there is

presence of active aluminum in the cementitious phase greatly increases the possibility of secondary ettringite formation due to sulfate ions influx to the concrete structure during sulfate attack [40]. Neubauer and Götz-Neunhoeffler studied The pure C_4AF phase. They found that the higher the Al/Fe ratio, the greater the reactivity of C_4AF . C_4AF phase can react with gypsum to produce the secondary ettringite, as shown in equation 3 [41]:

no direct relevancy between the observed expansion and the amount of ettringite formation.

Recently, the crystal growth pressure theory is widely used to explain concrete expansion mechanism during sulfate attack. Although, the Issue has definitely not been proven. Crystallization pressures are generated when ettringite formation has occurred in the pores which are nearly 100 nm or smaller. Crystallization pressure in small pores goes beyond the tensile strength of the cement. In fact, the super saturation of the pore solution causes the driving force of crystallization pressure [5,6]. Also, it must be noted that there is no danger of generating crystallization pressures and significant expansion when needle-shaped and bulky ettringite crystals are produced in the spaces which aren't very small. Concrete deterioration due to ettringite formation occurs when pores occupied by ettringite crystals are about 10 nm or smaller. The stress increases due to the expansion amplification in the cementitious system. Sometimes the stress goes beyond the tolerable stress level of concrete, such as 8

MPa for tensile strength. Increment of C3A contents and sulfate concentrations cause higher stresses and amplify concrete deterioration. Restraint degrees of the concrete sample have effect on deterioration mechanism due to ettringite formation. The

higher degree of restraint causes the lower expansion amount. The expansion of unrestrained concrete sample is faster and larger in comparison to the restrained sample [43].

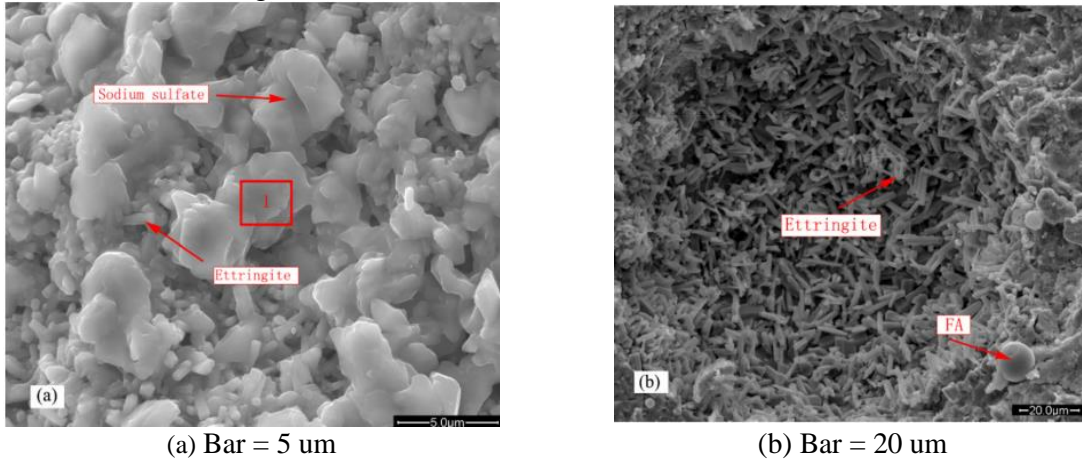


Fig. 1. Microstructure figures of concrete made with fly ash under sulfate attack for 120 days (Prepared by SEM (Scanning Electron Microscopy)) [42]

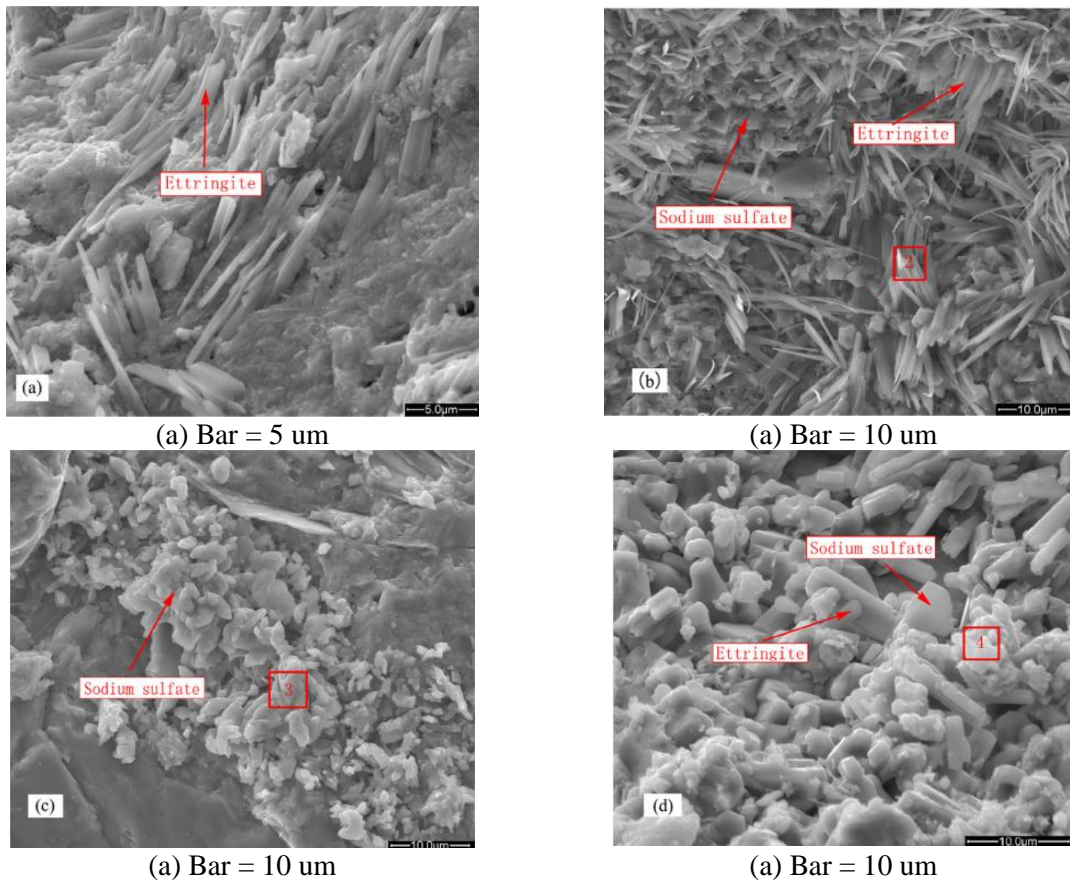


Fig. 2. Microstructure figures of concrete made with GGBS under sulfate attack for 120 days (Prepared by SEM (Scanning Electron Microscopy)) [42].

2.2.2. Thaumasite formation

A person named Nordenskiöld explore thaumasite mineral for the first time in the world, in 1878 [44]. It is a rare mineral in nature. The mineral thaumasite usually exists as a secondary mineral in metamorphic rocks. It is observed beside ettringite, calcite, aragonite, gypsum, portlandite, merwinite, gehlenite, spurrite, anhydrite, and

tobermorite [45-47]. The structure of thaumasite was determined by Moore and Taylor [48] and Edge and Taylor [49] in 1969. Morphologically, thaumasite is so similar to ettringite (needle-like and sometimes column-like crystals) and can be mistaken for each other [50-52]. The microstructure of thaumasite is shown in figure 3 [41].

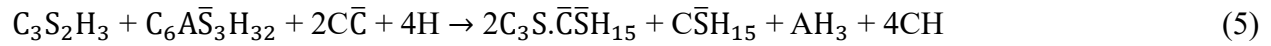
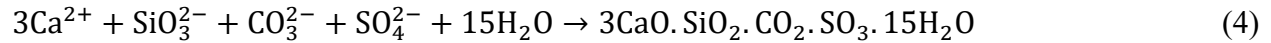


Fig. 3. SEM figures of the natural mineral thaumasite [41]

Thaumasite ($\text{CaSiO}_3\text{CaCO}_3\text{CaSO}_4 \cdot 15\text{H}_2\text{O}$) is another most important product besides gypsum and ettringite generated during the chemical reaction between penetrating sulfates and hardened cement binder. Source of calcium silicate, sulfite ions (SO_3^{2-}) or sulfate (SO_4^{2-}), carbonate ions released from some fillers or aggregates of the concrete, carbonation phenomenon in concrete, soils, groundwaters, and the air, or easily from limestone, and also, low temperatures of about 15°C or lower, and a very wet environment are necessary conditions to produce thaumasite and it doesn't need aluminate phases to form. Low temperature for thaumasite formation isn't a certain condition, but it boosts thaumasite formation

ability; in 1999, the sulfate attack occurrence in southern California showed signs of thaumasite formation, while low temperatures were unlikely in that area [53]. Thaumasite sulfate attack (shortly TSA) is concrete deterioration due to thaumasite formation caused by the sulfate ions penetration from an external source. The presence of thaumasite in the porous concrete structure doesn't always mean the thaumasite sulfate attack occurrence, but the overproduction of thaumasite in the structure of concrete is indicative of thaumasite sulfate attack. Thaumasite can be produced in two forms (direct and indirect). In the presence of a large number of sulfate ions and adequate moisture, calcium silicate hydrate (C-S-H)

reacts with calcite and as a result, thaumasite is produced directly (equation 4). But, in the indirect form, ettringite is converted to thaumasite. At first, in the presence of sulfate ions, ettringite is formed in a wet



The calcium silicate hydrates (C-S-H gel) are attacked during the TSA. The danger of formation thaumasite is because of the nonbinding crystalline phase, formed at the expense of the C-S-H phase, results in conversion hard concrete to the soft and mushy content which no longer is cohesive. In other words, the bonding property of the cementitious system is lost and the concrete color turns to white [28,35,37,38,39,55]. In Figure 4, the appearance of concrete suffered thaumasite sulfate attack is shown. The TSA is more likely to occur for the buried construction because sulfate ions flow in groundwater and cause the chemical attack on concrete. So, deterioration due to TSA can be found in the concrete foundations, pillars, floor slabs, columns, slabs of foundations of buildings, pavement, drainage construction,

environment; then it reacts with hydrated calcium silicate (CSH) and carbonate or bicarbonate. Finally, according to equation 5, thaumasite is formed due to ettringite conversion [41,54].

sewer pipes, road pavements, tunnel linings, concrete anchors, in a grout in a mine, water dams, in historical buildings, and lime stabilisations [37,39,47,56,57,58]. For the first time, Erlin and Stark identified and reported on the thaumasite formation in cement-based systems during the sulfate attack. They discussed four different cases of thaumasite formation during sulfate attack over two years in the 1960s. The thaumasite suffering cases located in different parts of America, and they included structures such as pavement, sewer pipes, and mine [59]. Sample of thaumasite sulfate attack has been summarized as shown in table 1. From this table, it is clear that the occurrence of thaumasite sulfate attack in different structures depends on many factors.



(a) The appearance of precast concrete as a retaining wall under thaumasite sulfate attack



(b) The appearance of concrete under thaumasite sulfate attack

Fig. 4. thaumasite sulfate attack [60]

Table 1. Summary of thaumasite sulfate attack.

Type of structure	Location	Deterioration	Possible Causes	Source
Ferenc Puskás (old Ferenc stadium, which is made of concrete)	Budapest	Corroded reinforced concrete elements, mushy concrete	It is affected by the waste gases produced by a nearby rubber plant and railway station	[38]
Buried structures on the M5 Motorway such as columns and bridge foundations	Gloucestershire, UK	severe surface deterioration and expansion	low temperature, limestone aggregates, unweathered clay, very wet, groundwater enriched in sulfates	[28,39,58,61]
glazed wall tiles	France	to lose cementitious bonding between underlying render and glazed wall tiles because of the changing concrete to thaumasite completely	cool temperature, water penetration, and gypsum/lime plasters with cement-based impurities	[62]
Concrete piles belong to a building that is 4 years old	UK	white, pulpy mass	limestone aggregate, very wet, Sulfates groundwater	[63]
three piers of Tredington/Ashchurch bridge	UK	Softening of the concrete, significant expansion, wide cracks	presence of mobile groundwater, limestone aggregate, Lower Lias Clay	[64]
the concrete foundations	Southern California, USA	Much of the cement paste had lost its integrity	soils contained significant quantities of sodium chloride, magnesium sulfate, and sodium sulfate.	[65,66]
railway tunnel	France	significant skewed flaking	Presence of SO_4^{2-} ions in surroundings	[67]
tunnels	northeast of Italy	Crack growth and conversion of concrete to a mushy material	aggregate was containing dolomite contaminated with anhydrite and/or gypsum (sulfate and carbonate sources in aggregate)	[68]
bridge	northeast of Italy	Concrete cracking, transforming into a pulpy mass	aggregate consisted of dolomite contaminated with anhydrite and/or gypsum (sulfate and carbonate sources in aggregate)	[68]
tunnel lining	Italy	plastic mass	very high humidity, low temperatures, CO_3^{2-} and SO_4^{2-} ions dissolved in the groundwater, ground rock composed of pyrite and calcareous schists (source of carbonate ions)	[69]

parapet walls of three important buildings	southeast of England	Severe crack and deterioration	sulfate-bearing clay bricks, wet condition	[70]
reinforced concrete foundations, columns and slab	Canadian Arctic	Expansion	severe cold weather, high levels of sulfates in the groundwater, aggregate contained dolomite and calcite (source of carbonate ions)	[71]
lime stabilized layer as subbase	central England	150 mm upheave of ground during one winter season	combination of lime and clay contained pyrite, cold weather, wet condition	[72]
swimming pool	Germany	Loss of resistance	contact with groundwater contaminated with sulfate, microcrystalline partial biogenic limestone aggregates, low temperatures	[73]
tunnel walls	Germany	Only presence of small pieces or wet mush instead of firm concrete structures	sulfate-containing water, low temperatures, ground limestone	[73]

Although environmental conditions such as adjacent soil and ground water plays a key role in the deterioration intensity caused by sulfate attack, thaumasite corrosion can happen in normal conditions. Slomka-slupik and Zybura indicated that thaumasite alone or a combination of thaumasite and ettringite as a solid solution could be formed without the need to supply sulfate ions from outside of the concrete system at the ambient pressure and temperature (298.15 K and 102 ± 1 kPa) called thaumasite non-sulfate attack (T n S A). For example, thaumasite can directly and extend produce at the expense of the calcium silicate hydrates (C-S-H gel) in case of cement paste containing slag without a corrosive medium like a sulfate salt solution [55].

3. Factors influencing phases containing sulfate formation

3.1. pH, water content

Usually reported that thaumasite formation and leaching are respectively at pH 12.5 and

below pH 8.0 [74]; however, concrete paste ingredient (cement type) affects thaumasite formation pH. pH range of thaumasite in case of CEM I and CEM III is respectively 8.78-9.12 and 8.45-11.70. Nevertheless, changes are in a certain range and drop of pH value occur during thaumasite formation [55]. Ettringite is unstable and starts to dissolve at lower pH.

As expected, A lower water to cement ratio (W/C) increases the concrete sulfate durability. As the W/C increases, porosity and permeability increase too, which lead to increment of the sulfate ions penetration and subsequently, reduction of concrete durability [75]. Monteiro and Kurtis in 2003, proposed a safe zone for the water to cement ratio (0.45) that concrete structures show favorable performance in this range [76].

3.2. Temperature

The temperature ranges below 15°C (ideally about 5°C) accelerates thaumasite formation, in the opposite, the temperature range more

than 15°C is suitable to form ettringite [77]. Conversion step where thaumasite formed from ettringite to be temperature range between 15°C and 20°C [28]. As previously mentioned, thaumasite formation at high temperatures (more than the threshold of about 15°C) is possible; however, its formation rate isn't as much as at lower temperatures. In other words, the lower the temperature, the higher the thaumasite formation rate [78,79]. Increasing carbon dioxide solubility and thaumasite insolubility with decreasing temperature is why thaumasite sulfate attack is more likely to happen at lower temperatures [80-82]. Also, the part of the thaumasite structure identified six-co-ordinated $[\text{Si}(\text{OH})_6]^{2-}$ groups as Bensted [79] states, is more stable at lower temperatures.

3.3. Sulfate solutions

The expansion is faster, when the initial concentration of sulfate ion is so high (about

30 g/L) [43]. As we know, high initial sulfate concentration leads to increase formation of secondary ettringite during sulfate attack; however, Irbe et al. in 2019, investigated the effect of the changing sulfate solution concentration on the amount of ettringite produced during the sulfate attack (secondary ettringite) in the range of 3 to 30 g/L SO_4 . Contrary to what is expected, the increment of sulfate solution concentration boosts only a small amount of ettringite formation, while high aluminum availability of the C-A-S-H phase has more effect on the amount of ettringite compared to the increment of sulfate solution concentration. The relationship of the formation of secondary ettringite and Al/Si ratio for different sulfate solution concentration is shown in figure 5 [83].

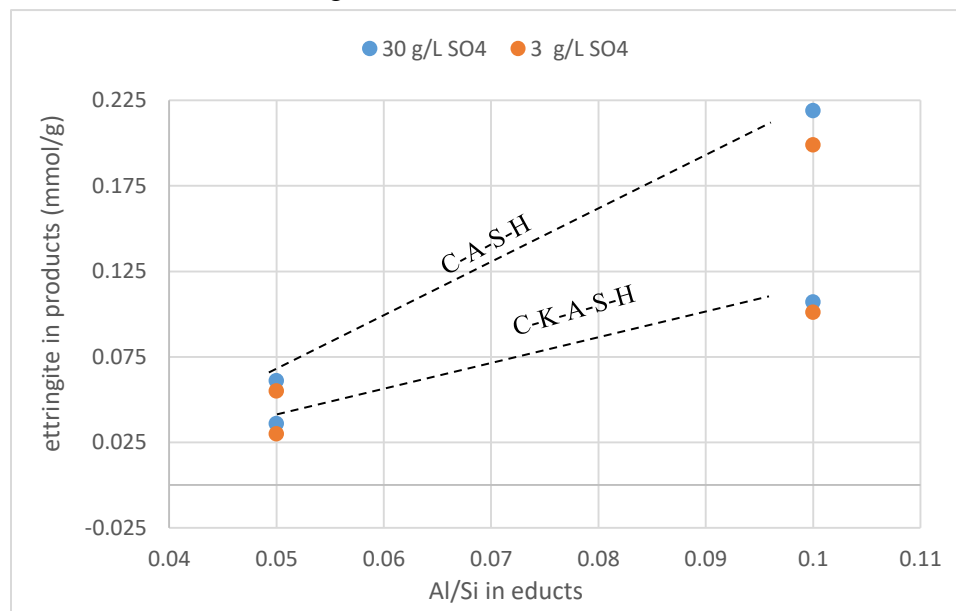


Fig 5. The amount of ettringite versus the initial Al/Si ratio for the different sulfate solution concentration [83].

Magnesium sulfate and calcium sulfate are respectively, the most and the least

aggressive in comparison with other sulfate solutions forms. Magnesium sulfate can

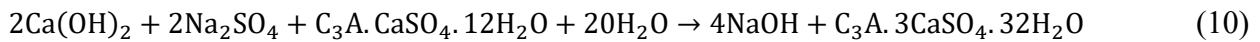
involve in the chemical reactions with most of the reproduced phases of the hydrated cement; so, it is known as the most aggressive sulfate solution during sulfate attack. According to equation 6, Brucite (magnesium hydroxide) and gypsum are formed through reaction of magnesium sulfate and portlandite. magnesium hydroxide isn't soluble and pH of the solution

$$\text{Mg}^{2+} + \text{SO}_4^{2-} + \text{Ca}(\text{OH})_2 + 2\text{H}_2\text{O} \rightarrow \text{Mg}(\text{OH})_2 + \text{CaSO}_4 \cdot 2\text{H}_2\text{O} \quad (6)$$

Low solubility of calcium sulfate makes it as the least aggressive sulfate phase. Calcium sulfate reactions during sulfate attack is caused to ettringite formation from calcium

$$\text{C}_3\text{A} \cdot \text{CaSO}_4 \cdot 12\text{H}_2\text{O} + 2\text{CaSO}_4 + 20\text{H}_2\text{O} \rightarrow \text{C}_3\text{A} \cdot 3\text{CaSO}_4 \cdot 32\text{H}_2\text{O} \quad (7)$$

The solubility of sodium sulfate is much larger than calcium sulfate solubility. Sodium hydroxide and gypsum are produced through the reaction of sodium sulfate and portlandite (equation 8). The pore solution pH rises up to 13.5 because of increment in sodium hydroxide formation In the next step,

$$\text{Na}_2\text{SO}_4 \cdot \text{H}_2\text{O} + \text{Ca}(\text{OH})_2 \rightarrow 2\text{NaOH} + \text{CaSO}_4 \cdot \text{H}_2\text{O} \downarrow \quad (8)$$


3.4. Time

Expansion rate during time isn't steady. In the early weeks, the expansion rate is low. Afterward, the rate increases rapidly up to a critical level. The critical point depends on the various parameters such as the amount of tricalcium aluminate (C_3A) and sulfate solution concentration. Whereas the expansion rate in the restrained concrete is high in the early weeks and then gradually decreases, it continues until the concrete structures crack and disintegrate completely at a certain time or the expansion rate decreases and approaches a constant value. A

containing high amount of magnesium hydroxide (saturated with magnesium hydroxide) is about 10.5. The brucite production requires a lot of portlandite, and when portlandite is completely consumed, magnesium sulfate demands more calcium. So, calcium silicate hydrates (C-S-H) decomposition is increased [75]

alumina-ferrite hydrates (AFm). The delayed ettringite formation due to calcium sulfate attack is presented in equation 7 [75].

Secondary ettringite could be formed through the chemical reaction between gypsum and some hydration products like as monosulfate, calcium aluminate hydrate, and non-hydrated tricalcium aluminate (C_3A) (equation 9 and 10) [75].

set of irregular cracks appear for the concrete structures which are restricted, while a regular pattern of a crack and in the direction of tension arises in the restrained concrete structures [43].

4. Experimental studies of sulfate attack

For many years, experimental methods have been used to investigate the mechanism and prevention solutions of sulfate attack. Despite the good progress in developing the experimental methods, many aspects of sulfate attack can't be easily surveyed

simultaneously. Each of these experiments considers just a part of sulfate attack phenomenon lonely and other parts are disregarded. Also, the effect of parameter changes on a critical range of other parameters has not been thoroughly investigated, because common experimental methods related to concrete sulfate durability evaluation have limited ability to simulate the real environment conditions. In the following, the history of experimental studies on concrete sulfate attacks is discussed, whose valuable results can be the basis of future numerical studies.

Manget et al in 1992, investigated the curing conditions effects on samples sulfate resistance and also, they studied the effect of different pozzolans addition, such as slag and silica fume. cement was replaced by 40 and 80 percent. They observed the expansion of samples containing 40 percent slag was more than control samples and even are failed due to sulfate attack. It means that low percentages of slag replacement aren't desirable under the sulfate situation [84]. Lee et al. in 2005, conducted an experimental research to investigate the effect of water-cement ratio variation and silica fume replacement on samples sulfate resistance in sodium and magnesium sulfate solutions. results show that resistance to sodium sulfate attack improves by replacing 5 to 10 percent of silica fume however, as the replacement increases, compressive strength decreases [85]. Another study, the effect of rice husk ash (RHA) on the durability of mortars against sulfate attack was investigated by Chindaprasirt et al in 2007, The results show that expansion of mortar prisms was reduced by replacing rice husk ash [86]. Giaccio et al in 2007, tested the

concrete specimens contained 0 and 10 percent rice husk ash replacement of cement and water-cement ratios of 0.28, 0.32, 0.4 and 0.5. Sulfate resistance of concrete samples increased by rice husk ash replacement [87]. Samples with water-cement ratios of 0.55 and 0.65, and replacement of the cement with two types of rice husk ash were subjected to sulfate durability test by Chatveera et al in 2009. It was found that, although samples containing rice husk ash showed good performance in sodium sulfate solution but they showed poor performance in magnesium sulfate solution [88]. Ramezaniapour et al in 2010, investigated the mechanism of sulfate attacks in concrete by experimental methods. They found that parts of hydrated cement contained calcium and aluminum hydroxide, increase the durability of concrete and cement against sulfate ions. In fact, the deterioration due to sulfate attacks is related to ettringite formation. Swelling due to sorption of water by the ettringite molecule and exerting pressure caused by rising crystals of ettringite are factors caused concrete deterioration during sulfate attack [89]. Tuson in 2012, studied the effect of limestone replacement on the mortars durability exposed to high-density sulfate solutions. they concluded that the high rate replacement of limestone had negative effects on the durability of mortars although, lower rate replacement of it can lead to better performance under sulfate situation [90]. Chu et al 2013, evaluated the pulse velocity of concrete samples exposed to sulfate environment. Wave velocity in concrete was calculated using an attenuation coefficient which depends on factors such as elastic wave velocity and exposure time to sulfate solution. Attenuation coefficients of

ultrasound waves velocity in concrete samples contained fly ash for different concrete mixing plans under the sulfate attacks was discussed. The results show that, when attenuation coefficients is increased, the sulfate deterioration in concrete is intensified [91]. Also, Zhu et al in 2013, studied the dynamical properties of cement mortar under sulfate attack using ultrasonic method [92]. Kunther et al in 2013, investigated the sulfate attack on cements containing 70 percent blast furnace slag exposed to pure sodium sulfate and magnesium sulfate solutions as well as sodium sulfate and magnesium sulfate solutions containing impurities. The lab results indicated the main failure mechanism of Portland cement due to sulfate attack was expansion, while the main failure mechanism of cements containing blast furnace slag was surface deterioration. Also, they found conflicts in the results of common experimental methods related to concrete sulfate durability evaluation and observations of sulfate attack in reality. The presence of impurities such as sodium, potassium and calcium in magnesium sulfate solution, in comparison with pure magnesium sulfate solution, caused less expansion and subsequently less concrete deterioration. Therefore, the presence of a mixture of cations in a sulfate solution is one of the reasons why lab results and real observations of sulfate attack are not always in good agreement [93,94]. Ramezani pour et al in 2013, assessed the durability of concrete made with rice husk ash (RHA). After 28 curing days, samples containing 7, 10 and 15 percent rice husk ash replacement of cement were exposed to magnesium sulfate and sodium sulfate solution. The sulfate attack severity was evaluated by mass loss and

compressive strength measurement continuously and periodically. The expansion rate of the control mortar prisms was higher than the blended cements samples. In addition, the microstructure of the samples under the sulfate solution was investigated by electron microscopy [95]. Ramezani pour et al in 2014, studied the effect of natural pozzolans such as terrace, pumice and tuff on the concrete durability during sulfate attack experimentally. Samples containing different percent pozzolan replacement of cement were exposed to magnesium sulfate and sodium sulfate solution. mass loss and compressive strength of samples were measured at different ages. Also, the expansion of prism samples was determined. Finally, the results indicated that undesirable effects due to sulfate attack is decreased by natural pozzolans replacement and the samples stability improvement depends on the properties of natural pozzolans and the amount of replacement [96]. Farrokhzad et al in 2016, examined the durability of concrete containing additives during sulfate attacks. They concluded that concretes containing micro silica and nano silica had the highest resistance against sulfate attacks [97]. Despite there is a few studies about the performance of concrete samples which cement is replaced by calcined clay more than 35 percent; Mwititi et al in 2018, evaluated the stability of concrete samples containing 40, 45 and 50 percent (percent by mass) calcined clay as cement replacement and activated with sodium sulfate. The results showed that chemically activated cements have more pozzolanic activity, less porosity, less setting time of concrete and high resistance in corrosive environments [98]. Akasha et al in 2018, investigated the effect of metakaolin replacement for cements

submerged in a 5 percent sodium sulfate solution. Sulfate durability of samples containing 0, 10, 15 and 20 percent of metakaolin replacement was evaluated by the expansion measurement test. They found that silica available in metakaolin reacts with the lime released during the hydration process and an extra C-S-H gel is produced. Although density of the extra C-S-H gel is lower than the former C-S-H gel, latter gel has an effective role in reducing the mortar permeability. Therefore, sulfate durability improvement of samples containing metakaolin is attributed to secondary C-S-H gel formation and portlandite consumption during the metakaolin pozzolanic reaction. The optimum metakaolin replacement rate for all mortars was 20 percent [99]. With the aim of the large scale and small scale study about concrete deterioration mechanisms exposed to sulfate media; Yu et al in 2018, investigated LC3 (Lime stone, Calcined Clay and Cement) cement performance under sulfate situation at large scale and small scale experimentally. The results showed that expansion rate, dynamic modulus and mass loss of LC3 cement in comparison with pure Portland cement are lower. They stated that limestone and calcined clay combination improves the pore size distribution of cement lead to minimize the ingress of sulfate ions and prevent the corrosive products formation; finally, the sulfate resistance of LC3 cement is increased [100]. Also, Shi et al in 2019, studied the LC3 cement sulfate resistance under a 0.13 molar sodium sulfate solution at 5 °C and 20 °C. calcined montmorillonite and metakaolin were used as calcined section of LC3 cement and the total replacement rate was 35 percent of Portland cement. Experiments results indicated that expansion due to sulfate attack was restricted

significantly [101]. Liu et al in 2020, evaluated the sulfate attack process by combining experimental methods and chemical thermodynamic analysis. The results were used to determine the relationship between temperature and Gibbs free energy. Also, the interaction of influencing factors on the sulfate attack (relative humidity, temperature, and sulfate ion concentration) was investigated. For example, the AFt phase is stable at a thermodynamic temperature of about 97 ° C, and the concentration of SO_4^{2-} of AFt phase is strongly influenced by temperature. However, in the stable temperature range, its concentration isn't less 2.8×10^{-3} mol / L [102].

5. Numerical studies of sulfate attack

Proper mix design of concrete for Long life and efficiency in adverse conditions is one of the most important preventive measures in aggressive environments. Determining service life parameters, proper selection of the type and amount of materials are some main steps in concrete designs for service. Appropriate modeling can help to pass these steps. Some criticism is pointed to the current testing methods for evaluating sulfate attack lately. These test methods succeeded to show the mechanisms related to the sulfate attack, but they aren't sufficient way in prediction of field performance. Skalny and Pierce [20] suggested that the existing testing methods for evaluating sulfate attack doesn't express properly the real field situation. A prerequisite for improving the quality of concrete evaluation tests is to consider all the conditions that the concrete structures actually experiences in the environment. In

other words, test conditions should be closer to the actual conditions. Efforts are needed to make more reliable models [1]. Cohen and Mather stated that it is necessary to lead the directions of studies and researches in the right way, in the first step [19]. They aimed to reach a clear picture of the concrete sulfate attack and develop more effective experimental methods for evaluating concrete sulfate attack using diverse criteria. For the first time, Grove and Wood in 1979, introduced a model considered diffusion and chemistry components separately. This model was able to predict the diffusion of sulfate salts in groundwater. The proposed method included two separate steps which are one dimensional ion movement and chemical reactions. The diffusion equations were initially solved without considering any chemical reactions, and then, in the next step, the chemical reactions included nonlinear ion exchange, dissolution, precipitation, ion complex formation and dissolution were solved using the substances concentration estimation of former step as an initial guess [103]. Another model was presented by Atkinson et al in 1988, which is based on the empirical relationship between the volume expansion and the amount of ettringite formation [104]. Saetta et al in 1993, proposed a general framework for assessing mechanical behavior of concrete under physical-chemical attack. The model takes into account the combined effect of moisture, temperature and chemical species but fraction and expansion due to chemical attack aren't considered. Concrete is a porous medium so, the penetration of corrosive species in concrete is under the influence of pore structure and moisture content. A numerical method was developed for solving differential equations governing the diffusion

of corrosive species, humidity and temperature based on finite element method by Saetta et al [105]. Pommersheim and Clifton in 1994, developed a model assuming that the volume changes due to chemical reactions result in the expansion of the cement sample and also, the cement expansion is related to the amount of ettringite linearly. However, no evidence has been found yet that the production of ettringite increases the cement expansion directly [106]. Casanova et al in 1997, proposed a geometric model that estimates the extent of internal sulfate attack caused by the reaction of oxidation sulfide. In many cases, Sulfur bearing aggregate is the main reason of internal sulfate attack occurrence. Oxidation of sulfur-bearing aggregate is caused to release the sulfate ions. Sulfate ions diffusion and the reaction of sulfate ions with C_3A leads to the internal sulfate attack occurrence. Oxidation occurs slowly. Factors such as solutions pH, abundant molecular oxygen, and aggregates size effect on the oxidation rate. The geometric model is a kind of the physico-chemical model which is included several steps such as assessment of sulfide amount available in aggregates, chemical kinetics model of sulfur oxidation and estimation of free sulfate ions amount, considering the formation of ettringite or monosulfoaluminate based on the related chemical reactions and finally, the total expansion calculation based on the volume changes due to oxidation and expanding phase formation reactions [107]. Truc et al in 2000, presented a multi-species diffusion model, which is used to simulate concrete stability exposed to chloride and sulfate situation simultaneously. They used a SNIA algorithm for diffusion and chemistry combination. An important distinction of the

model is consideration of the ionic interaction between several chemical species. For this purpose, the flux of each ionic species within the mortar is estimated by the Nernst-Planck equation and then they combined together [108]. Rothstein et al in 2002, utilized thermodynamic science to know better the process occurred in the cement paste and the composition of the pore solution using saturation index [109]. Another numerical model was developed by Shazali et al in 2006, to study the concrete deterioration under sulfate attack, but this model was only related to the gypsum formation [110]. Samson and Marchand in 2007, presented a numerical model for the ion diffusion mechanism explanation in the unsaturated cement system, but the mechanical properties changes due to fraction hadn't been considered [111]. Lottenbach et al, did a lot of studies on single thermodynamic modeling and thermodynamic modeling along with diffusion codes. They have been working on thermodynamic modeling of cement hydration since 2006 and the modeling of aggressive environments such as sulfate-rich environments, using the GEM software. Simulation results were verified by XRD and TGA experimental results [112-117]. In recent decades, considerable effort has been made to develop chemical- diffusion-mechanical models in order to prediction of the cement materials behavior under the external sulfate attack. These models are based on the main chemical reactions occurred during the deterioration process. In this regard, several theories were presented for evaluation of concrete properties that each of them has similarities and differences. Mori Tanak's theory is used to estimate the mechanical parameters such as elastic and

diffusion properties, Davis equations calculate the chemical activity coefficient, Corren's equation used for computing the crystallization pressure, and Mazars damage model used to simulate the fraction caused by crystallization pressure [118,119]. In some other papers, Delaunay/Voronoi tessellation theory as geometric pattern have also been used [120]. The 4SIGHT model is proposed with the aim of the concrete structures durability prediction. This model evaluates the serviceability of concrete samples due to the sulfate penetration using mechanical properties and diffusion [121,122]. Diffusion equation solution expression. The STADIUM method is applied for numerical modeling to evaluate the serviceability of concrete structures. It is a multi-species diffusion model consisted of damage mechanics and equilibrium calculations. In some researches, chemical reactions and ion diffusion are modeled using Fick's law [123]. Some other researchers found 3D modeling suitable for durability assessment because of that one-dimensional modeling is not able to taking into consideration all aspects [124]. Schmidt et al in 2009, investigated the sulfate durability of limestone-containing cements using thermodynamic modeling. The results showed that limestone has a physical effect on the sulfate durability of the blended cement, which means that it effects on the porosity of the cement. Small amounts of limestone in cement is caused the permeability and porosity reduction and thus sulfate durability is improved. But using more than 25 percent limestone increases porosity and subsequently decreases the sulfate durability of the blended cement [125]. Kunther et al in 2013, made a comparison between the volume changes predicted by thermodynamic modeling and

the samples expansion measured by experimental studies during the sulfate attack. The result showed that the mortars expansion (length changes) isn't related only to the volume increment of the sulfate-containing phases formation. Crystallization pressure theory is a possible mechanism for the expansion due to sulfate attack. When the crystals form in a supersaturated solution, the high pressure is applied on the surroundings, which causes the material expansion. Although, volume increment caused by sulfate-containing phases formation is one of the main reasons for the concrete expansion during sulfate attack, it can't be considered as the only factor caused volume changes due to sulfate attack [126]. Tarighat et al in 2016, investigated the chemical reactions and mechanism of sulfate attack using the thermodynamic models. The results indicated that a layer of brucite is formed near the sample surface in a magnesium sulfate solution which can reduce the sulfate ions penetration. 10 percent replacement of the slag reduces harmful phases such as gypsum and increases brucite and silica gel formation. Also, in another study, they investigated the effect of silica fume on sulfate durability of the blended cement. In the same year, they used thermodynamic modeling to better understanding of the hydration mechanism and chemical reactions for cement containing slag and silica fume. Various phases produced due to cement hydration were predicted properly using thermodynamic models [127-130]. Tarighat et al in 2016, used thermodynamic models to study the concentration changes of the pore solution for cement containing 20, 40 and 60 percent slag during cement hydration at 20°C. The thermodynamic calculations were used to measure the pore solution

composition with the aim of the identifying the formed phases under different conditions. Finally, the thermodynamic calculations showed that the use of slag in the blended cement leads to reduce calcium, alkali, hydroxide and sulfur at the early age which indicates the portlandite consumption during pozzolanic reactions and useful phases formation such as calcium silicate hydrate during hydration; ultimately resulting in the improvement of cement structural properties, performance and durability [131]. Tarighat et al in 2018, investigated the behavior of hardened cement paste during the external sulfate attack using thermodynamic simulation. The model was based on the minimizing Gibbs free energy method. Type and volume of the formed phases in the cement paste were investigated during the sulfate attack; in addition, other effective parameters on the concrete sulfate durability such as sulfate solutions concentration, cement chemistry, slag and wollastonite with or without silica fume effect and replacement percentages were evaluated. The results indicated that magnesium sulfate solution in comparison to other sulfate solutions caused the most serious adverse effects on concrete durability. Also, thermodynamic results indicated that below 40 percent of slag replacement didn't have favorable effects on concrete sulfate durability due to the increase of harmful phases formation, such as monosulfate and ettringite; however, over 60 percent of slag replacement leads to a good performance. In the same year, thermodynamic models were used to evaluate the hydration mechanism of cement containing slag with 10- 80 percent replacement, at a constant temperature of 20°C by Tarighat et al. The composition of the formed phases, the pore solution

concentration and chemical shrinkage of cement containing slag were investigated. The thermodynamic calculations showed that the use of slag in cement led to consumption of portlandite and increment of the calcium silicate content; in addition, the presence of slag in cement reduced the volume of pore solution and increased chemical shrinkage. Ultimately, slag use in the production of blended cement increased the total volume of hydration products and improved the structural properties, performance and durability of the cement paste [132-135]. Kunthera and Lothenbach in 2018, evaluated the sulfate durability of Portland cements containing 70 percent of the blast furnace slag in the presence of bicarbonate. The study aims to investigate sulfate durability of samples paste both experimentally and numerically. The results showed that calcium carbonate precipitation (Ca) decreased the possibility of CaO available for the formation of phases containing sulfate (ettringite and gypsum), on the other hand, it decreased the super saturation of ettringite in the pore solution. So, the presence of bicarbonate ions prevents the expansion of the both Portland cement and cements containing slag, and superficial deterioration of cements containing slag caused by the sulfate attack. The presence of bicarbonate which commonly found in normal water, is one of the main reason why common experimental methods related to concrete sulfate resistance evaluation are not always in good agreement with the real observations. Concrete deterioration intensity caused due to sulfate attack become less than what is expected from experimental studies because of presence of bicarbonate in water [126]. Cao et al in 2020, proposed a thermodynamic

model that could predict the hydration of sulfate and chloride cement by considering the kinetics in the short term. The results showed that the presence of NaCl, Na₂SO₄, and CaCl₂ in cement paste increases the hydration rate. The output of the thermodynamic model is able to predict hydration products during hydration time which can be generalized to simulate cement degradation processes such as sulfate attack [136]. Cao et al in 2020, investigated the interaction between sulfate and chloride on the cementitious material using thermodynamic simulations. Hydration simulation was performed by the GEMS software; the phase equilibrium and surface reactions were simulated using the geochemical model PHREEQC. Also, The Cemdata18 thermodynamic database was used to define the thermodynamic properties. The results showed that sulfate attack has a great effect on the production of Friedel's salt in Chloride-Sulfate environments. The presence of sulfates reduces the production of Friedel's salt and the adsorption of chloride by CSH [137]. Li et al in 2020, proposed a new approach based on thermodynamic studies, with the aim of better understanding the sulfate attack mechanism. The ions migration under the electric field during sulfate attack was modeled using the geochemical code GEMS. Ion migration could be analyzed using the results of the thermodynamic simulation. The migration of OH⁻ ions causes neutralization, which is the main reason for the ettringite decomposition [138].

A number of thermodynamic simulations about sulfate attack are listed in table 2. some necessities of thermodynamic modeling application are mentioned too.

Table 2: Summary of thermodynamic modeling application related to sulfate attack

Reason for using the thermodynamic models	Thermodynamic modeling results	Year	source
Investigate the conditions of thaumasite formation during sulfate attack	The prerequisite for thaumasite attack is that SO_3/Al_2O_3 ratio exceeds 3. Thaumasite is produced as one of the last phases of sulfate attack kinetically.	2008	[74]
Study the microstructural changes of the blended Portland cement exposed to sulfate solution	Determine the suitable amount of limestone additions (5 wt.%) to decrease the rate of sulfate ingress One of the main causes of thaumasite production can be concrete expansion which makes the microstructure of the concrete wider.	2009	[125]
Predict sulfate ingress in Portland cement using thermodynamic modeling coupled with transport codes	Ettringite formation within the matrix and the increase of crystallization pressure in small pores are the main expansion mechanism related to sulfate attack	2010	[115]
Modeling the experiments on prismatic cement paste cases [139] (only the flexural strength and expansions were determined experimentally because of complexity related to the experimental quantification of ettringite profiles)	Determine the expansion coefficient value for concrete sample exposed to sulfate solutions	2011	[120]
Study the mortar deterioration under sulfate attack	The presence of a mixture of different cations is one of the reasons why common experimental methods related to concrete sulfate resistance evaluation are not always in good agreement with the real observation	2013	[93]
Evaluation of mineral dissolution/precipitation of cement hydrates using coupled thermodynamic and kinetics model	Explain some important physical interpretations of concrete deterioration mechanism under the sulfate attack	2017	[140]
Investigate the interaction effect of sulfate and bicarbonate ions on cement exposed to magnesium sulfate solution	Waters containing bicarbonate ions are the reason for inconsistency between observations and experimental results	2018	[126]
Study the effect of wollastonite alone and the combination of wollastonite and silica fume addition on cement-based materials behavior exposed to sulfate solution and also saving in time and cost	Wollastonite alone isn't so useful for reducing the destructive effects due to sulfate attack, however the substitution of a part of cement by wollastonite (5%) and silica fume (10%) is highly effective	2018	[132]
A better interpretation of experimental test results	Al/Si ratio of C-K-A-S-H and C-A-S-H doesn't change greatly during sulfate attack, and increase of the pH hasn't much impact on Al/Si ratio changes for the C-K-A-S-H samples	2019	[83]
Thermodynamic modeling of the sulfate and chloride cement	An accurate thermodynamic model is proposed which could predict hydration products of the sulfate and chloride cement during hydration time. This model can be developed to simulate the sulfate attack of such cement.	2020	[136]
Effect of sulfate attack on the cement paste exposed to the chloride-sulfate environments	The interaction between sulfate and chloride on the cementitious material is analyzed	2020	[137]
Better understanding of the sulfate attack mechanism	New approach based on thermodynamic studies and ions migration under the electric field is proposed	2020	[138]

6. Conclusions

Unfavorable effects of sulfate attack on concrete structures isn't possible to repair simply. The experimental methods related to concrete sulfate resistance evaluation are efficient in the understanding of the sulfate attack mechanisms. However, they aren't suitable enough for concrete performance prediction under the real environmental conditions. It is difficult to simulate the real environmental conditions experimentally. In some cases, the same redox conditions in an experimental simulation as in the natural system are impossible. The sulfate attack resulted in a lot of undesirable effects on the concrete structures. The mechanism related to each of these effects individually can be studied experimentally. But it is important to note that the combination of two or more these effects makes complexities which can't be assessed by the conventional experimental methods related to concrete sulfate resistance evaluation because these conventional methods can consider one of these effects. Numerical studies, especially thermodynamic simulations, with appropriate adaptations to related experimental results, can be known as a suitable alternative instead of common experimental methods related to concrete sulfate resistance evaluation. In particular, in some cases, as it mentioned, the results of common experimental methods related to concrete sulfate resistance evaluation are not always in good agreement with the real observations, because the common experimental methods aren't able to simulate the all real environmental conditions properly. Therefore, numerical models, in particular, thermodynamic simulation as a fast and inexpensive solution, either alone or in combination with experimental studies, can be a desirable solution for enhancing the

ability to predict engineering behavior of concrete structures in the sulfate environment. On the other hand, gathering the results of the conducted studies on concrete sulfate attack and a closely look at various aspects of the results help to formation of the efficient framework for applying to simulate concrete structures performance exposed to the corrosive environments. It is proposed to apply the thermodynamic modeling to design the concrete mixing plan appropriate to different environmental conditions and predicts concrete structure performance. Also, unknown aspects of sulfate attack expansion mechanisms can be investigated by thermodynamic simulation.

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