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Thaumasite — background and nature in deterioration of cements, mortars and concretes

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Abstract

Thaumasite has been shown to form at low temperatures, particularly 0–5°C, as a non-binding calcium carbonate silicate sulphate hydrate under conditions of destructive sulphate attack. Formation of thaumasite arises generally from calcium silicate hydrate C–S–H and Ca^{2+}, CO_3^{2-}, SO_4^{2-}, CO_2 and water, or from ettringite in the presence of C–S–H, carbonate and/or carbon dioxide and water. It basically resembles a carbonated ettringite, with which it has often been confused in the past. Conversion of the main cementitious binder C–S–H into the non-binder thaumasite is a destructive form of sulphate attack. Greater awareness of the potential problems that thaumasite can cause has arisen with the increased use of limestone fillers in cements, the common employment of limestone aggregates in concrete and the introduction of Portland limestone cements, together with the realisation that structural foundations of buildings are, on average, below ambient temperature and (more often than not with on- and above-ground construction) are within the optimum temperature range for thaumasite to be formed. Instances have been found in specific studies of large quantities of thaumasite being formed in foundation concretes with no evidence for any structural damage above ground level. It is important to be aware of the propensity of thaumasite to form at low temperatures for mix designs, so as not to encourage any destructive sulphate attack by thaumasite to arise. This means utilising low water/cement ratios for workable mortars and concretes, so as to give reduced permeability. This will prevent, or at least suitably hinder, ingress of destructive ions and water, so that the potential for destructive sulphate attack by formation of thaumasite is not actually realised in practice. © 1999 Elsevier Science Ltd. All rights reserved.

Keywords: Thaumasite; Cement; Mortar; Concrete

1. Introduction

Thaumasite has long been known as a naturally occurring mineral of the type $CaSiO_3{\cdot}CaSO_4{\cdot}CaCO_3{\cdot}15H_2O$, which has been found in metamorphosed rocks that have undergone hydrothermal changes with the passage of time [1–3]. It often arises in thin veinlets in association with both ettringite $3CaO{\cdot}Al_2O_3{\cdot}3CaSO_4{\cdot}31\text{–}32H_2O$ and calcite $CaCO_3$ [2,3]. In construction, thaumasite has been found as a product of deterioration of cements, mortars and concretes in a wide number of instances. Occurrences of thaumasite in deteriorated concrete were discussed at length in a detailed report in the 1960s [4]. At that time, although the mineral thaumasite was thought to have formed at a very low temperature and also at a very late stage of mineralisation [5], neither its method of synthesis nor its precise structure were known. Controversies over whether the silicon in thaumasite was 4- or 6-co-ordinated by oxygen (hydroxyl) needed to be resolved in order to understand the nature of thaumasite, its formation and the implications for structures in which its appearance as a deterioration product had already been identified.

2. Structural considerations

Controversy reigned over the actual structure of thaumasite, in particular, the precise co-ordination of silicon by oxygen (hydroxyl), for a number of years.

The mineral thaumasite was originally classified as having a hexagonal pyramidal type of structure containing SiO_4 tetrahedra and CO_3 triangular planes. The formulation $Ca_6(CO_3)_2(SO_4)_2(SiO_3)_2{\cdot}30H_2O$ was used as a basis by Brú et al. for postulating a plausible structural layout [6,7]. An X-ray crystallographic investigation by Welin [8] led to the structure $2\{Ca_3H_2(CO_3/SO_4/SiO_3){\cdot}13H_2O\}$ being proposed. This was considered to be of the type $Ca_6H_4(SiO_4)_2(CO_3)_2(SO_4)_2{\cdot}26H_2O$ with a space group C_{3v}, based upon columns made from Ca^{2+} and SiO_4^{4-} ions and H_2O molecules, between which occurred CO_3^{2-} and SO_4^{2-}

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ions as well as additional H_2O molecules; the four H^+ ions could not be suitably placed and were thought to be attached to the SO_4^{2-} groups [8]. However, the orientation of the SO_4^{2-} ions in this proposed structure was questioned by Font-Altaba [9]. These earlier structural postulations all envisaged Si as 4-co-ordinated with oxygen in the thaumasite structure.

Such a structure containing Si in 4-co-ordination with oxygen was challenged by Moenke on the basis of an infrared spectroscopic examination [10,11]. He suggested that thaumasite actually contained silicon in 6-co-ordination with oxygen, because of the similarity of the infra-red wavebands in the 765–638 cm^{-1} and 935–887 cm^{-1} regions with the corresponding wavebands of stishovite, a high temperature–high pressure polymorph of silica that was known to contain silicon in 6-co-ordination with oxygen [12]. Kirov and Poulieff [13] strongly disputed Moenke's interpretation on the grounds that the IR waveband of thaumasite at 1100 cm^{-1} was a composite due to both SO_4^{2-} and SiO_4^{4-} groups. They had examined IR and X-ray diffraction data of thaumasite both at ambient temperature and upon heating at various temperatures between 200 and 1150°C. In their view [13], because the vibrational frequencies of SO_4^{2-} and SiO_4^{4-} coincided, causing only one absorption maximum at 1100 cm^{-1}, these vibrational frequencies would resolve upon heating and hence become independently identifiable. The flaw in this argument is that thaumasite actually breaks down at 110°C and transforms into a glassy phase known as thaumasite glass, in which the randomly distributed silicate groups contain Si 4-co-ordinated by oxygen [14].

Later X-ray crystallographic investigations of thaumasite suggested that its structure was of the type $Ca_6[Si(OH)_6]_2(CO_3)_2(SO_4)_2{\cdot}24H_2O$ [15–17], similar to that of ettringite $Ca_6[Al(OH)_6]_2(SO_4)_3{\cdot}26H_2O$. The stability of thaumasite and its $Si(OH)_6$ octahedra was considered to be due to the other electronegative atoms attached to oxygen, presumably drawing electrons away from the Si–O bonds to allow an increase in the Si co-ordination number from 4 to 6, and thus allowing the distorted $[Si(OH)_6]^{2-}$ octahedra to become stabilised.

Further confusion arose from IR investigations by Portnov and Solntsev [18], who assigned wavebands at 510 and 770 cm^{-1} to $H_2SiO_4^{4-}$ ions and a waveband at 780 cm^{-1} to $[Si(OH)_6]^{2-}$ ions. They thought that thaumasite contained silicon in both 4- and 6-co-ordination with oxygen/hydroxyl.

The spectroscopic uncertainties about the co-ordination of silicon were finally resolved by investigation of the laser Raman spectrum of thaumasite [3] and an infrared examination involving the preparation of the chromate analogue of thaumasite [14].

The Raman spectrum of thaumasite showed an absence of wavebands which could be associated with the symmetrical SiO_4^{4-} ion. Furthermore, the pattern observed for the silicate bands [13] was very different from the one produced by tetrahedral silicate ions SiO_4^{4-} in solution [19], and also from the spectra of cementititious silicates, like alite and belite [14,20,21], which showed clear evidence of SiO_4^{4-} tetrahedra, unlike for thaumasite.

In order to check out the ideas of Kirov and Poulieff,[13] who had suggested that the infrared vibration in thaumasite at 1100 cm^{-1} was a composite band due to both SiO_4^{2-} and SiO_4^{4-} ions, chromate–thaumasite was prepared and studied by infrared spectroscopy. Sulphate wavebands can be characterised on the basis of the well-known isomorphism of sulphates with chromates, where, in the corresponding minerals, the infrared wavebands are largely similar apart from the relative positions of those wavebands due to SO_4^{2-} or CrO_4^{2-}. The CrO_4^{2-} wavebands arise at lower wavenumbers than the SiO_4^{2-} ones. In this way, the actual positions of SiO_4^{2-} wavebands can be confirmed [22,23]. This technique is somewhat analogous to the characterisation of H_2O and OH^- bands in compounds by deuteration, where the corresponding D_2O and OD^- wavebands are shifted to lower wavenumbers, whilst the wavebands of other functional groups remain largely unchanged [24,25]. The strong band at 1100 cm^{-1} in thaumasite had moved to 880 cm^{-1} in chromate–thaumasite, confirming that the 1100 cm^{-1} in thaumasite is a sulphate waveband. Some minor absorption remaining near 1100 cm^{-1}, which was revealed in chromate–thaumasite, is due either to an Si–O–H bending mode, or to any residual calcium silicate hydrate that had not reacted to form chromate–thaumasite. This absorption was not of the type expected for an SiO_4 stretching mode in chromate–thaumasite and hence in thaumasite. Also, the vibrations at 640 and 590 cm^{-1} in thaumasite are sulphate bands, since they are absent from the spectrum of chromate–thaumasite [14]. Therefore, the infrared spectrum of thaumasite shows Si in 6-co-ordination with OH, which supports the earlier conclusions of Moenke [10].

$^{29}Si\{^1H\}$ cross polarisation magic-angle spinning nuclear magnetic resonance has shed more light on the $Si(OH)_6$ groups in thaumasite, which has enabled both qualitative and quantitative determinations of thaumasite in cementitious materials to be made [26]. Different geometries for the AlO_6 octahedra in ettringite and for the SiO_6 octahedra in thaumasite, which permit substitution of Si for Al in ettringite to give a different resonance from that found in thaumasite, enable clear unambiguous quantification of thaumasite to be obtained in the presence of ettringite [26], which is not always what happens with other techniques [27].

3. Formation of thaumasite

3.1. Chemical reactions

Thaumasite is produced experimentally by the interaction of starting materials optimally in stoichiometric proportions for the formation of thaumasite, preferably at 0–5°C and certainly below approx. 10°C. The starting materials should include:

- A source of calcium silicate, such as calcium oxide (or hydroxide) plus silica, tricalcium silicate Ca_3SiO_5, dicalcium silicate Ca_2SiO_4 preferably in the β-form.
- A source of calcium sulphate, like gypsum $CaSO_4{\cdot}2H_2O$, the hemihydrate (bassanite) $CaSO_4{\cdot}0.5H_2O$ or anhydrite $CaSO_4$.
- A source of calcium carbonate, for instance calcite $CaCO_3$ or atmospheric carbon dioxide interacting with a source of Ca^{2+} ions.
- Excess water.

In other words, thaumasite is formed by a general reaction involving calcium ions, silicate, sulphate, carbonate and sufficient water to permit both transport of the potentially reactive species and to form thaumasite where these species can interact under the low temperature conditions prevailing. This is a very important point.

The silicates need to be hydraulic so that the main cementitious binder calcium silicate hydrate C–S–H can be forming, in order that thaumasite can be produced. The general chemical reaction can be expressed as follows using the approximate formula $Ca_3Si_2O_7{\cdot}3H_2O$ for the non-stoichiometric calcium silicate hydrate binder C–S–H:

$$\text{“}Ca_3Si_2O_7{\cdot}3H_2O\text{”} + 2\{CaSO_4{\cdot}2H_2O\} + CaCO_3 + CO_2 + 23H_2O \rightarrow Ca_6[Si(OH)_6]_2(CO_3)_2(SO_4)_2{\cdot}24H_2O$$

or

$$\text{“}Ca_3Si_2O_7{\cdot}3H_2O\text{”} + 2\{CaSO_4{\cdot}2H_2O\} + CaCO_3 + 24H_2O \rightarrow Ca_6[Si(OH)_6]_2(CO_3)_2(SO_4)_2{\cdot}24H_2O) + Ca(OH)_2$$

The reaction is slow and can often take 6 months to 1 year or more to obtain significant yields of thaumasite.

Thaumasite can also be formed slowly at low temperatures, as above, from the interaction of ettringite, with which it can also enter into partial solid solution, with calcium silicate hydrate C–S–H and calcite or atmospheric CO_2 in the presence of Ca^{2+} ions:

$$Ca_6[Al(OH)_6]_2(SO_4)_3{\cdot}26H_2O + \text{“}Ca_3Si_2O_7{\cdot}3H_2O\text{”} + CaCO_3 + CO_2 + xH_2O \rightarrow Ca_6[Si(OH)_6]_2(CO_3)_2(SO_4)_2{\cdot}24H_2O + CaSO_4{\cdot}2H_2O + Al_2O_3{\cdot}xH_2O + 3Ca(OH)_2$$

The calcium hydroxide $Ca(OH)_2$ formed here can readily carbonate:

$$Ca(OH)_2 + CO_2 \rightarrow CaCO_3 + H_2O.$$

The equation for thaumasite formation from ettringite is only approximate because some of the aluminium can be contained in solid solution within the thaumasite structure. Also, there is some iron(III) in solid solution with aluminium(III) in ettringite, particularly at late ages when ferrite phase hydration is substantial.[28] This would result ultimately in an iron(III) oxide gel $Fe_2O_3{\cdot}yH_2O$ along with the alumina gel when thaumasite is formed from ettringite.

3.2. Significance of the 6-co-ordination of silicon by hydroxyl in thaumasite

At a first glance the formation of thaumasite seems somewhat puzzling, since it only forms at low temperatures. However, once formed, it is stable up to approx. 110°C, when it decomposes to form the disordered structure known as thaumasite glass. This stability is to be contrasted with that of its close structural relative ettringite which, although formed at ordinary temperatures, suffers a lattice collapse at approx. 60°C and decomposes at approx. 90°C. Most structures that contain silicon in 6-co-ordination with oxygen are formed at high pressures and usually high temperatures [29]. However, a few, like certain silicon phosphates and thaumasite are formed either at or near atmospheric pressure [29].

The reason why thaumasite only forms at low temperatures can be attributed to the need to form a transition state intermediate involving a hexagonal arrangement of OH^- ions around a core Si^{4+} ion long enough to allow charge delocalisation away from the embryonic $[Si(OH)_6]^{2-}$ groups as they are being formed onto the carbonate groups present; this allows a stable, configuration of $[Si(OH)_6]^{2-}$ octahedra to be produced. Clearly, as indicated from the appearance of the infrared and Raman spectral bands attributed to SiO_6 [3,10,14], the symmetry is heavily distorted away from O_h to lower symmetries. In other words, a low temperature is essential for producing the transition state intermediate for thaumasite to form. Once formed, however with the necessary charge delocalisation for stabilising the $[Si(OH)_6]^{2-}$ groups, the thaumasite structure is stable up to approx. 110°C. Unlike for ettringite, there is no lattice collapse within thaumasite preceding its true decomposition. Structures containing silicon in 6-co-ordination with oxygen are very rare,[29] unlike for germanium [30]. The only known structures containing $[Si(OH)_6]^{2-}$ groups are thaumasite and its chromate analogue $Ca_6[Si(OH)_6]_2(CO_3)_2(CrO_4)_2 \cdot 24H_2O$. The hydrated sodium silicate $Na_2SiO_3 \cdot 3H_2O$, previously thought to be composed of $Na_2Si(OH)_6$ units [29], is now known to contain silicon in 4-co-ordination with oxygen (J. Bensted, unpublished).

3.3. General considerations

It is clear that the non-binder thaumasite can be formed from the binder calcium silicate hydrate C–S–H in the presence of carbonates and/or atmospheric carbon dioxide, sulphates and moisture at low temperatures, optimally 0–5°C, where reaction conditions like relative solubilities are at their most favourable. It can also be produced from ettringite and C–S–H when carbonate and/or CO_2 and sufficient water are present at the low temperatures measured. In the past, thaumasite formation was not readily recognised, because of its similarity, when measured by various techniques, to carbonated ettringite [27]. Thaumasite, when produced in such structures, like C–S–H, ettringite etc. is not a pure mineral and contains other cations and anions in solid solution.

However, now thaumasite is more readily known about, it is being located more frequently in structures where deterioration is taking place. An example of this is in foundations of buildings where the temperatures may commonly average approx. 10°C when the ambient temperatures are around 20°C. Also, where limestone aggregates have been employed in concrete, the fines content in particular can participate in the thaumasite formation reaction. Limestone 'fillers' in cement and Portland limestone cement, where up to 35% ground-in limestone may be contained, can be susceptible to thaumasite formation if the conditions are right. Also, sulphate-resisting Portland cement can be subject to thaumasite formation, again if the conditions are right.

The thaumasite formation reaction effectively ceases when the temperature rises above approx. 15°C, but starts being produced again when the temperature is lowered below 15°C, and especially when it lies between 0 and 5°C. In relative small amounts locally it may not be serious if the ratio of binder C–S–H to non-binder thaumasite remains large enough not to affect the basic structural integrity. However, if this ratio becomes small, then the structure is in danger of serious deterioration. Figures for safe or unsafe ratios cannot be generalised, as they would depend upon the precise circumstances and locations for the structures in question.

Although thaumasite formation cannot be prevented in toto if the temperatures are low enough, its negative effects can be minimised by employing water/cement ratios as low as practicable (for good workability considerations) in cements, mortars and concretes. This will allow low permeabilities to be obtained, which will prevent, or at least seriously frustrate, the transport of ions like Ca^{2+}, CO_3^{2-}, SO_4^{2-} and sufficient moisture to the hardened cement surfaces. Good mix design is a key consideration in seeking to avoid any potential structural problems that might arise if thaumasite is to be formed.

4. Conclusion

The background and nature of the formation of thaumasite, a non-binding calcium carbonate silicate sulphate hydrate, in the deterioration of cements, mortars and concretes is discussed. It is known that thaumasite is readily formed when the hardened cement binder calcium silicate hydrate C–S–H is in contact with Ca^{2+}, carbon dioxide, carbonates, sulphates and sufficient moisture at low temperatures below approx. 15°C and particularly at approx. 0–5°C. Such formation is becoming more clearly recognised now that thaumasite is being properly identified and not confused with carbonated ettringite. Limestone is not chemically inert in hardened cements, mortars and concretes, but can react, if the conditions are right, to produce thaumasite. This can arise in limestone aggregates with the (more reactive) fines content, in Portland limestone cement and masonry cements containing ground chalk or limestone, as well as in sulphate-resisting Portland cements.

Indeed, thaumasite has been found in large quantities in some foundation concretes examined [31] without any evidence of structural damage above ground, indicating that similar problems might occur elsewhere and have so far gone unreported. Formation of thaumasite can be accompanied by swelling or expansion of the affected concrete, and added problems of freeze–thaw probably contribute to the degradation process [31].

The potential for structural problems with thaumasite may not arise in practice if the mortars and concretes are well made, using a low water/cement ratio to minimise internal transport of ions like Ca^{2+}, CO_3^{2-}, SO_4^{2-} and of water as the carrier for them. If this is achieved, when the temperature falls below 15°C such that formation of thaumasite can arise, then the relative absence of these ions and of water to transport them will ensure that thaumasite is not formed in such localities and deterioration from its formation will not ensue there. Conversely, where mortars or concretes are badly produced at high water/cement ratios, where internal transport of the aforementioned ions and water is facilitated, then, when the temperature is sufficiently low, thaumasite can readily form [32].

Awareness of the dangers that thaumasite can bring should permit designs of more mortars and concretes to minimise its potentially destructive occurrence.

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