Experimental Approach on Sulfate Attack Mechanism of Ordinary Portland Cement Matrix: Part I. Sodium Sulfate Attack

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Abstract

This paper introduces a study carried out to investigate sodium sulfate attack caused by various reactive products. Experiments were performed on mortar and paste specimens made with ordinary Portland cement (OPC) conforming to KS L 5201 Type I. The water-cement ratios were varied from 0.35 to 0.55. It was found from the laboratory study that the water-cement ratio may be a key to control the deterioration of OPC matrix during sodium sulfate attack. Furthermore, X-ray diffraction (XRD) confirmed that ettringite, gypsum and thaumasite were the main products formed by sodium sulfate attack. These findings were well supported by thermal analysis through differential scanning calorimetry (DSC), and confirmed the long-term understanding that deterioration mechanism by sodium sulfate attack is a complicated process. Most importantly, deterioration due to sodium sulfate attack is characterized as the drastic reduction in compressive strength as well as the expansion (especially in cement matrix with a higher water-cement ratio).

Keywords : sodium sulfate attack, ordinary Portland cement, ettringite, gypsum, thaumasite

1. Introduction

This paper is a part of experimental researches on the deterioration mechanism of cement matrix, particularly ordinary Portland cement matrix, subjected to sulfate attack, and is related to deterioration characteristics due to sodium sulfate attack.

There are many causes of hardened cement-based materials' degradation such as exposed to carbonation, alkali aggregate reaction, freezing-thawing cycles, seawater attack and sulfate attack. In particular, deterioration of concrete structural components exposed to soils and underground water contaminated with sulfate salts is a serious problem in durability of concrete. A number of hypotheses have been proposed to describe the mechanisms responsible for deterioration of concrete by sodium sulfate attack. Wee et al.[1] stated the role of mineral admixtures to mitigate sodium sulfate attack through an immersion test for 32 weeks. However, they failed to detect ettringite formation by XRD and showed somewhat ambiguous microstructural results.

Torii et al.[2] reported that $Ca(OH)_2$ content measured by DSC-TG curves significantly influences on sodium sulfate attack of high-volume fly ash concrete. Similarly, according to ACI Building Code 318[3], one of the reasons for recommending the replacement of pozzolanic materials for increasing sulfate resistance is to remove excessive $Ca(OH)_2$ from the hydrated cement paste.

Based on the work by Irassar et al.[4], the sulfate resistance of concrete buried in soil contaminated with sulfate substances depends mainly on a high capillary suction of concrete. He also suggested that, under this environment, blended cement concretes containing high-volume slag could be more effective. Verbeck[5] reported that severity level as well as C_3A and C_4AF content may determine the extent of sodium sulfate attack. Numerous field case studies on sulfate-generated distressed concrete structures are currently in progress[6-8].

Many researchers have tried to manifest this complex phenomenon through immersion tests in laboratory[9,10] as well as the field cases. However it is difficult to reflect completely on the complexity of sulfate attack on the deterioration in field concrete through laboratory tests, because of their different exposure conditions. Similarly, Mehta[11]

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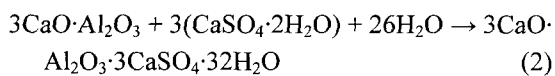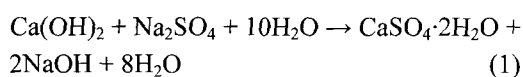Tel.: +82-2-2290-0323   Fax.: +82-2-2293-9977
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noted that there are several gaps in the knowledge of sulfate attack mechanisms. And he also insisted in his another literature[12] that the permeability of concrete rather than cement chemistry should appear to be the most important factor in sulfate attack. In this study, authors tried to evaluate the deterioration mechanism of cement matrix by sodium sulfate attack through laboratory immersion test. Data presented in this paper include compressive strength monitoring of cubic mortar specimens of 50 mm dimension and expansion of prismatic mortar specimens prepared according to ASTM C 1012. Additionally, XRD and DSC analysis were performed on the paste samples subjected to 5% sodium sulfate solution and/or tap water after 510 days.

2. Deterioration modes of sodium sulfate attack

Sulfate attack on concrete is destructive and corrosive process. It is caused by the formation of expansive salts within concrete structures that are exposed to sulfate environment. The damage is usually believed to be caused by each of two major sulfate reactions: (1) The sulfate ions react with C_3A and its hydration products to form ettringite with increase in volume, which results in expansion and subsequent cracking of the concrete; (2) The sulfate ions react with calcium hydroxide to form gypsum. It is generally accepted that gypsum formation is to be harmful because of its softening effect, but the specific mechanism is not well understood. Similar suggestions[13,14] have also been reported previously in their publications.

Generally, the principle chemical processes of formation of gypsum and ettringite by sodium sulfate attack may be displayed as equation (1) and (2), respectively.

$$Ca(OH)_2 + Na_2SO_4 + 10H_2O \rightarrow CaSO_4 \cdot 2H_2O + 2NaOH + 8H_2O \qquad (1)$$

$$3CaO \cdot Al_2O_3 + 3(CaSO_4 \cdot 2H_2O) + 26H_2O \rightarrow 3CaO \cdot Al_2O_3 \cdot 3CaSO_4 \cdot 32H_2O \qquad (2)$$

The mechanisms of sulfate attack in sodium sulfate environment have been also reported by Al-Amoudi et al.[15] and Wee et al.[1] In summary, these mechanisms follow equations (1) and (2), and they are correlated directly with content of C_3A and calcium hydroxide. Above authors also suggested that the expansion would be the most dominant factor with respect to durability of sulfate attack, especially when concrete is subjected to sodium sulfate attack.

For further investigation by Tian and Cohen[16] on the effect of expansion on the sulfate deterioration, alite cement pastes, which have a very little amount of Al_2O_3, were immersed in sodium sulfate solution of 33,800 ppm (5%, by weight) for 480 days. They revealed that expansion of the paste may be attributable to the formation of secondary gypsum, but not that of ettringite. This was obviously confirmed by their XRD examination.

In general, in terms of material project, thus the superior performance of concrete against sodium sulfate attack needs the following ways:

1) low w/c and a small content of C_3A and C_3S
2) using the mineral admixtures such as silica fume and GGBFS to reduce the calcium hydroxide content liberated in the cement paste
3) reducing the pH value in pore solution and making more impermeable microstructure

In the present study, authors suggest two deterioration modes of cement matrix associated with sodium sulfate attack. The first mode of deterioration, as shown in Fig. 1 (a), which is commonly characterized by expansion and cracking, can be observed in cement matrix when the reactive aluminate phases available, present in sufficient quantities, combine with sulfate ions, thereby forming ettringite in pore spaces. The characteristics of ettringite formation are changeable with the condition of pore solution. In other words, in high pH environment, the ettringite is stable and shows an expansive characteristic. The second mode of deterioration by sodium sulfate attack is the onion-peeling type (Fig. 1 (b)). This mode results from scaling and peeling-off of the surface in successive layer, and is related to the formation of a large amount of subhedral gypsum tabulae in cracks produced by delamination. Actually, this mode type was visibly observed in OPC mortar specimens immersed in 5% sodium sulfate solution after about 2 years.

3. Experimental details

3.1 Materials

KS L 5201 Type I OPC was used in preparing cement paste and mortar specimens. The chemical composition and mineralogical components of the cement are shown in Table 1.
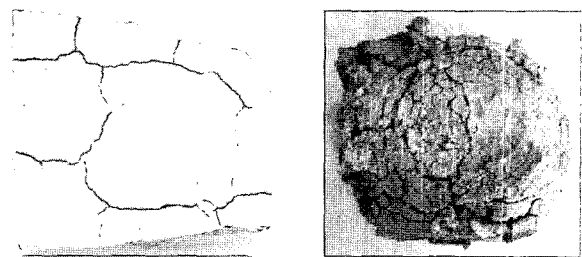
(a) Expansive type   (b) Onion-peeling type
Fig. 1 Deterioration modes of cement matrix due to sodium sulfate attack

River sand, with a maximum size of 5mm, was used as fine aggregate. The specific gravity, absorption and fineness modulus of the fine aggregate are 2.60, 0.80% and 2.80, respectively. The weight ratio of fine aggregate to cement was 2.0. In addition, superplasticizer (SP) used in manufacturing mortar specimens was a polycarbonic acid-based chemical admixture. The SP was incorporated in the mortar mixture proportions with water-cement ratio of 0.35 and 0.45, and added to the mixing water at a level of 0.5% by mass of total cementitious materials. It was well stirred until completely dissolved before addition to the mortar mixtures.

3.2 Test solution

The 5% sodium sulfate solution used for the immersion test was refreshed every 4 weeks to minimize the increase in pH due to the leaching of OH⁻ ions from the paste and mortar specimens. The temperature of test solution was kept at 20 ± 1 ℃ during test period. For comparison, tap water, kept at a similar temperature, was used as a reference solution for control samples. The test solution and specimens were kept in plastic containers with enough space among the specimens. The test solution used to provide sulfate attack to the cement matrix were made by dissolving regent grade chemicals in tap water.

3.3 Specimen preparation

The mixture proportion of mortars is cement : sand = 1 : 2. Mortar specimens were made at water-cement ratio of 0.35, 0.45 and 0.55. The mixing procedure of mortar followed a method based on KS L 5105. After 1 day from casting, the mortar specimens were demoulded and then initially cured in tap water for additional 6 days.

Table 1 Chemical composition and mineralogical components of cement

Chemical composition (%)	
SiO₂	20.2
Al₂O₃	5.8
Fe₂O₃	3.0
CaO	63.3
MgO	3.4
SO₃	2.1
L.O.I	2.1
Mineralogical components (%)	
C₃S	54.9
C₂S	16.6
C₃A	10.3
C₄AF	9.1

The water-cement ratio of paste specimens was fixed at 0.45. The paste samples were made using 100g of total cement and 45g of diluted water. Some efforts were made to avoid segregation. After hand-mixing, the pastes were rotated for 8 hours. All pastes were cast into plastic cylinder with 10 mm diameter and then demoulded after 1 day. Similarly, after demoulding, initial curing of paste samples in tap water was for additional 6 days. The paste samples were coated using epoxy except upper side. Microstructural analyses were performed on the deteriorated part of upper side of the paste samples.

3.4 Test details

3.4.1 Visual examination
Visual appearance of 50mm cubic mortar specimens immersed in sodium sulfate solution was periodically examined to check spalling, cracking and expansion, in addition to mass loss by chemical attack oriented from sodium sulfate solution.

3.4.2 Compressive strength
The deterioration of 50mm cubic mortar specimens was evaluated by measuring the compressive strength loss after predetermined periods of immersion to sodium sulfate solution. Companion specimens cured in tap water were also tested along with the specimens immersed in sulfate solution. The compressive strength loss was determined using the following relationship.

$$\text{Compressive strength loss (\%)} = [(f'w - f's) / f'w] \times 100$$

where, $f'w$ is the average strength of specimens cured in tap water and $f's$ means the average strength of specimens immersed in sodium sulfate solution. Three mortar specimens from tap water and sodium sulfate solution were tested at each test period.

3.4.3 Expansion
Expansion measurements of prismatic mortar specimens, 25×25×285mm size, were performed according to ASTM C 1012 and measured on three mortar specimens withdrawn from sodium sulfate solution. The average value was adopted.

3.4.4 X-ray diffraction (XRD)
In order to examine the products formed by sodium sulfate attack as well as hydration, the cement pastes with a water-cement ratio of 0.45, which had been pre-cured for 7 days, was investigated after 510 days of immersion or curing. The XRD was conducted as using the RINT D/max

2500 (Rigaku) X-ray diffractometer. For the detailed items of XRD, CuKα radiation with a wavelength of 1.54□ at a voltage of 30 kV, scanning speed of 2 °/min. and current of 30 mA were used.

3.4.5 Differential scanning calorimetry (DSC)

The cement paste sample powder for DSC was collected from the surface of paste specimens exposed to sodium sulfate solutions for 510 days and then ground in the presence of acetone in a ceramic vessel. The powder samples passed through a No. 100 sieve were put in the specific aluminium pan for testing in the thermal analysis system. DSC test was conducted to recognize the products by hydration and chemical reaction and carried out by using DSC 2010 Differential Scanning Calorimeter (TA Instruments, U.S.A). Amount of paste sample powder used was approximately 10mg. The powder sample was heated from ambient temperature to 600℃ at a rate of 10℃/min. in atmosphere of nitrogen. The DSC quantitative analysis is based on determining the peak of each phase on the DSC curve. The starting and ending points of each dehydration peak on the DSC curve are defined as the points where the gradient of the curve changes abruptly. These points were determined by inspection and with the help of the second derivative curves, which were automatically produced by the instrument's software.

4. Experimental results

4.1 Visual examination

OPC mortar specimens were periodically retrieved from sodium sulfate solution and visually inspected to characterize the deterioration. Based on Al-Amoudi's work[17], the ratings to evaluate visually the degree of deterioration in tested mortar specimens are shown in Fig. 2. The visual ratings are divided into six stages. The visual examination were regularly carried out to investigate the visible softening, cracking, spalling and expansion in mortar specimens by sodium sulfate attack.

Fig. 3 presents the visual ratings of mortar specimens with immersion period. As expected, the mortar specimens made with a low water-cement ratio showed low visual rating regardless of immersion period.

For mortar specimens with a water-cement ratio of 0.55, it should be noted that the deterioration of them was significantly pronounced even at the early age of immersion, which indicated 4 level of the visual rating after 180 days of immersion, but no deterioration in mortar specimens with a water-cement ratio of 0.35. Finally, the mortar specimens (cubic) with a water-cement ratio of 0.55 showed consider-

able mass loss and then totally disintegrated after 360 days of immersion. Hence, it is clear that water-cement ratio may be a key to control the deterioration of OPC matrix during sodium sulfate attack Practically, the visual ratings investigated in this study showed a reliable consistence relationship with mass loss by sodium sulfate attack.

4.2 Deterioration of mortar specimens due to sodium sulfate attack

Compressive strength of mortar specimens cured/ immersed in tap water or sodium sulfate solution was regularly measured. The results of the compressive strength loss obtained in this study are shown in Fig. 4.
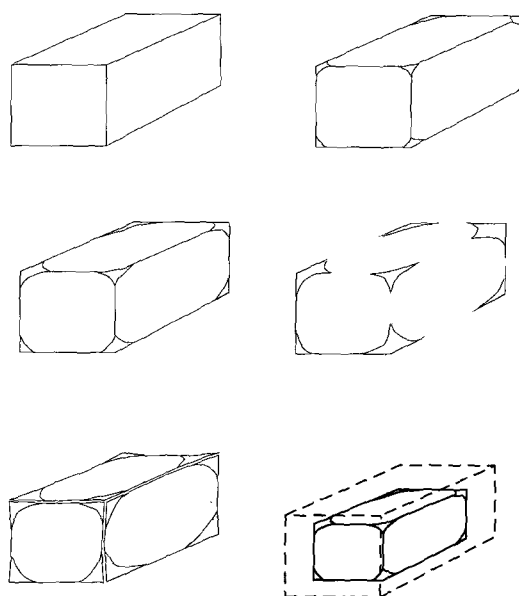

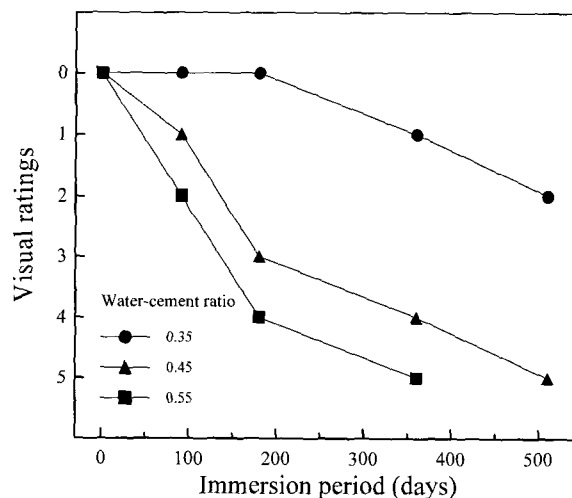**Fig. 2** Visual ratings of deterioration due to sulfate attack

Fig. 3 Visual ratings of mortar specimens exposed to sodium sulfate solution with immersion period

The data in this figure show that the compressive strength losses of mortar specimens were gradually increased after 28 days of exposure. In particular, mortar specimen with water-cement ratio of 0.55 showed a remarkable increase in compressive strength loss. At the 28 days of exposure, negative values of compressive strength loss were also observed. These data signify that the compressive strength of mortar specimens exposed to sodium sulfate solution was greater than those cured in tap water for the same period. It is thought that the negative values in compressive strength loss are attributed to the filling up to the pore space by the expansive product, densifying the mortar matrix in the early period exposure.

After 510 days of exposure, the compressive strength loss of mortar specimens was about 37.5, 63.7 and 91.7% for water-cement ratio of 0.35, 0.45 and 0.55, respectively. The significant strength loss of mortar specimen with a water-cement ratio of 0.55 has a good agreement with visual ratings shown previously. Actually, the cracking and spalling observed in the mortar specimens exposed to sodium sulfate solution led to the reduction in cross-section of it. However, mortar specimens with a low water-cement ratio exhibited visually less or negligible deterioration level up to 180 days of exposure because of their low permeability of SO_4^{2-} ions into the mortar matrix.

Expansion of mortar specimens tested in present study was measured on the basis of ASTM C 1012. The expansion of OPC mortar specimens with different water-cement ratio stored in sodium sulfate solution was presented in Fig. 5. It was clearly evident that mortar specimen with a higher water-cement ratio of 0.55 showed a significant expansion (approximately 0.817% after 180 days of exposure) in the solution. For mortar specimen with water-cement ratio of 0.35, expansion value was as small as about 0.132% even at 510 days of exposure. The low permeability of the mortar specimen due to a low water-cement ratio restricted the diffusion of SO_4^{2-} ions forming the expansive products. Both mortar specimens with water-cement ratios of 0.45 and 0.55 had disintegrated at 210 days and 180 days of exposure to sodium sulfate solution, respectively, whereas mortar specimen with a water-cement ratio of 0.35 showed a relatively low expansion at the same ages.

Most importantly, the data shown in Fig. 4, 5 confirm that the water-cement ratio is a key factor in the deterioration of mortar specimens exposed to sodium sulfate attack.

4.3 Microstructural observations

Changes in mineralogy of OPC paste samples with continued curing/exposure to water and sodium sulfate solution for 510 days were examined by XRD.
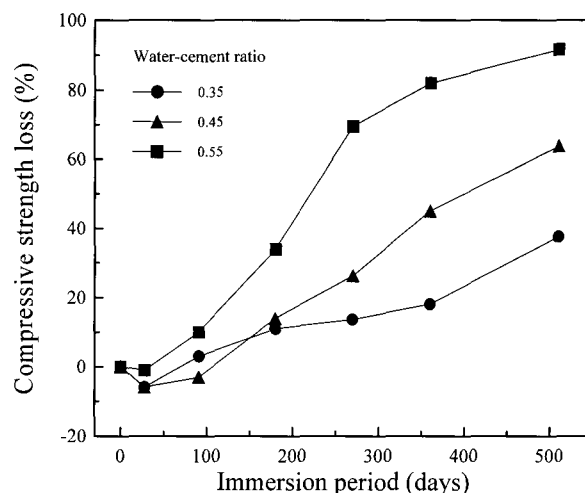

Fig. 4 Compressive strength loss of mortar specimens exposed to sodium sulfate solution with immersion period
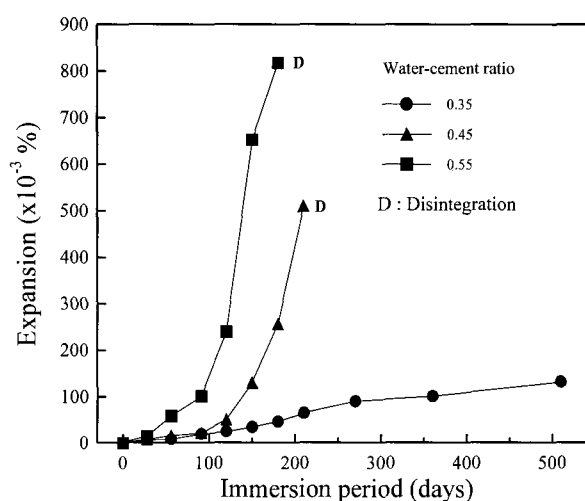

Fig. 5 Expansion of mortar specimens exposed to sodium sulfate solution with immersion period

Fig. 6 shows that there are remarkable differences between formed products due to hydration and sulfate attack. The mineralogy of paste sample cured in water (Fig. 6 (a)) corresponded with that of a normal hydrated Portland cement paste containing portlandite, ettringite and calcite, along with a small amount of gypsum. Strong peaks at 18.1°, 34.0° and 28.7° 2θ indicated the presence of portlandite due to cement hydration. On the other hand, XRD pattern of powder obtained from the paste sample exposed to sodium sulfate solution (Fig. 6(b)) revealed the coexistence of ettringite and thaumasite at 9.1° and 16.0° 2θ. In fact, thaumasite is often indistinguishable from ettringite due to their nearly identical XRD pattern.[18]

There is still a subject of controversy with respect to the similarities in structure of both ettringite and thaumasite. Thus, a discussion of this is beyond the scope of this paper.

Comparing two XRD patterns, it was observed that the

dramatic reduction (18.1° and 34.0° 2θ) or disappearance (28.7° 2θ) in peak intensity of portlandite is attributed to the formation of significant ettringite and gypsum.

The solid phases detected in XRD patterns of OPC paste exposed to 5% sodium sulfate solution after various immersion periods were presented in Table. 2. The following peaks were used as indicators for different solid phases in XRD patterns: ettringite (9.1° 2θ), gypsum (11.7° 2θ), thaumasite (9.2° 2θ) and portlandite (18.1° 2θ).

This observation is well supported by the proof thrown from DSC analysis of paste sample exposed to sodium sulfate attack for 510 days, as shown in Fig. 7. This curve shows four prominent endothermic peaks at around 85, 125, 145 and 462 ℃. The endotherm at 85 ℃ was attributed to dehydration of water in the paste sample powder. Although the endothermic peak for thaumasite is commonly accepted at 115 ℃, the endotherm at 125 ℃ was for ettringite and thaumasite, indicating the enthalpy of 157.9 J/g. Thermal analysis peak temperatures vary according to the apparatus and the amount of the phase present. As expected, a small a- mount of portlandite remained in the paste sample powder, showing the weak endothermic peak at around 462 ℃. At that temperature, the enthalpy was as small as about 30.87 J/g.

5. Further discussion

Data obtained from XRD and DSC confirmed that deterioration of OPC paste and mortar specimens is a result of the formations of ettringite, thaumasite and gypsum. Numerous authors[1,2,9-11,15] have reported that the deterioration by sodium sulfate attack oriented from the reaction with sulfate ions and hydration products such as calcium hydroxide and calcium silicate hydrate (or aluminum silicate hydrate). In the literatures, ettringite and gypsum have been focused on the main reactants from laboratory studies as well as field case investigations.

It is absolutely true that sulfate-induced expansion of cement matrix is attributable to the formation of ettringite. This process involves the reaction of tricalcium aluminate (C_3A). If the pore fluid becomes depleted with respect to the sulfate ions before the C_3A and its hydration products are completely combined, then sulfate ions from ettringite are probably apt to go into the pore fluid and react with remaining C_3A and proceed to form monosulfate. Once available sulfate ions from external source, for example sodium sulfate solution, are brought in the cement system, ettringite may be formed again.
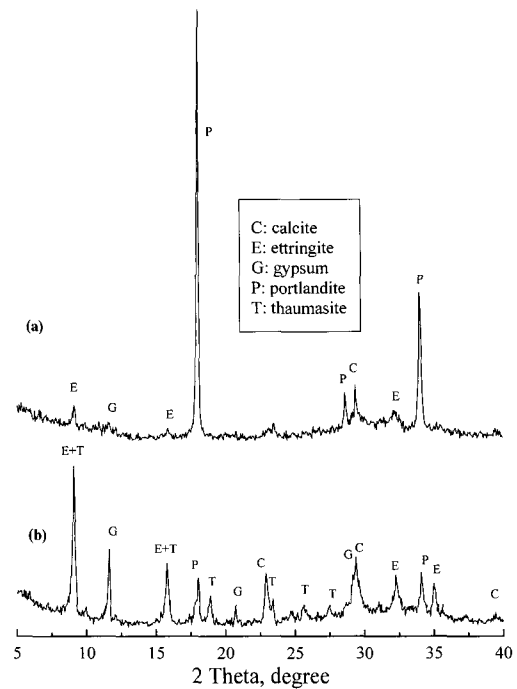

Fig. 6 XRD patterns of paste samples ((a) cured in water, (b) immersed in sodium sulfate solution for 510 days)

Table 2 Solid phases detected in XRD pattern of OPC paste exposed to sodium sulfate solution

Imm. period	Ettringite	Gypsum	Thaumasite	Portlandite
91 days	II	I	I	IV
180 days	III	II	I	III
360 days	IV	IV	II	I
510 days	IV	IV	III	I

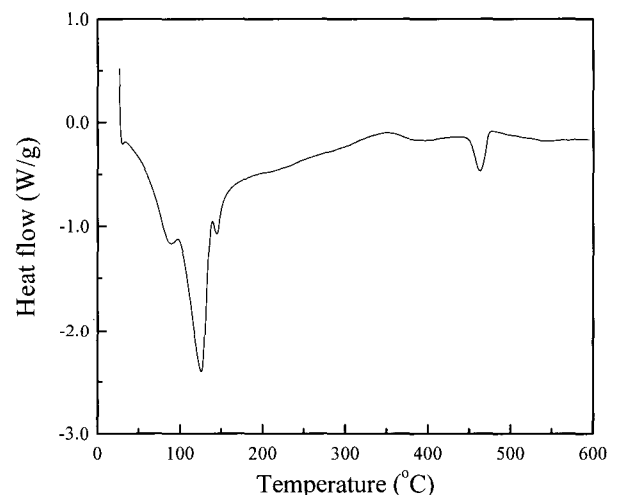0: negligible, I: weak, II: middle, III: strong, IV: very strong

Fig. 7 DSC curve for paste sample exposed to sodium sulfate solution for 510 days

Continued formation of ettringite within confined pore or solid causes internal stress, particularly bending stress, leading to expansion and subsequent cracking.[19] The formation also results in the negative values in terms of strength loss at the early immersion age due to effect of filling-up on pore space, as shown in Fig. 4. Gypsum formation, leading to softening type of sodium sulfate attack, has also reported elsewhere. Mehta[20] stated that deterioration of cement matrix by gypsum involves a process leading to reduction of stiffness and strength of the system.

Several methods for protection and maintenance of concrete against sulfate attack have been suggested elsewhere[9,12,14,15]. With respect to cementitious material, research related to concrete deterioration due to sulfate attack resulted in the development of the sulfate resisting Type V cement[21]. However, the chemical composition of ordinary Portland cements has undergone several changes resulting in a significant increase in the C_3S content to achieve a rapid development of strength. Unfortunately, this change renders cement matrix prepared with even Type V cement vulnerable to sulfate attack for the reason of production of significant calcium hydroxide content.[22] This consideration has led to a further observation of the possibility of utilizing mineral admixtures, such as silica fume,[23] fly ash[1] and ground granulated blast-furnace slag[24] for enhancing the sulfate resistance of cement.

From the visual ratings reported in section 4.1, it is obvious that the mortar specimens with a water-cement ratio of 0.35 that had been stored in sodium sulfate solution showed little or negligible deterioration up to 180 days of exposure, whereas a significant deterioration, corresponding 4 level of visual ratings was exhibited in the mortar specimens with a water cement ratio of 0.55 after the same immersion. This is because a great amount of products containing SO_4^{2-} ions was formed in the cement system, which was obviously confirmed by microstructural observations presented in Section 4.3. Actually, the long-term investigation worked by Brown and Hooton[25] reported that the portland cement concrete exposed to sodium sulfate solution for 21 years was greatly deteriorated due to the formation of ettringite and thaumasite, but not the formation of gypsum. In addition, they insisted that the interaction of Al and Si ions released from ettringite and thaumasite would take place in the region showing mixtures of them. Thus, the expansion shown in Fig. 5 may be responsible for the both formations of ettringite and thaumasite. On the other hand, as presented in previous literature,[23] the compressive strength loss of the mortar specimens seems to be related to gypsum formation as well as the both products. This is a good agreement with Al-Amoudi et al.[15]. However, many publications have overlooked the effect of thaumasite sulfate attack (TSA) on

the strength loss. Actually, the strength loss has been linked to decalcification of the cement paste hydrate, in particular C-S-H, or sulfate attack on C-S-H in the presence of carbonate ions to form thaumasite. The thaumasite formation is accompanied by strength loss and results in transformation of hardened cement matrix into a friable mass since a significant part of C-S-H can be destroyed.

6. Conclusions

This paper shows a detailed study on the process of deterioration and the formation of reactants by chemical reaction of ordinary Portland cement mortars and pastes exposed to 5% sodium sulfate solution.

From the results of variation of engineering properties such as visual rating, compressive strength loss and expansion, it can be confirmed that water-cement ratio may be a key to control the deterioration of cement matrix during sodium sulfate attack. The capability for higher absorption of sulfate ions into cement matrix with a high water-cement ratio may explain the severe deterioration of the cement matrix.

More importantly, microstructural observations such as XRD and DSC revealed that the deterioration of the cement matrix resulted from the formation of ettringite, gypsum and thaumasite. However, more detailed investigations on the structural difference between ettringite and thaumasite are needed in the future. In practice, the results of XRD analysis with immersion period confirm that calcium hydroxide content must be reduced in the cement matrix for further improving durability against sodium sulfate attack.

Conclusively, the mechanical and microstructural deterioration of ordinary Portland cement matrix was a result of the formation of products containing SO_4^{2-} ions. These products eventually led to the expansion, strength reduction and cohesiveness loss in the cement system.

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