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The effect of $MgSO_4$ on thaumasite formation

Zanqun Liu [a,c,*], Dehua Deng [a,c], Geert De Schutter [b], Zhiwu Yu [a,c]

[a] School of Civil Engineering, Central South University, Changsha, Hunan 410075, PR China
[b] Magnel Laboratory for Concrete Research, Department of Structural Engineering, Ghent University, Ghent 9052, Belgium
[c] National Engineering Laboratory for High Speed Railway Construction, Changsha, Hunan 410075, PR China

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ABSTRACT

Normally, $MgSO_4$ is considered to promote thaumasite sulfate attack (TSA) due to pH drop of solution caused by insoluble brucite formation. However, some test results indicate that thaumasite formation (TF) should occur in the solution with relatively high pH.

In the current paper, cement, cement + fly ash, cement + limestone powder and high sulfate resisting (HSR) cement pastes were immersed in sealed bottles containing Na_2SO_4:$MgSO_4$ = 0:5, 1:5, 3:5 and 10:30 at 10 °C. The pH was measured and corrosion products were identified by means of XRD. The results indicated that the existence of relatively low content $MgSO_4$ (even 5%) may not cause significant pH drop under non-exposed condition. The pH was still kept at the level of 13.0, while the increase of $MgSO_4$ could promote TF. The pH fell below 10.0 in the solution containing saturated $MgSO_4$, however, gypsum dominated the mechanism of paste deterioration.

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1. Introduction

As generally known, $MgSO_4$ not only reacts with calcium hydroxide but also leads to the decomposition of the C–S–H phase, forming not only expansive gypsum but also unbound products of brucite and magnesium silicate hydrate (M–S–H). $Mg(OH)_2$ (brucite) is insoluble and its saturated solution has a pH of 10.5. Thus, generally, it is agreed upon that the damage from $MgSO_4$ attack on concrete is more severe than from Na_2SO_4 attack [1–3].

Thaumasite sulfate attack (TSA) was identified as the major mechanism for the extensive and sensitive deterioration of deeply buried foundation concrete of a number of 30-year-old M5 motorway bridges in Gloucestershire, west England in spring, 1998 [4–9]. Hobbs and Taylor analyzed these field cases of TSA [10]. They indicated that the oxidation of pyrites within the backfill leads to the formation of sulfuric acid, reducing the pH of the groundwater. Magnesium ions and the formation of insoluble $Mg(OH)_2$ also reduce the pH, increasing the degree of chemical attack on the C–S–H, playing a major role in increasing the risk of thaumasite attack [11]. Sharp et al. [12] compared the thaumasite formation (TF) of Portland-limestone cement pastes in different sulfate solutions, the results indicated that the storage in the $MgSO_4$ and Na_2SO_4 mixed solution behaves in a similar scaling manner to simple $MgSO_4$ solution. This suggests that magnesium ions plays a considerable role in the TF due to the generation of low soluble $Mg(OH)_2$, resulting in the drop of pH and the disintegration of C–S–H. In summary, a low pH storage condition due to the existence of $MgSO_4$ favors TF.

However, a number of reports show contrary results. Jallad et al. [13] studied the stability of pure thaumasite samples in solutions with different pH ranging from 6.0 to 13.0. The results indicated that thaumasite reacts with the ions present in the solutions, and some conversions to calcium phosphate, calcium silicate and calcium carbonate are detected at low pH levels ($\leqslant$11.0). At pH level >11.0, minor amounts of calcium carbonate are generated, while only in highly basic conditions (pH = 13.0) thaumasite is stable. Zhou et al. [14] studied the role of sulfuric acid in thaumasite sulfate attack. The results showed that the existence of acid cannot promote TF. TSA occurs in the sulfate solution at pH above 12.0. The pH of acidified solution is maintained between 2.0 and 6.0, gypsum is the dominating reactive phase in the solution. Hagelia pointed out thaumasite decomposes and forms gypsum and calcite at low pH level [15,16]. The experimental results attained by Gaze and Crammond [17] presented that, after 90 days exposure to $MgSO_4$ solution under "non-exposed" condition (separated from atmosphere), calcite and gypsum are the main reaction products although the pH is 12.5. Only in the K_2SO_4 solution with pH of 13.0 thaumasite is the predominant product. Therefore, the above experimental results support that TSA plays a major role in cementitious materials deterioration only in the sulfate environment with high pH close to 13.0.

Obviously, the above two views are conflicting. The former view indicates that the pH drop of solution by $MgSO_4$ favors TF, and the increase of $MgSO_4$ can promote TSA. However, the latter view

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* Corresponding author at: School of Civil Engineering, Central South University, Changsha, Hunan 410075, PR China.
E-mail address: liuzanqun_2001@hotmail.com (Z. Liu).

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concludes that high pH is an essential factor for TSA. In the current paper, tests were carried out to try to explain the differing views and any possible connection between them.

2. Experiments and raw materials

Concerning the pH of solution, it should be noted that the pH of sulfate solution is controlled by the combined effects of three factors: the Mg^{2+} content, the replace rate of fresh solution and the relative volume ratio of concrete to solution [18]. Also, the storage condition is also an important factor. In the abovementioned report [17], Gaze and Crammond compared the pH of the same $MgSO_4$ solution or K_2SO_4 solution under exposed and non-exposed storage conditions respectively. The results showed that under non-exposed condition the pH of solution remains at a high level (>12.5) during the whole process, but the pH drops to 6.0 or 8.1 if the solution is in contact with air (exposed condition).

Concerning the field experience of M5 motorway bridges, an interesting appearance should not be overlooked that TSA was just found on the top of deeply buried foundation [4,5,7,8]. Combining the relationship between groundwater and foundation [19–21], this means that TSA just occurs in a perched solution formed on the top of concrete foundation, and the perched solution is stagnant and not frequently replaced by fresh solution. The volume of solution is much less than the concrete foundation. The foundations are deeply buried underground and far separated from atmosphere. This can be regarded as a kind of non-exposed storage condition.

In order to simulate such field exposure conditions, in the current paper the specimens were orderly organized in prismatic plastic bottles with two covers. Sulfate solutions just enough to cover the surfaces of specimens were irrigated into the bottles respectively, and not replaced during the whole progress. The pH of solutions was measured and corrosion products were identified by means of XRD.

Table 1 gives the chemical composition of ordinary Portland cement, high sulfate resisting cement (HSR), fly ash (FA) and limestone powder (LP) used in this study. Four pastes with different porportions were made (Table 2). Four solutions with different proportions of Na_2SO_4 to $MgSO_4$ (Na_2SO_4:$MgSO_4$ = 0:5, 1:5, 3:5 and 10:30 by mass) were used. The exposure temperature was kept at 10 °C.

Firstly, specimens were cast and demolded after 24 h, then cured in water at 20 °C for 28 days. The specimens were then dried in a climate room at 20 ± 2 °C and 60 ± 5% RH for 1 week, and moved into four plastic bottles with two tight-fitting lids. Sulfate solutions were irrigated into bottles. At last, the sealed bottles were placed in a refrigerator at 10 °C.

Specimens were taken out for visual observation and XRD analysis at the exposure ages of 1, 3 and 6 months. The outer layer of the specimen was cut off by a knife and grinded to pass a 53 μm sieve; the powder was dried in a vacuum container with silica gel for XRD analysis. The pH of the exposure solution was measured.

Table 1
Chemical composition of cement, fly ash and limestone powder.

Code (%)	C(I) CEM I 52.5	C(III) (HSR) CEM I 42.5 NHSR LA	Fly ash (FA)	Limestone powder (LP)
SiO_2	19.60	19.94	53.31	0.86
Al_2O_3	4.90	3.13	26.43	0.08
Fe_2O_3	3.10	4.76	7.53	0.34
CaO	63.60	61.56	4.46	56.3
MgO	0.90	–	2.54	0.58
K_2O	0.77	0.66	3.58	0.05
Na_2O	0.41	0.24	1.15	0.08
SO_3	3.30	2.54	0.90	–
LOI	2.10	5.45	4.10	42.0

Table 2
Mixture proportions of cement paste.

	Cement	Fly ash (FA)	Limestone power (LP)	W/C
Cement	1(C I)	–	–	0.5
Cement + FA	0.75(I)	0.25	–	0.5
HSR	1(C III)	–	–	0.5
Cement + LP	0.75(I)	–	0.25	0.5

3. Results

3.1. Visual observation

Figs. 1–3 show the visual appearance of pastes after 6 months immersion. In Fig. 1, after 6 months immersion in the Na_2SO_4:$MgSO_4$ = 3:5 solution it can be found that: (1) the HSR pastes looked sound; and (2) the outer layers of cement, cement + FA and cement + LP were both softened and white substance was formed on the surfaces.

Compared with Fig. 1, after 6 months exposure to 5% $MgSO_4$ solution it can be observed in Fig. 2 that: (1) the HSR pastes were still sound; (2) there was not a significant change of cement + LP; and (3) the edges of cement and cement + FA specimens were severely eroded. Large mass loss could be found, with cement + FA likely showing the most severe loss.

In Fig. 3, in the Na_2SO_4:$MgSO_4$ = 10:30 mixed solution it can be found that: (1) the HSR paste, which showed the best performance in Figs. 1 and 2, was severely damaged. A layer of gel-like and soft substance was formed around the core; (2) soft blister material was formed on the surface of the cement paste and cement + FA paste specimens; and (3) cement + LP specimens exhibited the best visual appearance.

3.2. XRD analysis

3.2.1. Exposure for 1 and 3 months

The XRD patterns of ettringite and thaumasite show similarities, with just small differences in the d-spacings of the two major peaks, at around 9.1 and 16.0° 2θ. It can be difficult to distinguish

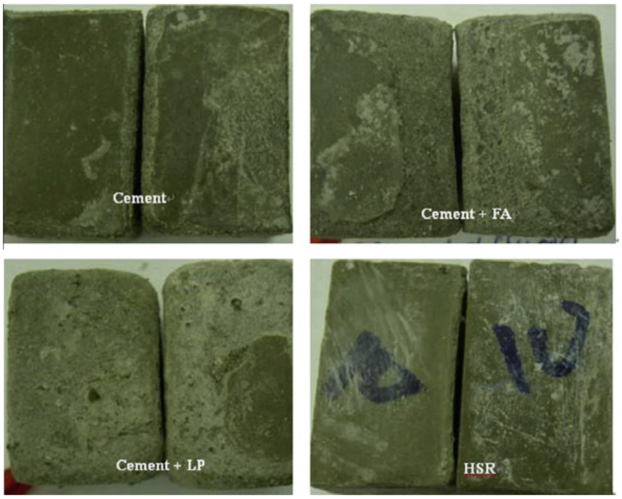

Fig. 1. Visual appearance of pastes exposed to Na_2SO_4:$MgSO_4$ = 3:5 solution for 6 months.

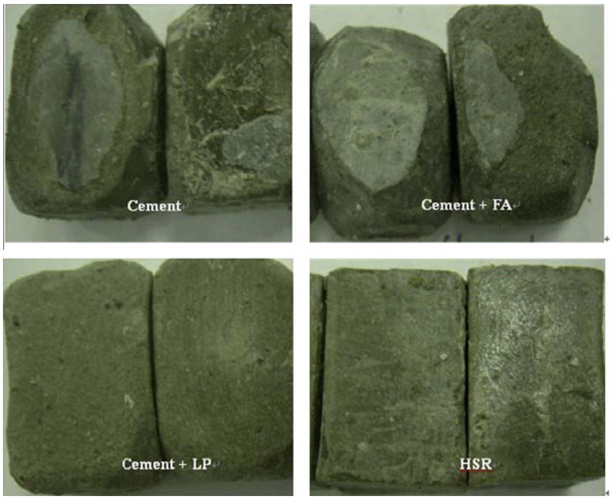

Fig. 2. Visual appearance of pastes exposed to 5% $MgSO_4$ solution for 6 months.

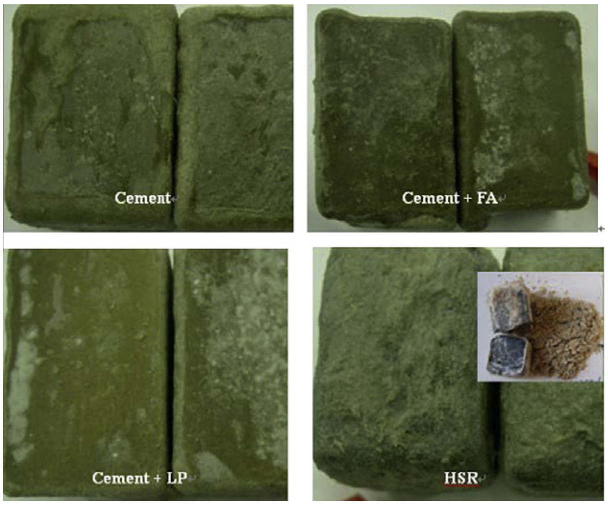

Fig. 3. Visual appearance of pastes exposed to Na_2SO_4:$MgSO_4$ = 10:30 mixed solution for 6 months.

them when only small amounts are present. A relatively clear difference is that another major peak of thaumasite at around 23.4° 2θ is absent from the ettringite [22]. Therefore, this peak is a main sign to identify TF.

In Fig. 4, TF corresponding to a clear peak at 23.4° 2θ in XRD patterns was observed in the cement + LP paste after 1 and 3 months exposure to 5% $MgSO_4$ solution, and the peak was sharper after 3 months exposure. The pH of 5% $MgSO_4$ solution after 1 month exposure was 11.6, while after 3 months the pH increased to 12.7. As to cement paste, after 1 month exposure to 5% $MgSO_4$ solution there was no a peak at 23.4° 2θ in XRD pattern, this means that the main corrosion product in paste was ettringite.

In Fig. 5, after 1 month exposure, the pH of 5% $MgSO_4$ simple solution was 11.6, and the pH of Na_2SO_4:$MgSO_4$ = 1:5 mixed solution was 13.0. However, the peak at 23.4° 2θ of paste exposed to mixed solution was sharper than paste in simple solution. It seems that TF is susceptive to pH of solution.

In Fig. 6, the main ettringite was ettringite. Because the ettringite is stable when the pH is higher than 11.5 [23,24], the XRD patterns of cement paste exposed to 5% $MgSO_4$ solution (pH = 11.6) and Na_2SO_4:$MgSO_4$ = 1:5 mixed solution (pH = 13.0) did not show significant difference.

3.2.2. Exposure for 6 months

After 6 months exposure, the products of different pastes immersed in Na_2SO_4:$MgSO_4$ = 3:5 mixed solution and 5% $MgSO_4$ solution are shown in Figs. 7–9.

As to cement + LP pastes immersed 5% $MgSO_4$ and Na:Mg = 3:5 mixed solution, the visual appearance did not significantly change with the increase of $MgSO_4$ content (shown in Figs. 1 and 2). The XRD patterns in Fig. 7 also present similar results.

In Figs. 8 and 9, with respect to cement paste and cement + FA paste: (1) compared with XRD patterns of pastes exposed to Na_2SO_4:$MgSO_4$ = 3:5 mixed solution and 5% $MgSO_4$ solution, the two major peaks at around 9.1 and 16.0° 2θ had significantly increased; (2) the peaks at around 23.4° 2θ were apparently checked in the XRD patterns of two pastes exposed to 5% $MgSO_4$ solution. The peak of cement + FA paste looked sharper than that of cement paste; and (3) the peaks of $Ca(OH)_2$ in the pastes exposed to 5% $MgSO_4$ solution almost disappeared compared to those of pastes in Na_2SO_4:$MgSO_4$ = 3:5 mixed solution. According to Sharp et al. [12], $Ca(OH)_2$ is a reactant rather than a reaction product for TF. Therefore, the decrease of $Ca(OH)_2$ indicated that the increase of $MgSO_4$ promoted TF in the cement paste and cement + FA paste.

In Fig. 2, HSR paste showed the best visul appearance. In Fig. 10, it can be found that the peaks at 9.1 and 16.0° 2θ were much lower than in cement paste, and there was no apparent thaumasite peak at around 23.4° 2θ. This likely means that the ettringite formation had pushing influence on TF.

In Fig. 11, when different pastes were immersed in the saturated sulfate solution with low pH(<10.0), gypsum became the dominant corrosion product. In HSR paste, almost pure gypsum was generated. In cement, cement + FA and cement + LP pastes there were still some traces of thaumasite or ettringite.

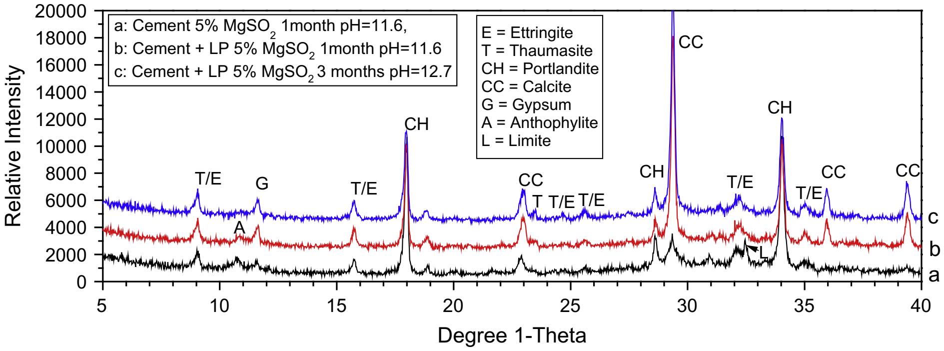

Fig. 4. XRD patterns of cement and cement + LP pastes exposed to 5% $MgSO_4$ solution for 1 and 3 months.

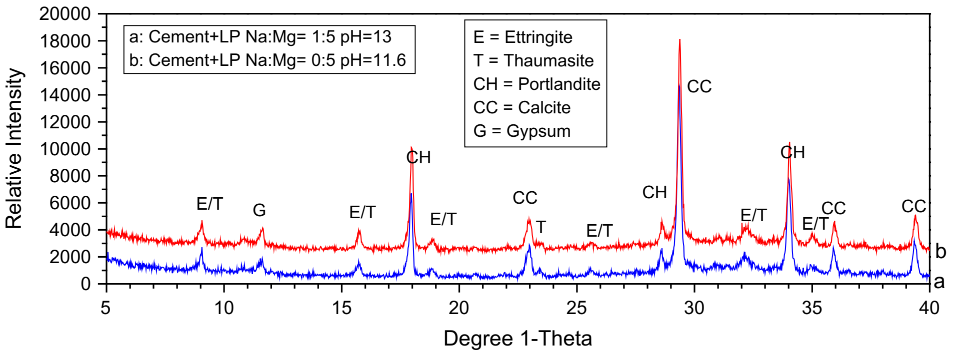

Fig. 5. XRD patterns of cement + LP paste exposed to Na_2SO_4:$MgSO_4$ = 1:5 mixed and 5% $MgSO_4$ solution for 1 month.

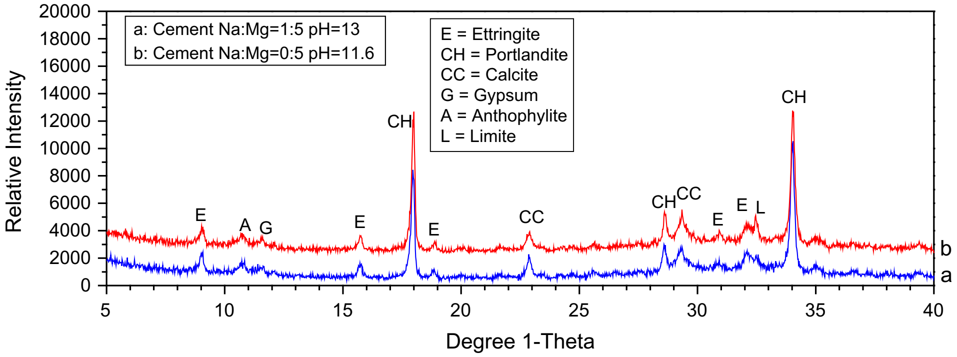

Fig. 6. XRD patterns of cement paste exposed to Na_2SO_4:$MgSO_4$ = 1:5 mixed and 5% $MgSO_4$ solution for 1 month.

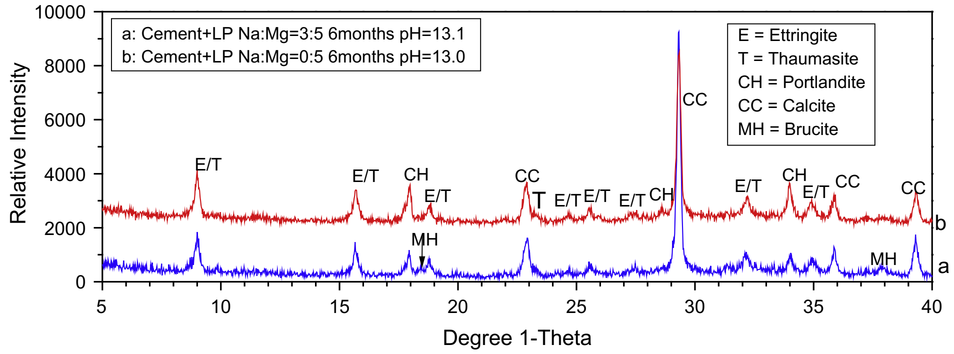

Fig. 7. XRD patterns of cement + LP paste exposed to 5% $MgSO_4$ and Na:Mg = 3:5 mixed solution for 6 months.

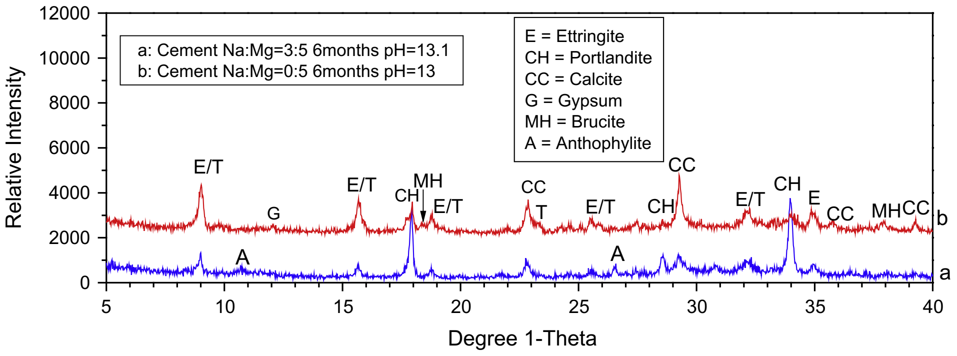

Fig. 8. XRD patterns of cement paste exposed to 5% $MgSO_4$ and Na_2SO_4:$MgSO_4$ = 3:5 mixed solution for 6 months.

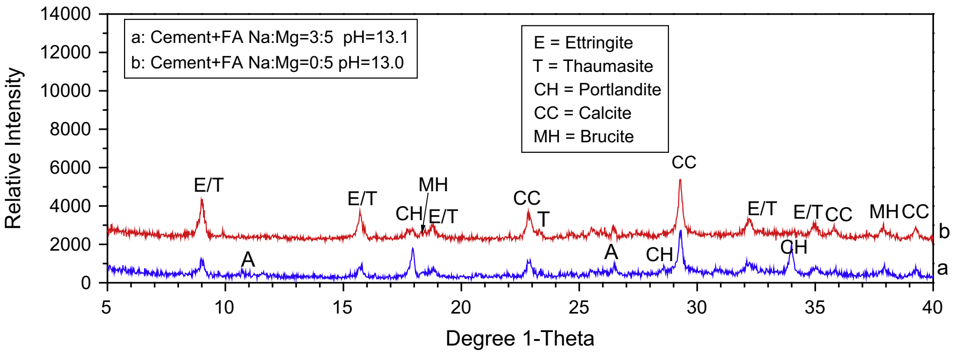

Fig. 9. XRD patterns of cement + FA paste exposed to 5% $MgSO_4$ and Na_2SO_4:$MgSO_4$ = 3:5 mixed solution for 6 months.

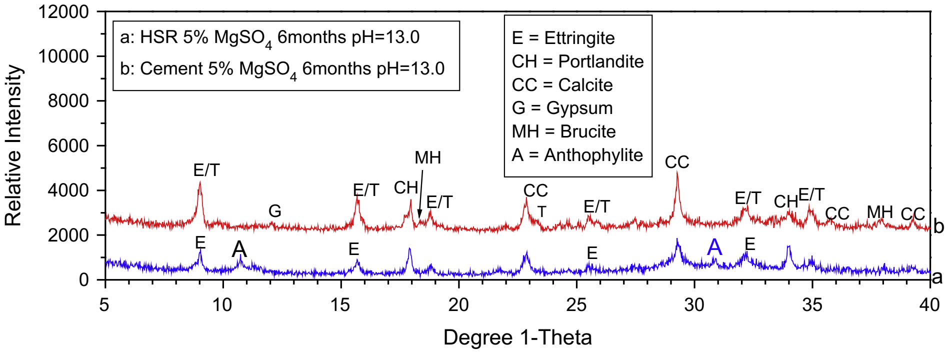

Fig. 10. XRD patterns of cement paste and HSR paste exposed to 5% $MgSO_4$ solution for 6 months.

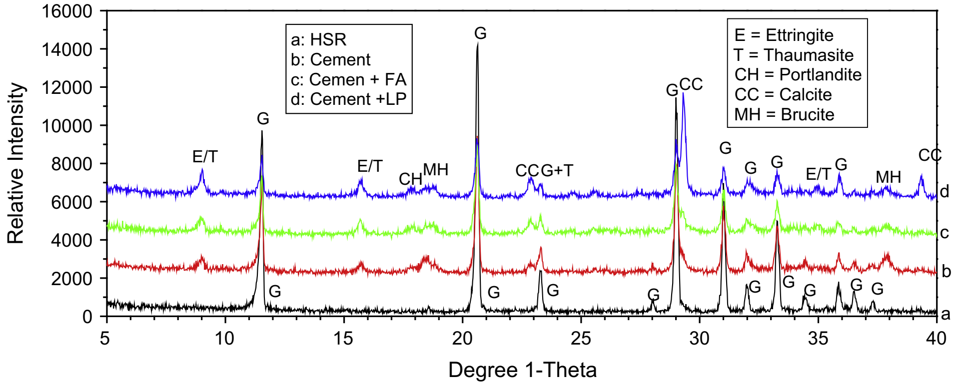

Fig. 11. XRD patterns of different pastes immersed in Na_2SO_4:$MgSO_4$ = 10:30 solution for 6 months.

3.3. pH of exposure solution

Table 3 gives the pHs of solutions with different $MgSO_4$ contents at different exposure ages. As to the Na_2SO_4:$MgSO_4$ = 1:5 and 3:5 mixed solutions, the pH was maintained at the high level of 13.0. Concerning the 5% solution, although the pH was just 11.6 after 1 month exposure, the value was already 12.7 after 3 months exposure, and reached 13.0 after 6 months. Only the pH of Na_2SO_4:$MgSO_4$ = 10:30 solution containing saturated $MgSO_4$ dropped below 10.5, the value of saturated $Mg(OH)_2$ solution.

Table 3
pH of different mixed sulfate solutions at 1, 3 and 6 months.

Solution	1 month		3 months		6 months	
	T (°C)	pH	*T* (°C)	pH	*T* (°C)	pH
Na_2SO_4:$MgSO_4$ = 1:5	10.3	13.0	10.2	13.0	11.1	13.1
Na_2SO_4:$MgSO_4$ = 3:5	9.6	13.0	10.2	13.0	10.3	13.1
Na_2SO_4:$MgSO_4$ = 0:5	11.5	11.6	11.7	12.75	10.2	13.0
Na_2SO_4:$MgSO_4$ = 10:30	9.6	9.43	13.3	9.36	10.1	9.0

4. Discussion

The pH of solution plays an important role in sulfate attack on concrete. The pH influences not only the formation of sulfate attack products, such as ettringite, thaumasite and gypsum but also the stability of C–S–H.

Bellmann et al. [25] indicated that gypsum formation occurs in a relatively low pH environments. The precipitation of gypsum can take place until pH reaches approximately 12.9. Beyond that mark, a further increase of the sulfate concentration is unable to lead to the gypsum formation.

As to ettringite, it is not stable in low-lime environments when the pH falls below 11.5–12.0. At this low pH range, ettringite decomposes and forms gypsum [23,24].

$$3CaO \cdot Al_2O_3 \cdot 3CaSO_4 \cdot 32H_2O + 4SO_4^{2-} + 8H+ \rightarrow 4CaSO_4 \cdot 2H_2O + 2Al(OH)_3 + 12H_2O \quad (1)$$

As mentioned in the introduction, the pH should be higher than at least 12.0 and reach 13.0 for strong formation of thaumasite. In low pH environment, thaumasite decomposes and forms gypsum and calcite [15,16]:

$$\text{Thaumasite} + H_2O \rightarrow CaCO_3 \pm CaSO_4 \cdot 2H_2O(\text{free } SO_4^{2-}) + Ca^{2-} + \text{Si-water complex (or amorphous silica)} \quad (2)$$

On the other hand, if the pH of the pore solution in hydrated cement phase is less than 11.4, the decomposition of C–S–H will occur and leads to the formation of gypsum:

$$C{-}S{-}H + 2H_2O + SO_4^{2-} \rightarrow \text{Ca-depleted } C{-}S{-}H \text{ (or amorphous silica)} + CaSO_4 \cdot 2H_2O \quad (3)$$

The sequential reactions of $MgSO_4$ with the cement hydration products are shown below [2,3]:

$$xMg^{2+} + xSO_4^{2-} + xCaO \cdot SiO_2 \cdot aq + 3xH_2O \rightarrow xCaSO_4 \cdot 2H_2O + xMg(OH)_2 + SiO_2 \cdot aq \quad (4)$$

$$2xMg^{2+} + 2xSO_4^{2-} + 2xCaO \cdot SiO_2 \cdot aq\ SH_n + yH_2O \rightarrow 3MgO \cdot 2SiO_2 \cdot 2H_2O + 2xCaSO_4 \cdot 2H_2O + (2x-3)Mg(OH)_2 \quad (5)$$

In the $MgSO_4$–C–S–H–H_2O system shown in Eqs. (4), (5), the insoluble $Mg(OH)_2$ formation may cause two possibilities: (1) according to chemical kinetics, to foster the chemical reactions of the decomposition of C–S–H; and (2) pH drop of systems.

Thaumasite forms from calcium, sulfate, carbonate and silicon. The latter originates from the decomposition of C–S–H formed as shown in Eq. (3). Consequently, it is commonly agreed upon that the existence of $MgSO_4$ can accelerate TF due to the pH drop.

However, according to the experimental results shown in Table 3, even in the 5% $MgSO_4$ solution by mass, which is much higher than the monitored value of field cases and usually used as accelerated laboratory test [26], the pH of solution under non-exposed condition was still kept at the high level of 13.0. Likely, it can be concluded that the existence of $MgSO_4$ cannot significantly reduce the pH of stagnant solution under non-exposed condition. On the other hand, the survey data of M5 motorway bridges showed that highly soluble $MgSO_4$ decreases with proximity to concrete and with increasing thaumasite attack [7,27–30]. Therefore, in the field experience TSA should also occur in the sulfate environment with high pH. Only in the solution containing saturated $MgSO_4$ it was possible that the pH fell below 10.5 (shown in Table 3), while ettringite, thaumasite and C–S–H would decompose as Eqs. (1)–(3), performing severe gypsum-type attack (shown in Fig. 11), exhibiting loss of cohesion and softening [31] (shown in Fig. 3).

According to XRD patterns shown in Fig. 5, although the $MgSO_4$ content in Na_2SO_4:$MgSO_4$ = 1:5 mixed solution was less than in 5% $MgSO_4$ solution, the peaks at 23.4° 2θ of cement + LP pastes exposed to these two solutions were shifted. it can be found that the pH of Na_2SO_4:$MgSO_4$ = 1:5 mixed solution was 13.0, while the pH of 5% $MgSO_4$ solution was just 11.6. This appearance further supported that the high pH (close to 13.0) favors TF [13–16].

Combining the appearances in Figs. 1, 2, 8 and 9, it can be confirmed that the increase of $MgSO_4$ could accelerate TSA in the cement paste and cement + FA paste while the pH of solution was kept at a level of 13.0. The reason for the acceleration may be the improving effect on the progress of chemical reactions of decomposition of C–S–H shown in Figs. 4 and 5 due to the formation of insoluble $Mg(OH)_2$ based on the chemical kinetics.

Normally ordinary Portland cement paste containing limestone additions is susceptible to TSA [12], and the fly ash addition can improve the resistance against TSA of cementitious materials [32,33]. However, the visual appearances in Figs. 1 and 2 showed that the cement paste and cement + FA paste were susceptive to TSA with the increase of $MgSO_4$ content. The results in Figs. 7–9 further supported the visual observation. This may be due to the higher Al^{3+} contents in the cement paste and cement + FA paste than in cement + LP paste. Research [34] has pointed out that alumina must be present in sufficient quantities within the clinker or gel structure, rather than as an addition, to initiate TF. In Fig. 10, HSR paste did not show apparent trace of TF due to the low content of C_3A, causing relatively weak deterioration shown in Figs. 1 and 2. Due to the higher content of Al_2O_3, more ettringite could be generated in cement + FA paste than cement paste, causing more thaumasite formation (shown in Figs. 8 and 9) and resulting in more severe deterioration (shown in Figs. 1 and 2). In effect, the experimental results [35] also showed that concrete and mortars containing fly ash appeared to be more vulnerable to sulfate attack at low temperatures due to TF. This result may indicate that the woodfordite route of TF by reaction between ettringite, silicate (particularly C–S–H) and carbonate (either CO_3^{2-} ions or atmospheric CO_2) in the existence of excess water would more likely be the major mechanism for TF but not direct route[9,36].

5. Conclusions

The exposure conditions of field cases of TSA on foundations were simulated to study the effect $MgSO_4$ on TF. According to the analysis of the test results, several conclusions can be drawn:

1. Under the non-exposed condition, relatively low content $MgSO_4$ (even 5%) could not cause a significant pH drop of the stagnant solution. The pH was kept at the level of 13.0, while the increase of $MgSO_4$ content could promote TF.
2. Under the non-exposed condition, the pH of the stagnant solution containing saturated MgSO4 at 10 °C decreased to below 10.4. However, Gypsum attack, forming soft and gel-like products, dominated the mechanisms of paste deterioration.
3. In the sulfate solution containing relatively low content $MgSO_4$ (even 5%), HRS paste showed the best resistance against TSA. With the increase of $MgSO_4$, cement paste and cement + FA pastes were more susceptible to TSA than cement + LP paste.
4. Cementitious paste containing higher Al element showed more severe damage by TSA. The woodfordite route of TF would likely be the major mechanism for TF.

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