



EVALUATION OF ATYPICAL SULFATE ATTACK OR A NEW FORM OF MORTAR DETERIORATION

Smith, Andrew¹

ABSTRACT

Sulfate attack of mortars due to the chemical reaction between a source of water-soluble sulfate from an external source, such as clay bricks, groundwater or contamination, and the tri-calcium aluminate (C3A) component of the cement clinker phases, in the presence of water, is known and widely reported in the scientific literature. In the UK, over the past 5 years or so, a number of cases have been reported and investigated that have been attributed to sulfate attack, normally with the primary source of the sulfate being the clay bricks. Closer inspection of the evidence suggests that this is not a form of "classic" sulfate attack, but may result from different chemical or physical processes. This paper presents a number of examples investigated by the author that relate to situations where damage, in the form of surface erosion of the mortar joint, predominantly in vertical, cavity wall construction of domestic dwellings within 5 years of construction, has been claimed to be attributed to sulfate attack. However, upon further investigation the damage observed and the evaluation of the mortar itself, chemically and mineralogically, suggest that the damage results from a different form of deleterious process. Whilst water-soluble sulfates and Ettringite have been detected throughout the mortar joint, only the surface 2-10mm of the mortar joint are affected. In addition, bricks which are known to have low to very low water-soluble sulfate content exhibit the same type and appearance of mortar damage between them, as those with higher sulfate content. The paper presents the analytical data, and proposes that the damage results from the deposition of crystalline salts within the pore structure, some derived from the mortar itself, and potentially the damage is enhanced by the aerated pore structure of the mortar.

KEYWORDS: mortar, sulfate attack, salt crystallisation

BACKGROUND

'Sulfate Attack' in Portland Cement based mortars has been known about and studied for many years, and in many parts of the world. Cement mortars can be attacked by solutions containing sulfate, such as natural or polluted ground waters. Attack leads to strength loss, expansion,

¹ Programme Leader, Clay, Cement & Concrete Technologies, Mineral Products Centre, University of Derby, Enterprise Centre, Bridge Street, Derby, DE1 3LD, UK, a.smith5@derby.ac.uk

cracking, and ultimately, disintegration [1]. These impacts affect the integrity of the mortar joints, and thus often result in disruption to the structure of the masonry. The expansion of mortar attacked by sulfate is generally attributed to the formation of ettringite and gypsum [2], resulting in an expansion and breakdown of the mortar.

Up until around 5 years ago, in the UK, sulfate attack of mortar was most commonly observed in clay brick based masonry associated with the following contributory factors:

- A source of 'external' sulfate (clay bricks, ground/soil water, contaminated land)
- Portland Cement based binder containing tri-calcium aluminate C3A (hydrated C3A in cement paste)
- Masonry exposed to high levels of saturation or repeated periods of cyclic saturation from wind driven rain; chimneys, brick masonry gate posts/pillars, free standing boundary walls, and parapet walls
- Masonry in contact with ground/soil water; any brick/block/stone masonry earth retaining walls

The 'classic' chemical and physical evidence of sulfate attack having taken place include the following characteristics:

- Elevated sulfate levels (>4% based on cement content within the mortar), typically in excess of 8% based on the cement content of the mortar.
- Deposition of ettringite and or gypsum within the mortar pore structure
- Cracking of the mortar joint, typically 'mid line' of the mortar bed joint.
- Occasionally the mortar appears as a white waxy/gelatinous mass in the centre of the joint.
- Expansion of the mortar joint resulting in either;
 - vertical displacement of brickwork (as seen in vertical cavity wall construction), or
 - asymmetric expansion of the brickwork (chimneys, brickwork pillars, free standing/parapet walls and earth retaining walls) resulting in the 'bending/leaning' of the brickwork.

Historically in the UK, a number of cases of sulfate attack in vertical masonry have been reporteded and investigated, typically cavity wall construction in domestic housing. In these the underlying cause was attributed to a combination of very high sulfate salt levels in the bricks and masonry that was saturated or prone to saturation. Very occasionally, saturated insulation in the cavity, has been identified as a likely source of the moisture in the brickwork masonry. With these 'vertical' brickwork masonry incidences, 'blame' has often been attached to the brick manufacturer, as the source of the sulfate has been the brick.

More recently in the UK, a significant number of investigations have been undertaken into a phenomena where the surface of the mortar joints are eroding after only a short period of time, typically between 6 and 24 months, post build. When chemical analysis of the mortar is undertaken, to the British Standard BS 4551 [3], the results often show only slightly elevated levels of sulfate, above the cement based sulfate level of a maximum of 4%, and rarely are significant

amounts of ettringite or gypsum found. More importantly, that damage only appears to be limited to the exposed outer surface of the mortar joint, and not the centre or rear of the joint.

In these cases, many of the 'classic' observable characteristic of sulfate attack are missing, and despite sulfate levels being elevated in the mortar samples tested, the adjacent bricks show levels of water-soluble salts, including sulfates, that are within the manufacturers declared values and thus compliant with the European Standard for clay masonry units, EN 771-1 [4].

SULFATE ATTACK OF MORTARS

Sulfates attack not only the surface layer of the cement mortar but also the inner part of the mortar joint. The sulfate media penetrate into the cement mortar though pores and react with hydrated crystals such as calcium hydroxide and calcium sulphoaluminate.[1]

Basista & Weglewski [5] have reviewed many of the key papers investigating the nature of sulfate attack in concretes, as opposed to mortars, however, they conclude that the most likely mechanism for the formation of expansive internal pressure is via the 'topochemical' formation of ettringite and gypsum, which is also valid, if not more so, for mortars than for concrete as a result of the greater permeability/porosity of the mortar.

They describe the following reactions that take place within the cement paste pore structure;

This process is initiated by the reaction between the sodium sulphate and calcium hydroxide (CH):

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

This is a through-solution reaction proceeding in the aqueous solution filling the pores of concrete. Calcium hydroxide in the pores reacts with the sodium sulphate from the ambient solution to form gypsum (CaSO₄ \cdot 2H₂O) and sodium hydroxide. However there is no associated volumetric change.

However, the chemical reaction proceeds further. The newly produced gypsum can react with some alumina-bearing phases like unhydrated tricalcium aluminate $3CaO \cdot Al_2O_3$ (C3A) or hydrated calcium sulfoaluminate (monosulphate) to form ettringite, e.g.:

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

The formation of the ettringite within the voids/pore structure of the mortar, results in a volumetric expansion and tensile (expansive) forces within the mortar cement paste pores. Ultimately if this is greater than the tensile strength of the mortar paste, cracks are induced, resulting in greater ingress and exposure of the hydrated cement paste to the sulfate rich solutions.

Santhanama et al [6][7] have proposed a progressive mechanism for the formation of both ettringite and gypsum within mortar, based on 3 identifiable zones, moving into the mortar joint:

- i. A cracked and highly 'deteriorated surface zone' (Zone 1)
- ii. A zone of 'deposition of attack products' (Zone 2), primarily gypsum around aggregates and in pores, and ettringite within the paste.
- iii. An 'interior chemically unaltered zone' (Zone 3), which is cracked.

The inference here being that there should be an identifiable 'zone' containing the 'attack products', namely gypsum and ettringite. This mechanism also assumes that the ingress of sulfate rich fluids into the mortar is from the outer surface of the mortar.

The deposition of the attack products, gypsum and ettringite, being volumetrically greater than the voids or materials they are formed from, results in expansion and the propagation of cracks and microcracks in the mortar. These cracks initially weaken the mortar and provide additional pathways for moisture ingress and further reaction and subsequent deposition of 'attack products' within these new cracks. Eventually the mortar joint integrity is lost, and vertical expansion of the bed joints, and horizontal expansion of the vertical 'perpendicular' joints leads to structural damage of the brickwork.

MORTAR EROSION – SITE INVESTIGATIONS

Due to commercial confidentiality, no site or manufacturer/supplier details are presented here, however the compositional characteristics of both the clay bricks and mortar are presented in such a way as not to be able to identify manufacturer/supplier.

Site A

- Brick: Red based multi coloured soft mud (moulded) brick (215 x 102 x 65mm), S2 (low soluble salts), F2 (fully frost resistant)
- Mortar: Lime:Sand (1:6) pre mixed and pigmented based mortar (delivered to site) to which 1 part cement (CEM I) was added to achieve a 1:1:6 mortar mix, a designation (iii) mortar suitable for general purpose building in accordance with BS 5628-3.[8] this would equate to a M4 class mortar, EN 998-2 [9]. Within the sand fraction it was reported that 25% of the sand was a sharp sand containing limestone or chalk fragments (calcium carbonate). 10mm nominal joint thickness. Joints are rubbed/brushed finish leaving a porous joint surface.
- Site: Eastern England, sheltered E, W and S elevations. Exposed (open fields to the North)
- Property: Single, large 4+ double bedrooms, domestic, 2 storey detached property, on the edge of a housing development (20+ properties). Built 2006. Inspection: 2013

Although the inspection was undertaken 7 years post build, the degree of damage to the mortar was limited to only a small number of localised areas of brickwork, in areas of the property that showed surface white efflorescence on the brickwork. The mortar joints were 'rubbed or brushed' rather than 'tooled', so the surface of the mortar joint remains porous.



Figure 1: One of the Localised Areas of Brickwork where the Mortar Joint has Eroded Back Approximately 10mm from the Brick Face (A). Blistered and Friable Mortar can be seen in Association with Mortar Joint Surface Efflorescence (B).

The primary form of damage to the mortar joints on this property were identified as being: 'localised erosion to a maximum depth of 10mm, associated with areas showing significant evidence of efflorescence on both bricks and mortar joints'. A typical such localised section of brickwork is shown in Figure 1. In Figure 1, Arrow B shows an area of mortar joint that is both eroded, but shows significant surface blistering and efflorescence of the mortar joint itself.

Table 1: Summary of the Sulfate Content (SO₃%), Expressed as a % of the Mortar Cement Content, for the 9 Mortar Samples Taken From the Property From Localised Areas of Mortar Joint Surface Erosion.

SO3 as a % of cement content	Front (0-35mm)	Middle (35-70mm)	Back (70-102mm)	
Max	24.3	18.5	16.8	
Min	13.8	13.0	11.3	
Average	19.4	15.1	13.9	

Analysis of the mortar from 9 locations where the mortar joints showed localised surface erosion, Table 1, shows that the mortar joints on the whole are enriched with sulfate. A 'normal' maximum sulfate content, based on the cement content of the mortar, would be in the order of 4%. It is therefore clear that sulfate from an external source has migrated into the mortar joints, most likely from the bricks. When the surface efflorescence of both the mortar joints and the associated bricks were analysed, it shows that the dominant sulfate in the bricks is sodium sulfate, with varying amounts of potassium and calcium sulfates. Of these sulfate species, sodium and potassium are known to have the greatest solubility, with calcium sulfate having a relatively low comparable solubility.

Efflorescence from the adjacent mortar surfaces again show relatively high sodium cation levels, but the combined sodium and potassium cation levels are well in excess of the calcium cation

content. This is different to what would normally be expected if sulfate attack was responsible for the formation of gypsum (calcium) based sulfates and ettringite, from the sodium and potassium based sulfates derived in the bricks, as per the sulfate attack process described by Basista & Weglewski [5] and Santhanama et al [6] [7]. It would therefore suggest that the root cause of the erosion of the mortar is 'crypto-florescence', the formation of efflorescence salts within the pore structure of the mortar rather than *sensu stricto* classic sulfate attack.

Site B

Brick:	Brick 1: Red based multi coloured 'Stock' soft mud (moulded) brick (215 x 102 x $\frac{1}{2}$
	65mm), S2 (low active soluble salts) occasionally transgressed to S1, F2 (fully frost
	resistant)
	Brick 2: Red extruded/wire cut brick, S2 (low active soluble salts), F2 (fully frost
	resistant)
Mortar:	Cement:Sand (1:6) Class M4 dry silo mix mortar with air-entrainer. Nominal 28d
	compressive strength of 4N/mm ² in accordance with EN 998-2 [9]
	The sand was a 0/2 grade (0-2mm) in accordance with EN 13139 [10]
	Nominal 10mm mortar joint thickness. Joints are tooled giving a bucket handle joint.
Site:	Southern England, sheltered in residential suburban area.
Properties:	Predominantly, large 3 and 4 double bedrooms, domestic, 2 storey detached properties,

on a housing development (40+ properties). Built 2010-12. Inspection: 2013

The site in the south of England is a major development of 40+ properties including detached 2 storey domestic houses, along with a number of multi occupancy 4 storey apartment units. The properties were constructed using cavity wall construction with concrete block inner leaf and single skin brickwork masonry external leaf. Two brick types were used, a red multi 'Stock' brick and a red textured and sanded 'Extruded' (perforated) brick from different brickworks. The primary investigation was aimed at the mortars associated with a Red Multi Stock brickwork that did show evidence of surface erosion of the joints, on some elevations of some properties. However, so did mortar joints associated with the red 'Extruded' brick as well.

Analysis of the red multi 'Stock' bricks, despite showing the residual sulfate levels to be S2 and thus the lowest of the active soluble salts classifications for clay masonry units in accordance with EN 771-1. However the red 'Extruded' bricks consistently show very low levels of water-soluble salts, well within the S2 class, both as tested from the manufacturer's historic quality control and declaration of performance data. As the majority of the investigation was conducted on brickwork above dpc and in cavity wall construction, it is therefore more than likely that the bricks are the source of the water-soluble sulfate levels found in the mortar.



Figure 2: Mortar erosion and mortar joint efflorescence as seen during the inspection of the brickwork at Site B. (a)-(b) 'Red Multi Stock' Brick Type 1, (c)-(d) 'Red Extruded' Brick Type 2. (a) typical erosion of the mortar joint surface, (b) (c) typical mortar joint efflorescence, (d) mortar joint erosion associated with the Red Extruded, Brick Type 2.

Whilst the investigation could not rule out that a form of sulfate attack may be the root cause of the erosion of the surface of the mortar joints, this is by no means a 'classic' case of sulfate attack. There were some aspects of the mortar and type of damage that do not fit with this diagnosis:

- No mid line mortar bed cracking.
- No deterioration of the middle part of the mortar joint.
- No expansion or disruption to the brickwork, only the mortars outer surface.
- No substantial deposits of the by-products of the sulfate attack reaction, ettringite and gypsum in the affected areas of mortar.

Figure 2 shows a selection of photographs taken at the time of inspection and sampling of the mortar at Site B. Of note was the fact that both the brick types, despite having been manufactured at different brickworks, from different clay types, and by different manufacturing processes, brickwork built from both exhibit mortar joint surface erosion (Figure 2a and 2d) and surface efflorescence (Figure 2b-2c) of a very similar nature.

Six mortar sampling locations were identified to cover all the primary levels of exposure, position of the brickwork to the prevailing weather directions, being either exposed or protected, and associated levels of mortar erosion. The results of the mortar testing are shown in Table 2.

Table 2: Sulfate: Cement ratio values along with residual water-soluble salts for mortars from 6 locations at Site B. The mortars for each location were sampled from 3 sections of the full bed joint width, namely the 'Outer' 35mm, 'Middle' 35mm and the 'Back' 33mm, of the nominal 102mm brick width. (Exp – Exposed, Pro – Protected)

Location	Position	Sulfate : Cement	Water-soluble Salt				
		Ratio	Ca	Mg ²⁺	Na ⁺	K ⁺	SO4 ²⁻
1 Exp	Outer	8.0	0.11	bdl	0.03	0.07	0.39
	Middle	10.9	0.22	bdl	0.01	0.04	0.24
	Back	10.2	0.21	bdl	0.02	0.07	0.35
2 Exp	Outer	8.5	0.13	bdl	0.03	0.07	0.45
	Middle	7.3	0.20	bdl	bdl	0.02	0.05
	Back	6.1	0.15	bdl	0.01	0.04	0.14
3 Pro	Outer	7.4	0.12	bdl	0.04	0.03	0.36
	Middle	4.9	0.14	bdl	bdl	0.02	0.16
	Back	4.1	0.26	bdl	bdl	0.02	0.02
4 Exp	Outer	8.6	0.20	bdl	0.06	0.06	0.54
	Middle	6.7	0.18	bdl	0.02	0.05	0.14
	Back	4.7	0.39	bdl	0.01	0.04	0.02
5 Exp	Outer	12.3	0.18	bdl	0.09	0.12	0.77
	Middle	9.0	0.21	bdl	0.03	0.06	0.41
	Back	9.1	0.17	bdl	0.04	0.07	0.20
6 Pro	Outer	5.2	0.10	bdl	0.02	0.02	0.28
	Middle	4.2	0.16	bdl	0.01	0.03	0.08
	Back	4.3	0.21	bdl	0.01	0.02	0.03

In nearly all cases, the surface 'Outer' 35mm of the mortar joints are enriched with sulfates, with significantly lower levels in the 'Middle' and 'Back'. If this is then viewed along with the watersoluble salt content of the same portions of the mortar joints, it can be seen that calcium is by far the most dominant soluble cation. This is to be expected as the soluble salt content will also contain water-soluble portlandite (Ca(OH)₂). Both are indicative of the movement of water-soluble salts from the main body of the mortar joint to the drying surface of the joint. What is also evident from the X-ray Diffraction (XRD) analysis of these mortars, is that there is little or no evidence of 'reaction products' in the mortar. With the exception of Sample 5, Outer and Back, where there approximately 1% gypsum was detected, none of the other mortar samples show any significant ettringite or gypsum deposits in the mortar. Location 6 in Table 2 represents brickwork associated with Brick Type 2, the red 'Extruded', a brick with a very low water-soluble salt content, and thus not expected to be a source for elevated levels of sulfate in the associated adjacent mortar. Whilst the sulfate:cement ratio is at or about the 4% level, there is a slight increase, again in the 'Outer' portion of the mortar joint. This brickwork is in a protected part of the building so not subjected to wind driven rain. It is therefore unlikely that the environmental requirements for chemical sulfate attack are present, and thus unlikely that sulfate attack could be the root cause of the surface erosion of this mortar joint.

DISCUSSION

Much of the evidence form these 2 site investigations show a situation where there are elevated levels of sulfate in the outer surface layer of the mortar joint, but little or no evidence of chemical sulfate attack of the mortar, only surface erosion through 'sanding'. In both cases, once the surface friable mortar was removed, the remaining mortar joint was found to be intact and relatively resistant to the drilling process to obtain samples. In both cases, surface efflorescence of the mortar joints were observed, indicative of the movement of salts through the mortar joint to the drying face of the mortar, where they precipitate.

Analysis of the mortars failed to identify significant sulfate attack reaction products, gypsum and ettringite, in the affected mortars, and on neither site was there any evidence of expansion vertically of the brickwork. Other investigators, identified the root cause to be sulfate attack, purely on the basis of the presence of elevated sulfate levels in the mortar.

Ettringite forms within hardened concrete or mortars, often referred to as 'Delayed Ettringite Formation' (DEF), although some authors [11] prefer the term 'external ettringite' to indicate a reaction involving an external source of sulfate ions and monosulfate, and subsequent chemical 'sulfate attack' of the concrete or mortar. Ettringite is generally stable in the pH range 10.5 to 13.0 [12]. With gradual decrease of pH, ettringite is not stable and decomposes to form gypsum.[13]. The chemical reaction and subsequent formation and deposition/crystallization of ettringite and gypsum within the pore structure and cracks/microcracks in the mortar results in expansive forces, resulting in further cracking and loss of competency of the mortar paste – aggregate bonding.

In the investigations presented here, these features were not observed, therefore the author concluded that it was unlikely that chemical sulfate attack was the underlying cause of the problem.

Examples of sulfate-induced degradation of concrete has also been reported in cases where ettringite and gypsum that are characteristic of chemical sulfate attack were <u>not</u> found [14]. In such cases a physical form of attack, whereby salt-bearing solutions rise in concrete by capillarity and after surface evaporation, the solution becomes supersaturated, leading to salt crystallization in the pores of concrete, therefore generating pressures that can cause cracking. Rodriguez-Navarro and Doehne [16], suggest that crystallization of thenardite in pores may be the source of most of the physical damage.

In addition to this physical form of deterioration resulting from the crystallization of salt crystals in the pores, other researchers in the literature report cyclical salt crystallization, resulting in salt hydration pressure in a porous material, as the source of physical damage that cause of the deterioration of building stone and brick [15–19]. They report that certain salts, such as Na₂SO₄ and sodium carbonate, can cycle between their hydrated and anhydrous forms. Such a conversion

of anhydrous Na₂SO₄ (thenardite) to decahydrate (mirabilite) involves an expansion of around 315%, known as 'salt hydration distress' [17] or 'salt crystallization'.

The presence of mortar joint efflorescence is indicative of the movement of water-soluble salts through the mortar. In both investigations, the mode of damage to the mortar is more akin to that described by Novak & Colville [14], resulting from the crystallisation and or cyclical hydration of the salts. Abu Bakar *et al* [19] provide a comprehensive review on the mechanism of 'salt attack' both chemical and physical, on masonry, including mortars. They state that salt crystallization in the pore structure, requires the pore fluid to be supersaturated. In general terms, the greater the degree of supersaturation, the greater the resultant crystallization pressure. For the crystallization of gypsum, values of c.28MPa and c.94MPa are quoted for supersaturation crystallization factors of 2 and 10 respectively, at zero degrees centigrade.

It is however also worth considering other processes that can impact on the outermost regions of the mortar joints. Repeated cyclical freeze-thaw damage, resulting from the crystallization of ice within the saturated pores, is also known to result in a general progressive erosion of the surface of the mortar joints.

Finally, washing down brickwork with 'brick acid' could contribute to the overall weakening of the mortar. 'Brick acid' cleaning agent, is widely used to remove mortar splashes from the surface of brickwork before the scaffolding is removed. If undertaken incorrectly, acid that is too strong, or a build-up of acid, can result in acid attack of the mortar, breaking down the bonding properties, thus weakening the surface of the mortar, making it more susceptible to other erosion processes.

In summary, it appears failure to investigate other potential causes or contributory causes, can result in not only a poor interpretation of the evidence, leading to the wrong conclusion, which may result in the wrong party being financially penalised. There are therefore a number of potentially co-existing processes that all individually, or in combination, can result in the surface erosion of the mortar joints:

- Chemical sulfate attack
- Physical sulfate salt crystallization
- Physical cyclical salt hydration distress
- Freeze-thaw cycling
- Hydrochloric acid (brick acid) attack of the mortar surface

The author recommends therefore that each of these be evaluated accordingly in an appropriate manner, and evidence and careful interpretation of the data, be gathered and used to rule in or rule out each as a cause or contributory factor.

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