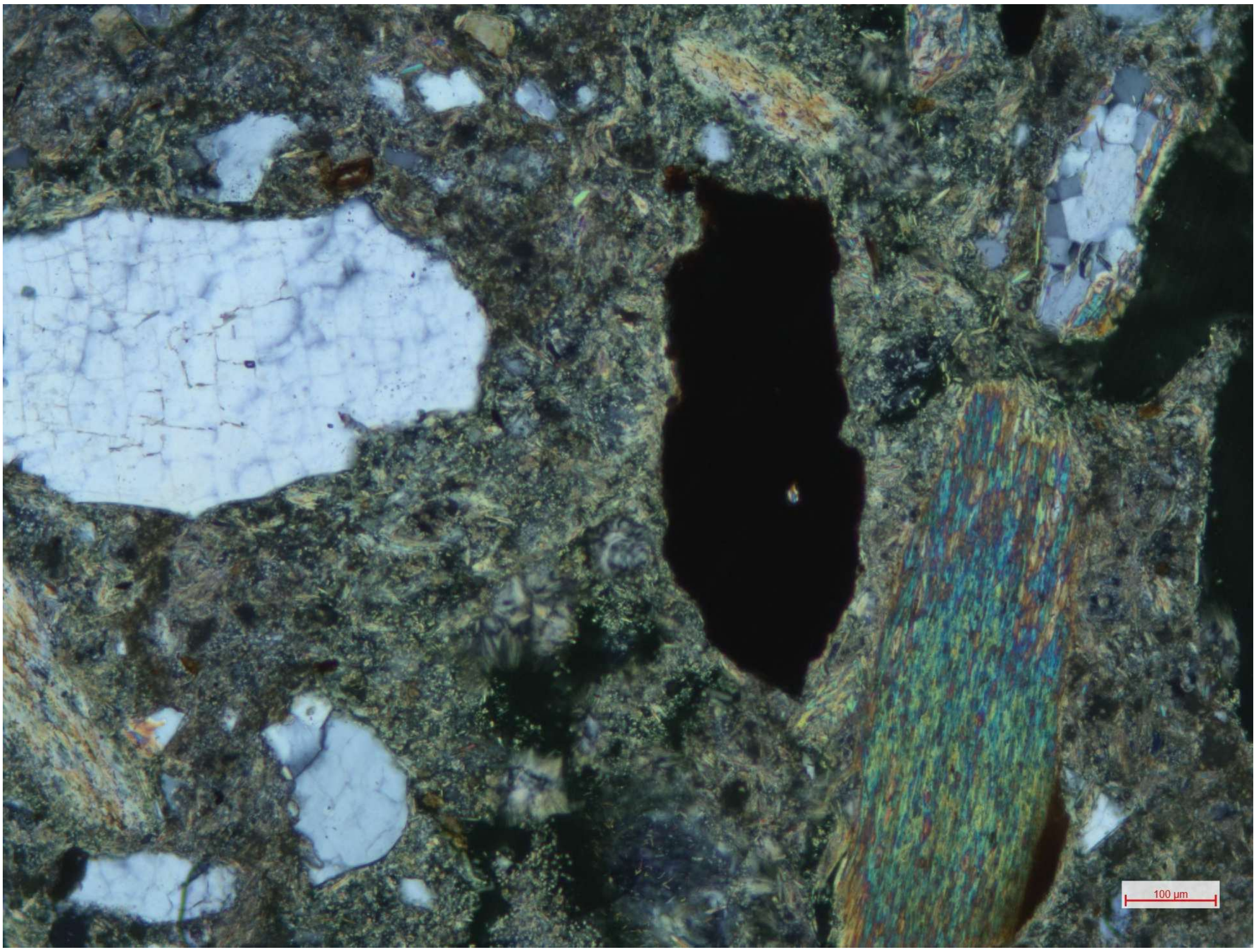

Sample 20511 C26 C CPL Photomicrograph of oxidising pyrrhotite surrounded by sulfate conversion of the cement matrix after CSA P3 adapted durability testing.

Department of Housing, Local Government and Heritage, and Geological Survey Ireland

Pyrrhotite-bearing concrete investigation, Co. Donegal

Laboratory Analysis Services in support of Geological Survey Ireland's "Irish Construction Materials" Project: Concrete Products

Phases 2 and 3 Report

1283831-04 (01)

MARCH 2025

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Report No.: 1283831-04 (01)

Title: Pyrrhotite-bearing concrete investigation, Co. Donegal - Laboratory Analysis Services in support of Geological Survey Ireland's "Irish Construction Materials" Project: Concrete Products Phases 2 and 3

Client: Department of Housing, Local Government and Heritage and Geological Survey Ireland

Date: 14 March 2025

RSK Office: 18 Frogmore Road, Hemel Hempstead, Hertfordshire, HP3 9RT

Status: Final

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Date:	14 March 2025	Date:	14 March 2025

GLOSSARY

21GD – 21 Glendale Drive (test property)

28AW – 28 Abbotts Wood (test property)

7MV – 7 Mulroy View (test property)

AMBT – Accelerated mortar bar test (CSA A23,1:19/A23.2:19 P3)

AOI – Area of interest

ASS – Acid soluble sulfate

ASTM – American Society of Testing and Materials

BS – British Standard

BSE – Backscatter electron

C – Carrowmore (control property)

CPL – Circular-polarised light

CSA P3 adapted - CSA A23,1:19/A23.2:19 Attachment P3 (informative) adapted oxidisation test

C-S-H – Calcium silica hydrates

DPM – Damp-proof membrane

EDX – Energy dispersive X-ray

F – Foundation

GSI – Geological Survey Ireland

HTC – High-temperature combustion

IL – Inner leaf

In situ – Whilst present within concrete (or in place within the original structure)

ISA – Internal sulfate attack

ITZ – Interfacial transition zone, cement-aggregate interfacial zone within the cement matrix.

NSAI – National Standard Authority of Ireland

OL – Outer leaf

OM – Optical microscopy

OS – Oxidisable sulfates

P3 Phase 1 – Phase 1 of the CSA A23,1:19/A23.2:19 Attachment P3 (informative) adapted oxidisation test.

P3 Phase 2 – Phase 2 CSA A23,1:19/A23.2:19 Attachment P3 (informative) adapted oxidisation test

Phase 1 – Initial characterisation testing (RSK Investigation)

Phase 2 – Accelerated durability testing (RSK Investigation)

Phase 3 – Post- durability testing, comparative testing (RSK Investigation)

PPL – Plane-polarised light

Pre-existing – Before use within concrete

RICS Stage 3 – RICS The Mundic Problem, Stage 3 expansion testing

RL – Reflected light

RW – Rising wall

SE – Secondary electron

SEM – Scanning electron microscope/Scanning electron microscopy

TPS – Total potential sulfate

TS – Total sulfur

TSA – Thaumasite form of sulfate attack

TTF – Time-to-failure

WSS – Water soluble sulfate

XPL – Cross-polarised light

XRD – X-Ray Diffraction

1 INTRODUCTION

1.1 Context

Concrete blocks containing deleterious aggregates were found to have been used in the Northwest of Ireland's domestic construction, primarily in the late 1990s until the late 2000s, during a period of Irish economic growth referred to as the 'Celtic Tiger' and its associated building boom. Initially, the issue was thought to be chiefly related to either or both muscovite mica contents or reactive pyrite (with different causes in different areas) and thought possibly to affect approximately 5,000 homes.[1] The scale of the problem led to the establishment of a multi-billion euro fund to assist the affected homeowners, and the introduction of Irish standard I.S. 465 giving sampling protocols and laboratory test methods to investigate the affected concrete.[2] However, the recent experience of consultants and academic research have highlighted the presence and oxidisation reactions of pyrrhotite as the most common cause of deterioration in many of the affected properties, through the mechanism of internal sulfate attack (ISA).[3,4]

Concrete blocks affected by ISA typically experience a primary deterioration mechanism triggered by the expansive oxidisation (rusting) of iron sulfides, resulting in the release of sulfuric acid and subsequent dissolution, alteration and weakening of the cement matrix, eventually leading to conversion of the cement matrix and failure of the concrete. These reactions are in part represented by the following equations well documented in the literature.[5,6,7]

$Fe_{(1-x)}S$ (pyrrhotite) + $(2-x/2)O_2 + xH_2O \rightarrow (1-x)Fe^{2+} + SO_4^{2-} + 2xH^+$

$Fe^{2+} + 1/4O_2 + 2H^+ \rightarrow Fe^{3+} + 1/2H_2O$

$Fe^{3+} + 3H_2O \rightarrow Fe(OH)_3$ (ferric oxyhydroxide, expansive) + $3H^+$

H_2SO_4(sulphuric acid) + $Ca(OH)_2$ (portlandite)$\rightarrow CaSO_4.2H_2O$ (gypsum)

$3(CaSO_4.2H_2O) + C_3A + 26H_2O \rightarrow C_3A{\cdot}3CaSO_4{\cdot}H_{32}$ (ettringite)·

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1 McCarthy, D. Kane, N. Lee, F. Blaney, D. Report of the Expert Panel on Concrete Blocks, 2017, https://www.gov.ie/en/publication/0218f-report-of-the-expert-panel-on-concrete-blocks/.

2 I.S. 465:2018+A1:2020, Assessment, Testing and Categorisation of Damaged Buildings Incorporating Concrete Blocks Containing Certain Deleterious Materials and Amendment 1, National Standards Authority of Ireland, 2020

3 A. Leemann, B. Lothenbach, B. Münch, T. Campbell, P. Dunlop, The "mica crisis" in Donegal, Ireland – a case of internal sulfate attack? Cem. Concr. Res. 168 (2023).

4 C. Brough, B. Staniforth , C. Garner , R. Garside , R. Colville , J. Strongman , J. Fletcher, High risk concrete blocks from County Donegal: The geology of defective aggregate and the wider implications, Construction and Building Materials 408 (2023) 133404

5 A. Rodrigues, J. Duchesne, B. Fournier, B. Durand, P. Rivard, M. Shehata, Mineralogical and chemical assessment of concrete damaged by the oxidation of sulfide-bearing aggregates: Importance of thaumasite formation on reaction mechanisms, Cement and Concrete Research, Volume 42, Issue 10, 2012, Pages 1336-1347, ISSN 0008-8846,

6 R. Zhong, K. Wille, Deterioration of residential concrete foundations: the role of pyrrhotite-bearing aggregate, Cem. Concr. Compos. 94 (2018) Pages 53–61.

7 D. Jana, Concrete Deterioration from the Oxidation of Pyrrhotite: A State-of-the-Art Review, Chapter 5, Maher, M.L.J, Pyrite and Pyrrhotite, 2023, Pages 139-221, ISBN 979-8-88697-329-7, Nova Science Publishers, Inc.

1.2 Instructions

To address the deterioration of thousands of homes affected by concrete block deterioration, the Irish Government Department of Housing, Local Government and Heritage in conjunction with the Geological Survey of Ireland (GSI) and National Standards Authority of Ireland (NSAI) established a research framework titled 'Laboratory Analysis Services in support of Geological Survey Ireland's "Irish Construction Materials" Project: Concrete Products'. Under this framework, GSI provided RSK and other research partners with a set of research topics to assess. These topics are summarised as follows:

- Long-term performance of the concrete blocks under Irish environmental conditions, e.g. accelerated ageing testing.
- Whether and under what circumstances the pyrrhotite within concrete blocks could oxidise.
- Risk of oxidation occurring within the range of environmental conditions found in Ireland.
- If oxidation occurs, what effect that process has on the integrity, dimensional properties, and compressive strength of the blocks relative to their state when received for testing.
- Risk of retention of pyrrhotite-bearing blockwork in affected dwelling.

It should be noted that RSK's submission was primarily limited to the underlined research topics.

The framework further expanded on the 4th research topic.

- If pyrrhotite alteration occurs in the concrete blocks assessed, does it:
  1. result in the expansion of the samples?
  2. affect the compressive strength of the samples?
  3. significantly impact the integrity of the samples?

To investigate the provided research topics GSI provided RSK with a sample set of pyrrhotite-bearing concrete blocks including some with adhered render and mortar, and mass concrete from buildings that have previously been evaluated in accordance with I.S. 465.[8] The sample set included a mixture of blocks and cores taken from the inner leaves, outer leaves, rising walls (excluding the control property) and foundations of four buildings, three test properties and one control property. The sample set was designed to be representative of blocks *in situ* (in use), to understand the risk posed by pyrrhotite and reflect the aggregate constituents of damaged and undamaged properties selected.

[8] I.S. 465:2018+A1:2020, See [2]

1.3 Objective

To investigate the instructed research topics (underlined in **1.2**) RSK developed a three-phase investigation as follows.

- Phase 1 – Initial characterisation of the received samples
- <u>Phase 2 – Durability - Accelerated moisture exposure and oxidisation (expansion) testing</u>
- <u>Phase 3 – Post-expansion testing characterisation and comparison</u>

This report is exclusively limited to the analyses conducted on samples supplied to RSK. It forms the Phase 2 accelerated durability testing and Phase 3 post-durability testing analysis to investigate the potential for further deterioration within the concrete of the supplied samples. The characterisation will concentrate on the following:

- Performance during durability testing.
- Physical condition.
- Relative comparisons with Stage 1 testing.
- Development of any deterioration mechanisms.
- Potential for further deterioration.

The results and conclusions presented herein will be (where possible) compared with the baseline for the Phase 3 post-durability testing of concrete blocks under accelerated ageing conditions.

Purposely, references to Phase 1 data are only made when relevant for comparative purposes. Phase 1 results are presented in more detail within RSK report, 1283831-01. Phase 2 and Phase 3 foundation analysis is discussed in detail within RSK report 1283831-03, however, data and limited comment are presented herein for the foundations.

2 SITE WORK

IGSL provided RSK with the sampling locations and details in a report entitled 'Donegal Concrete Blocks GSI Research Programme, Sampling Operations Record, April 2023', of which an extract is attached in **Appendix B** of this report[9].

2.1 Sampled properties

The properties were selected by Donegal County Council and advised to have been taken from four vacated houses in Co. Donegal. Three of the houses were considered test properties, these were 15-25 years old and were known to have experienced structural damage/defects typical of the area and identified in accordance with I.S. 465.[10][11] The other 'control' property was known to have been constructed in the 1980's and be in apparently good condition. The properties are listed below.

Test Properties

- 7 Mulroy View, Co. Donegal, (7MV)
- 21 Glendale Drive, Co. Donegal, (21GD)
- 28 Abbotts Wood, Co. Donegal, (28AW)

Control Property

- Carrowmore, Co. Donegal, (C)

The properties are typically constructed of poured mass concrete strip foundations with rising walls made of flat-laid precast concrete blocks (aggregate cement masonry units) built up from the foundations to a dampproof membrane (DPM). Above the DPM, there is a cavity wall made up of two leaves of concrete blocks laid on edge and cemented by a sand cement mortar (see **Figure 2-1**). The wall cavities at all properties were filled with apparent polystyrene-type insulation of various appearances. The outer leaf is often coated with a painted cementitious external render, whilst the inner leaf is coated by an internal plaster. The control property is notable for not being constructed with a DPM or a rising wall. Instead, both leaves of the cavity wall construction extend to the foundations below ground.

2.2 Sampling

The sampling was advised to have been conducted on behalf of Donegal County Council by Crana Cranes Ltd (advised to be a Donegal County Council approved sub-contractor for I.S. 465[12] sampling) under the supervision of IGSL (at the request of GSI). The sampling took place between 9th-12th January 2023, in conditions which appeared to be wet or overcast with saturated groundwater conditions based on supplied photographic evidence (**Figure 2-1**). The IGSL engineering geologist was advised to be Sean Cunningham who oversaw the works and photographed and recorded sample locations (see **Appendix B**).

[9] Quigley. P, (2023) 'Donegal Concrete Blocks GSI Research Programme, Sampling Operations Record', IGSL.

[10] Quigley. P, (2023) Donegal Concrete Blocks, IGSL. See [9]

[11] I.S. 465:2018+A1:2020, See [2]

[12] I.S. 465:2018+A1:2020, See [2]

Figure 2-1 Examples of sampling areas

1st Row Left – 7MV inner leaf sampling location. 1st Row Right – 21GD Rising Wall and Foundation sampling location. 2nd Row Left – 28AW An outer leaf sampling location. 2nd Row Left – C outer leaf sampling location. Photos taken from IGSL Sampling Reports. Source IGSL.

A selection of blocks and cores were extracted from the inner leaf, outer leaf, rising wall (where possible) and strip foundation building elements. Core samples were taken by coring rigs using approximately 100 mm inner diameter barrels. Trial pits were excavated with a digger to expose the foundations and rising walls (see **Figure 2-1**). More details of the sampling can be found in the sampling report and **Appendix B**.[13]

[13] Quigley. P, (2023) Donegal Concrete Blocks, IGSL. See [9]

3 SAMPLES – RSK

The samples received by RSK were advised to have first been transported from the sampled properties to IGSL's laboratory in Naas, Co. Kildare. There, samples were assigned to RSK and other research partners.

RSK received the assigned 46 sample batch comprising concrete blocks and concrete cores from the four properties on 28th March 2023. After a request for a further six samples from 28AW in February 2024, in total, RSK received 52 samples for analysis (see **Appendix A**).

Samples were photographed (see **Figure 3-1**, **Appendix D**) logged into RSK's laboratory sample management system and given unique sample references (see **Table 3-1**).

Figure 3-1 Examples of as-received samples

Photos of a variety of as-received sample types and conditions from across all four properties. 1st Row Left, 7MV IL intact. 1st Row Right, 21GD RW intact. 2nd Row Left, 28AW OL disintegrated. 2nd Row Right, C F fragmented. For references see Table 3-1.

Table 3-1 Samples received by RSK

Location	RSK ref	Client sample ref	Sample type	Element	Client area location	Date sampled	As-received condition
7MV	20511/B1	1A	Block	Inner Leaf	I, GE	10/01/2023	F/C
	20511/B2	1B	Block	Inner Leaf	I, GE	10/01/2023	In
	20511/B3	1G	Block	Inner Leaf	I, GE	10/01/2023	SC
	20511/C1	1L	Core	Inner Leaf	I, GE	10/01/2023	In
	20511/B4	2A	Block	Outer Leaf	E, FF, W	10/01/2023	In
	20511/B5	2D	Block	Outer Leaf	E, FF, W	10/01/2023	In
	20511/C2	2I	Core	Outer Leaf	E, FF, W	10/01/2023	In
	20511/C3	2M	Core	Outer Leaf	E, FF, W	10/01/2023	In
	20511/C4	3B	Core	Rising Wall	E, FF, W	10/01/2023	In
	20511/C5	3F	Core	Rising Wall	E, FF, W	10/01/2023	In
	20511/C6	3I	Core	Rising Wall	E, FF, W	10/01/2023	F
	20511/C7	4B	Core	Foundation	E, FF, W	10/01/2023	F/C
	20511/C8	4E	Core	Foundation	E, FF, W	10/01/2023	In
	20511/C9	4G	Core	Foundation	E, FF, W	10/01/2023	In
21GD	20511/B13	1A	Block	Inner Leaf	I, GE	09/01/2023	SD
	20511/B14	1D	Block	Inner Leaf	I, GE	09/01/2023	In
	20511/C22	1G	Core	Inner Leaf	I, GE	09/01/2023	In
	20511/B15	2B	Block	Inner Leaf	I, GE	09/01/2023	In
	20511/C23	2E	Core	Inner Leaf	I, GE	09/01/2023	SD
	20511/B18	3A	Block	Outer Leaf	E, GE	09/01/2023	In
	20511/B16	3D	Block	Outer Leaf	E, GE	09/01/2023	In
	20511/B17	3F	Block	Outer Leaf	E, GE	09/01/2023	In
	20511/C24	3J	Core	Outer Leaf	E, GE	09/01/2023	In
	20511/C25	4D	Core	Rising Wall	E, GE	09/01/2023	In
	20511/C26	4H	Core	Rising Wall	E, GE	09/01/2023	In
	20511/C27	5B	Core	Foundation	E, GE	09/01/2023	In
	20511/C28	5E	Core	Foundation	E, GE	09/01/2023	In
28AW	20511/B8	1A	Block	Inner Leaf	I, GE	11/01/2023	In
	20511/B9	1E	Block	Inner Leaf	I, GE	11/01/2023	SD
	20511/B10	1I	Block	Inner Leaf	I, FF	11/01/2023	In
	20511/B11	2A	Block	Outer Leaf	E, GE	11/01/2023	C
	20511/B12	2F	Block	Outer Leaf	E, GE	11/01/2023	F/C
	20511/C16	2I	Core	Outer Leaf	E, GE	11/01/2023	Di
	20511/C17	2M	Core	Outer Leaf	E, GE	11/01/2023	Di
	20511/C18	3B	Core	Rising Wall	E, GE	11/01/2023	In
	20511/C19	3F	Core	Rising Wall	E, GE	11/01/2023	In
	20511/C20	4A	Core	Foundation	E, GE	11/01/2023	In
	20511/C21	4D	Core	Foundation	E, GE	11/01/2023	In
	20954/B1	2H	Block	Outer leaf	E, GE	11/01/2023	F/C
	20954/C1	2K	Core	Outer leaf	As E, GE	11/01/2023	In
	20954/C2	2L	Core	Outer leaf	As E, GE	11/01/2023	In
	20954/C3	3H	Core	Rising wall	E, GE	11/01/2023	In
	20954/C4	3I	Core	Rising wall	E, GE	11/01/2023	In
	20954/C5	4C	Core	Foundation	E, GE	11/01/2023	In
C	20511/B6	1A	Block	Inner Leaf	I	12/01/2023	In
	20511/C10	1E	Core	Inner Leaf	I	12/01/2023	F
	20511/C11	1F	Core	Inner Leaf	I	12/01/2023	In
	20511/B7	2B	Block	Outer Leaf	E, FF	12/01/2023	In
	20511/C12	2F	Core	Outer Leaf	E, FF	12/01/2023	In
	20511/C13	3A	Core	Below GL	E, GE	12/01/2023	In
	20511/C14	4A	Core	Foundation	E, GE	12/01/2023	In
	20511/C15	4C	Core	Foundation	E, GE	12/01/2023	F

I-Interior, E-Exterior, GE-Gable end, FF-Front Facing, W-West, As-Assumed, F-Fragmented, C-Crumbly, In-Intact, SC-Slightly Crumbly, SD-Slight deterioration, Di-Disintegration

3.1 Sub-sampling

Sub-sampling was conducted on the samples once they had equilibrated to laboratory conditions whilst protecting the samples from excessive carbonation and drying out.

Blocks were sub-sampled for RICS Stage 3 moisture sensitivity expansion testing[14] and the residual oxidisation potential CSA test[15] using dry coring techniques. This was conducted to minimise the flow of water through the samples, which could result in additional oxidisation or unrecorded alteration of the as-sampled state. If, however, dry coring failed to retrieve samples due to the extra stress caused by dry coring and poor sample condition, a minimal amount of water was employed to facilitate sample retrieval. It was noted that many of the test property blocks were not in a suitable condition for any intact sub-sampling to be conducted.

Other sub-sampling was conducted through sawing with a diamond blade using the minimum amounts of water or without the use of water.

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[14] RICS Guidance Note. (2015). The Mundic Problem, RICS Professional Guidance Note, UK. 3rd edition. London: Royal Institution of Chartered Surveyors (RICS). ISBN 978 1 78321 094 7

[15] CSA A23.1:19/CSA A23.2:19 Concrete materials and methods of concrete construction/Test methods and standard practices for concrete, P3, Pages 370-381, CSA, Canada, ISBN 978-1-4883-0744-7

4 LABORATORY METHODS

4.1 RICS, The Mundic Problem, Stage 3 expansion test

Expansion testing was conducted in accordance with RICS guidance note 'The mundic problem, 3rd edition'.[16] The Stage 3 moisture sensitivity expansion test is primarily applicable to concrete blocks from a specific area of the Southwest region of the UK where spoil from metalliferous mining activities have historically been used locally as aggregate. Therefore, any criteria should not be thought to apply outside of that regional use nor be directly applicable for cast *in situ* concrete as with the foundation samples tested. The method involves measuring the unconstrained linear expansion of concrete cores that have been exposed to a water-saturated atmosphere at a constant temperature of 38°C for at least 250 days. The testing period can be expanded to at least 350 days if the expansion shown is progressing at a slow rate when 250 days of exposure is reached. During this investigation, all cores except 28AW OL cores (250 days) were kept in exposure conditions for 350 days to provide further time for any reactions to occur. This is particularly relevant for the foundation cores given that the test is designed for less dense and therefore more porous and permeable concrete blockwork samples rather than dense mass concrete samples.

4.2 CSA A23,1:19/A23.2:19 Attachment P3 (informative) adapted oxidisation test

To determine residual oxidisation potential and the possibility of thaumasite formation, an adaption of the test method developed by Andrea Rodrigues at Université Laval and incorporated in CSA A23, 1:19/CSA A23, 2:19, P3.[17] The methodology describes a procedure for determining the potential deleterious character of sulfide-bearing[18] aggregates through a two-phase accelerated mortar bar test. In this study, the methodology was adapted to test the concrete core samples with similar sample sizes and stud arrangements specified within RICS guidance note 'The mundic problem, 3rd edition'.

To prepare the sub-samples three pairs of bespoke titanium DEMEC Gauge studs spaced at 50 mm separation were fixed at equal intervals (120°) around the circumference of a set of up to four 75 mm diameter cores taken from the investigated elements (actual number dependent on sample availability).

Cores underwent immersion in 6% sodium hypochlorite for 3hrs±15min and were then removed, weighed and measured as a zero reading, then left to dry for 3hrs±15min. After drying, samples were stored above a saturated sodium chloride solution (75 %RH) at 80 °C. The bleach utilised was Ki-Chem UK Limited t/a Cleaning supplies 2U, 6% Bleach, which had an advertised NaOCl content of 6 % and determined pH of 12.3 and a NaOH concentration of 0.07 wt % (internal testing and calculation).

[16] RICS, The Mundic Problem. See [14]

[17] CSA A23.1/CSA A23.2, Concrete test methods, P3, See [15]17

[18] The method has been specifically developed following the construction of defective buildings incorporating concrete with pyrrhotite-bearing aggregates in the Trois Rivieres / Shawinigan areas of Quebec.

Every week, the cores underwent two immersion periods in the 6% sodium hypochlorite (as specified above) and once a week, after an immersion period, the length, mass and condition of each core were taken and recorded.

After 13 weeks of storage at 80 °C and 75 %RH (P3 phase 1) between immersions, samples transitioned to storage above water at 4°C (P3 phase 2) and continued the twice-weekly immersions in sodium hypochlorite and once-a-week measurements.

Samples were taken off test if they had disintegrated, lost structural integrity or had lost measuring stud adhesion repeatedly in P3 Phase 1. However, in P3 Phase 2, if the same sample deterioration as previously mentioned above occurred, requiring that measurements were stopped, disintegrated or failed samples continued to go through the cycling but in a perforated holding container to restrain the samples whilst permitting the possibility of thaumasite formation to occur and allow an equal comparison of the samples through the duration of the test (where possible).

4.3 Petrographic examination – ASTM C856-20[19] and I.S. 465 7.3

For investigated samples, one polished and one coverslipped thin section and a polished slice (approximately up to 100×100×20 mm sized, Phase 1 only) were produced using either the minimum of water required or alternative grinding media (e.g. ethylene glycol). Examination of the concrete was chiefly conducted using a polarising Zeiss Axioscope A1 petrographic microscope, utilising reflected, transmitted and reflected UV light sources.

4.4 SEM/EDX analysis

SEM/EDX analysis was conducted at an RSK-approved sub-contractor with on-instrument consultation given as needed by RSK personnel. Various sizes of concrete samples ranging from 25×25×25mm to 50×30×25 mm were vacuum impregnated with epoxy resin, polished to a 3-micron finish, and carbon-coated on a single face for analysis.

A JEOL 6480 LV SEM equipped with an Oxford Instruments X-MAX80 SD X-ray detector and INCA x-ray analysis system was used to image the samples and perform the EDX analysis. EDX analyses the characteristic X-rays produced by the interaction between the primary electron beam and the sample. The technique identifies all elements present with atomic numbers of 5 (boron) and greater with a detection limit of approximately 0.1 weight % with all measurements semi-quantitative. The SEM was operated at an accelerating voltage of 15 kV.

4.5 XRD analysis (semi-quantitative)

XRD analysis was conducted at an RSK-approved sub-contractor using a fully automated Bruker D8 powder diffractometer employing copper kα radiation (λ=0.15406nm) and an energy dispersive Si detector. The samples were continuously spun during data collection and were scanned using a step size of 0.02°2θ between the range of 5°-80°2θ. Phase identification using XRD is achieved by comparing the diffraction pattern obtained from the unknown, to a standard database that is compiled by the International Centre for Diffraction Data (ICDD).

[19] ASTM C856-20, Standard practice for petrographic examination of hardened concrete, ASTM, 2020

4.6 XRF analysis (semi-quantitative)

XRF analysis was conducted at a UKAS-accredited RSK approved sub-contractor using X-ray fluorescence (XRF) spectrometry (not specifically UKAS-accredited). The sample was irradiated, causing the emission of secondary (fluorescent) X-rays to emerge from the sample material. The elements in the sample were identified by the wavelengths of the emitted X-rays while the concentrations of the elements were quantified by the intensity of those X-rays. With XRF, typically elements between atomic numbers 9 and 92 (e.g. F to U) can be detected and quantified.

4.7 Compressive strength of core samples – BS EN 12504-1:2019

A set of 1:1 length-to-diameter ratio concrete core samples were sub-sampled, prepared (ground) and tested in accordance with BS EN 12504-1.[20] Compressive strengths were compared to cube strength specifications.

4.8 Density – BS EN 12390-7:2019+AC:2020

As-received dry densities were measured in accordance with BS EN 12390-7:2019+ AC:2020[21] on 1:1 cored concrete samples to provide a check on sample compaction.

4.9 Cement content – BS 1881-124:2015+A1:2021

Analysis was performed on 1 kg of material either previously tested for compressive strength or the remnants of the sample after sub-sampling. The analyses for insoluble residue, soluble silica and calcium oxide were carried out in accordance with BS 1881-124:2015+A1:2021[22], Clause 6. Note, that the cement content calculated as kg/m^3 used the determined densities for some samples, whereas other samples used the determined densities from the same or near similar element and constituents (See **Appendix C**).

4.10 Total sulfur – BS EN 1744-1: 2009+A1:2012

The total sulfur content was determined in accordance with BS EN 1744-1[23], Clause 11 acid digestion method. The extraction was conducted using hydrogen peroxide and dilute hydrochloric acid, and the sulfur was precipitated as barium sulfate. The result is reported to the nearest 0.1% by mass of dry aggregate. Note, that the test method describes testing aggregate samples. In this case, the concrete samples were additionally prepared to pass a 2 mm sieve before the specified sieving was conducted. Concerns have been raised about the reliability of the results obtained by this technique, which appears to significantly under-estimate the total sulfur of samples where petrographic examination confirms the presence of sulfide minerals.

[20] BS EN 12504-1:2019. Testing concrete in structures. Part 1 – Cored specimens. Taking, examining and testing in compression. BSI, London, 2019.

[21] BS EN 12390-7:2019+AC:2020, Testing hardened concrete - Density of hardened concrete, BSI, London, 2020

[22] BS 1881-124:2015+A1:2021, Testing Concrete - Methods for analysis of hardened concrete, BSI, London, 2021

[23] BS EN 1744-1:2009+A1:2012, Tests for chemical properties of aggregates - Chemical analysis, BSI, London, 2012

Subsequently, additional testing was undertaken wherein powdered samples were directly tested for total sulfur content utilising high-temperature combustion and infra-red analysis (LECO). This method is provided as an alternative method in BS EN 1744-1 Clause 11.2, although the acid digestion method is the reference method. A similar method is described in the Canadian standard CSA A23.1 as their preferred method for determining total sulfur in aggregate.

4.11 Acid soluble sulfate content – BS EN 1744-1:2009+A1:2012

The acid-soluble sulfate content was determined in accordance with BS EN 1744-1, Clause 12. The extraction was conducted using dilute hydrochloric acid and the sulfate was precipitated as barium sulfate. The sulfate content is reported to the nearest 0.1% by mass of dry aggregate (sample). Note, that the test method describes testing aggregate samples. In this case, the concrete samples were additionally prepared to pass a 2 mm sieve before the specified preparation procedure was conducted. It has been suggested that the test method may cause some dissolution of pyrrhotite, and therefore the results obtained potentially represent more sulfur than consistent with the total sulfate content of the sample.[24]

4.12 Water soluble sulfate content – BS EN 1744-1:2009+A1:2012

The water-soluble sulfate content was determined in accordance with BS EN 1744-1, Clause 10.[25] The 2:1 water extract was treated with an excess of barium chloride to precipitate the sulfate as barium sulfate, which was determined gravimetrically. The result was expressed as SO_3 to the nearest 0.01 % by mass of dry aggregate (sample). Note; the test method describes testing aggregate samples. In this case, the concrete samples were additionally prepared to pass a 2 mm sieve before the specified preparation procedure was conducted. The resulting material was tested to determine the water-soluble sulfate content in accordance with BS EN 1744-1 for fine aggregate.

4.13 Pyrite/Pyrrhotite content

The tests for acid-soluble sulfate and total sulfur (HTC) were carried out in accordance with BS EN 1744-1:2009 + A1:2012.[26] The oxidisable sulfides were calculated from the determined acid-soluble sulfate content and total sulfur content according to the formula provided in TRL 447 Table 8.1 and simple conversion of oxidisable sulfates into a pyrite or pyrrhotite content.[27] It should be noted that the calculated values are the maximum potential values for these mineral contents and should be treated with caution.

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[24] Personal communication of Mike Eden (Sandberg) based on commentary on pyrrhotite solubility in textbooks such as Deer, Howie and Zussman.

[25] BS EN 1744-1, See [23]

[26] BS EN 1744-1, See [23]

[27] Reid J M, Czerewko M A and Cripps J C. Sulfate specification for structural backfills. TRL Report TRL447. Crowthorne, Transport Research Laboratory, 2005, 2nd edn

4.14 Determination of sulfate – BS EN 196-2:2013

The sulfate content was determined in accordance with BS EN 196-2:2013[28]. The acid extract was treated with an excess of barium chloride to precipitate the sulfate as barium sulfate, which was determined gravimetrically. The result is expressed as sulfur trioxide, SO_3 by weight of sample and of cement and then converted to SO_4. Note that the method is for testing dry cement while the samples are concrete blockwork and mass concrete. The standard details the calculation for reporting the sulfate content by mass of sample. A further calculation was performed to report the sulfate by mass of cement in the concrete. This was performed following BS 1881-124:2015+A1:2021[29] and using the determined cement content. This calculation is not included in BS EN 196-2:2013.

4.15 Determination of sulfide – BS EN 196-2:2013

The sulfide content of the sample was determined in accordance with BS EN 196-2:2013, clause 4.4.5.[30] Note that the method is for testing dry cement while the samples tested comprised concrete. A conversion factor based on sample density (measured) and calculated aggregate content (1700 kg/m^3 from point count) was applied to the values to calculate the sulfide content of aggregates, assuming negligible sulfide content of the hardened cement.

4.16 BRE SD1 Suite D Brownfield Testing

Representative portions of concrete samples previously submitted for oxidisation/ expansion testing were submitted to Envirolab (an RSK Group company) for further BRE SD1:2005[31] Suite D brownfield testing consisting of pH (probe), water-soluble sulfate (colorimetry), acid-soluble sulfate (inductively coupled plasma, optical emission spectroscopy, ICP-OES) and total sulfur analyses (ICP-OES) using in-house standards.

Leachate samples from the bottom of containers used during the RICS Stage 3 testing were also sub-sampled and submitted for the same BRE SD1 Suite D brownfield testing for water samples. This comprised of pH (probe), water-soluble sulfate (colorimetry) and magnesium content (ICP-OES, when specified by results but not presented herein).

[28] BS EN 196-2:2013 Method of testing cement - Chemical analysis of cement, BSI, London

[29] BS 1881-124:2015+A1:2021, See [22]

[30] BS EN 196-2:2013, See [28]

[31] BRE Special Digest 1: 2005. Third Edition. Concrete in aggressive ground. BRE, Garston, UK. ISBN 1 86081 754 8

5 LABORATORY PROGRAM

Table 5-1 Laboratory program assignment – Phase 2 and 3 – Test Properties

Location	RSK Reference		CSA A23.1 P3 oxidisation	RICS - The Mundic Problem Stage 3 expansion testing	Petrographic thin section examination	SEM/EDX analysis,	XRF	XRD	Compressive strength	Density	BRE Suite D brownfield leachate	BRE Suite D brownfield concrete	Cement content	Total sulfur (Acid digestion)	Total sulfur (HTC)	Acid-soluble sulfate content	Water soluble sulfate content	Determination of sulfate	Determination of sulfide
7MV	20511/B2	E		X	X														
		F		X							X	X	X	X	X	X	X	X	X
		G		X					X	X									
		H		X	X	X	X	X											
	20511/B3	A	X						X	X									
		B	X		X	X	X	X							X				
		C	X									X	X	X		X	X	X	X
		D	X		X														
	20511/B4	E		X	X	X	X	X											
		F		X															
		G		X							X	X	X	X	X	X	X	X	X
		H		X					X	X									
	20511/B5	A	X		X	X													
		B	X		x		X	X							X				
		C	X									X	X	X		X	X	X	X
		D	X						X	X									
	20511/C4	E		X	X	X	X	X											
	20511/C5	B	X									X	X	X	X	X	X	X	X
		C	X		X	X	X	X											
	20511/C6	A		X					X	X	X	X	X	X	X	X	X	X	X
	20511/C7	E		X					X	X									
		F		X							X	X	X	X	X	X	X	X	X
	20511/C8	A	X		X	X	X	X							X				
		B	X		X	X	X	X							X				
	20511/C9	A		X	X	X	X	X							X				
		B		X															
		C	X						X	X									
		D	X									X	X	X	X	X	X	X	X

Table 5-1 Laboratory program assignment – Phase 2 and 3 – Test Properties Continued

Location	RSK Reference		CSA A23.1 P3 oxidisation	RICS - The Mundic Problem Stage 3 expansion testing	Petrographic thin section examination	SEM/EDX analysis,	XRF	XRD	Compressive strength	Density	BRE Suite D brownfield leachate	BRE Suite D brownfield concrete	Cement content	Total sulfur (Acid digestion)	Total sulfur (HTC)	Acid-soluble sulfate content	Water soluble sulfate content	Determination of sulfate	Determination of sulfide
21GD	20511/B14	A		X															
		B		X					X	X									
		C		X							X	X	X	X	X	X	X	X	X
		D		X	X	X	X	X											
	20511/B15	A	X						X	X									
		B	X									X	X	X		X	X	X	X
		C	X		X	X	X	X											
		D	X		x										X				
	20511/B16	A		X							X	X	X	X		X	X	X	X
		B		X															
		C		X					X	X					X				
		D		X	X	X	X	X											
	20511/B17	B	X		X														
		C	X		X	X	X	X							X				
		D	X						X	X									
		A	X									X	X	X	X	X	X	X	X
		E		X					X	X	X	X	X	X		X	X	X	X
	20511/C25	F		X	X	X	X	X											
	20511/C26	B	X						X	X		X	X	X		X	X	X	X
		C	X		X	X	X	X							X				
	20511/C27	E		X	X	X	X	X			X	X	X	X	X	X	X	X	X
	20511/C28	B	X		X	X	X	X				X	X	X	X	X	X	X	X

Table 5-1 Laboratory program assignment – Phase 2 and 3 – Test Properties Continued

Location	RSK Reference		CSA A23.1 P3 oxidisation	RICS - The Mundic Problem Stage 3 expansion testing	Petrographic thin section examination	SEM/EDX analysis,	XRF	XRD	Compressive strength	Density	BRE Suite D brownfield leachate	BRE Suite D brownfield concrete	Cement content	Total sulfur (Acid digestion)	Total sulfur (HTC)	Acid-soluble sulfate content	Water soluble sulfate content	Determination of sulfate	Determination of sulfide
28AW	20511/B8	A		X					X	X									
		B		X	X	X	X	X											
		C		X							X	X	X	X	X	X	X	X	X
		D		X															
	20511/B10	A	X		X														
		B	X																
		C	X									X	X	X	X	X	X	X	X
		D	X		X	X	X	X											
	20511/C18	E		X					X	X	X	X	X	X		X	X	X	X
		F		X	X	X	X	X											
	20511/C19	B	X		X	X	X	X											
	20511/C20	E		X	X	X	X	X			X				X				
	20511/C21	B	X		X	X	X	X							X				
	20954/B1	A		X	X	X	X	X											
		B		X								X	X	X	X	X	X	X	X
		C	X																
		D	X		X	X	X	X				X	X	X	X	X	X	X	X
	20954/C3	A	X										X	X	X	X	X	X	X
		B	X									X							
		C	X																
	20954/C4	A		X					X	X									
		B		X								X	X	X	X	X	X	X	X
	20954/C5	A		X					X	X		X	X	X	X	X	X	X	X
		B	X						X	X			X	X	X	X	X	X	X

Table 5-1 Laboratory program assignment – Phase 2 and 3 – Control Properties

Location	RSK Reference		CSA A23.1 P3 Oxidisation	RICS - The Mundic Problem Stage 3 Expansion Testing	Petrographic thin section examination	SEM/EDX analysis,	XRF	XRD	Compressive strength	Density	BRE Suite D Brownfield leachate	BRE Suite D Brownfield Concrete	Cement content	Total sulfur (Acid digestion)	Total sulfur (HTC)	Acid-soluble sulfate content	Water soluble sulfate content	Determination of sulfate	Determination of sulfide
C	20511/B6	E		X							X	X	X	X	X	X	X	X	X
		F		X					X	X									
		G		X	X	X	X	X											
		H	X		X	X	X	X											
		I	X						X	X									
		J	X									X	X	X	X	X	X	X	X
	20511/B7	E		X							X	X	X	X		X	X	X	X
		F		X					X	X									
		G		X	X	X	X	X											
		H	X		X	X	X	X						X					
		I	X		X														
		J	X						X	X		X	X	X		X	X	X	X

As part of the investigation (Phase 2), all sub-samples were tested (see **Table 5-1**) either in accordance with RICS, The Mundic Problem, Stage 3 expansion test (see **4.1**) or the adapted CSA A23.1 P3 Oxidisation test (see **4.2**). Upon completion of the Phase 2 durability testing, the samples were assigned a further testing programme of petrographic, instrumental, chemical and physical testing (Phase 3, see **Table 5-1**). The testing suite was designed to investigate the potential for further expansion and deterioration, and the potential factors behind any change in sample integrity and differences in relative performance.

6 RESULTS

To better illustrate the results, condensed summaries of the Phase 2 durability tests are produced in the following section. Full details of the results can be found in **Appendix C**. Data for foundations can be found in more detail in RSK report 1283831-03. All changes in results compare Phase 3 to Phase1 results.

6.1 RICS, The Mundic Problem, Stage 3 expansion test

The data presented for the RICS, The Mundic Problem, Stage 3 expansion test results is presented below in a series of expansion and mass change graphical representations. Further details and tabulated numerical data can be found in **Appendix C**.

Figure 6-1 RICS The Mundic Problem Stage 3, inner leaf, mean expansion

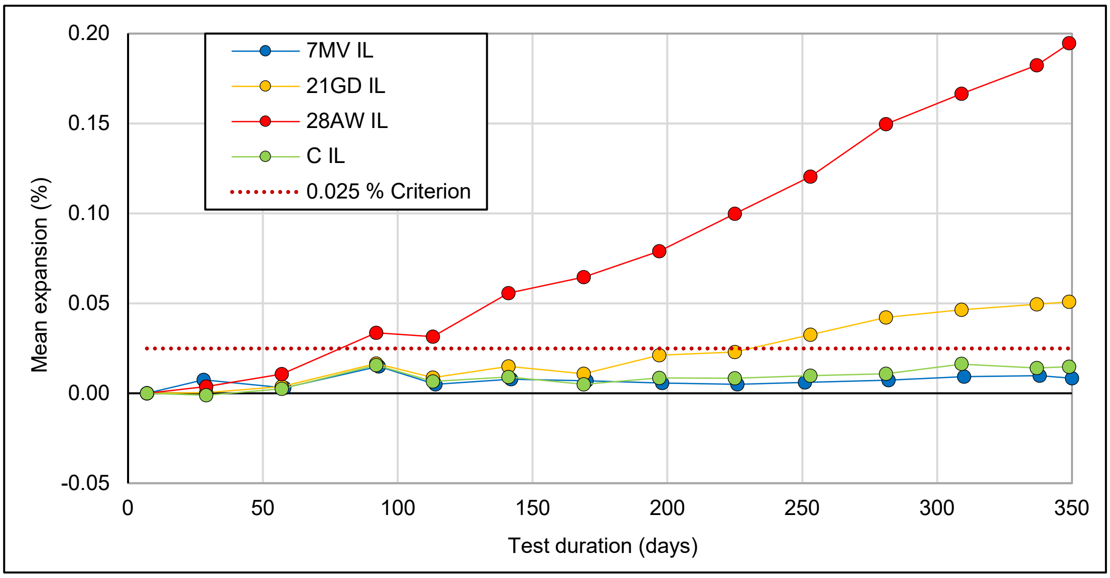
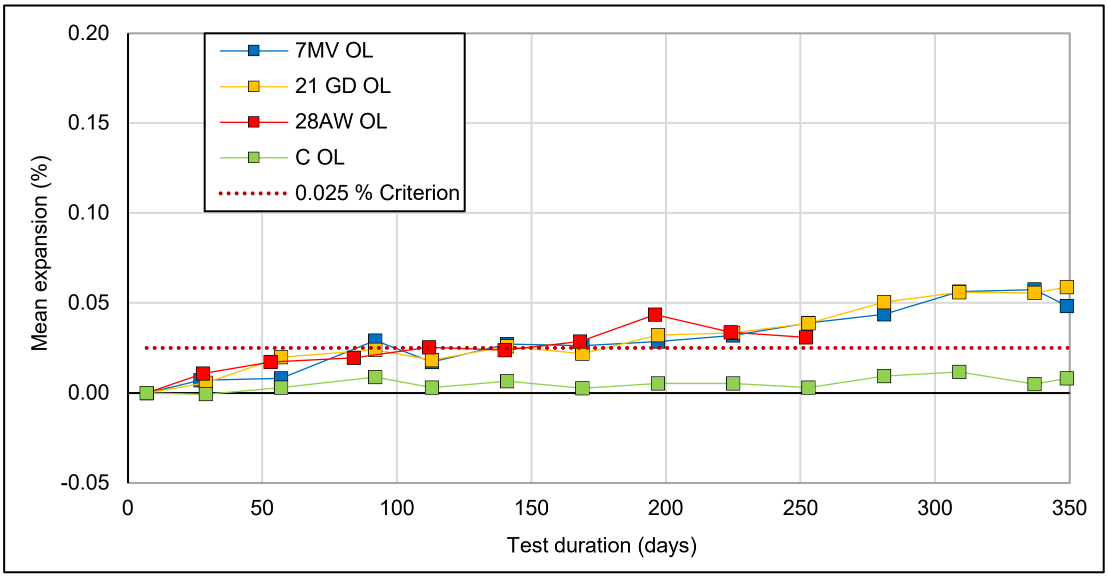


Figure 6-2 RICS The Mundic Problem Stage 3, outer leaf, mean expansion



Note that 28AW outer leaf was only tested for 250 days.

Figure 6-3 RICS The Mundic Problem Stage 3, rising wall, mean expansion

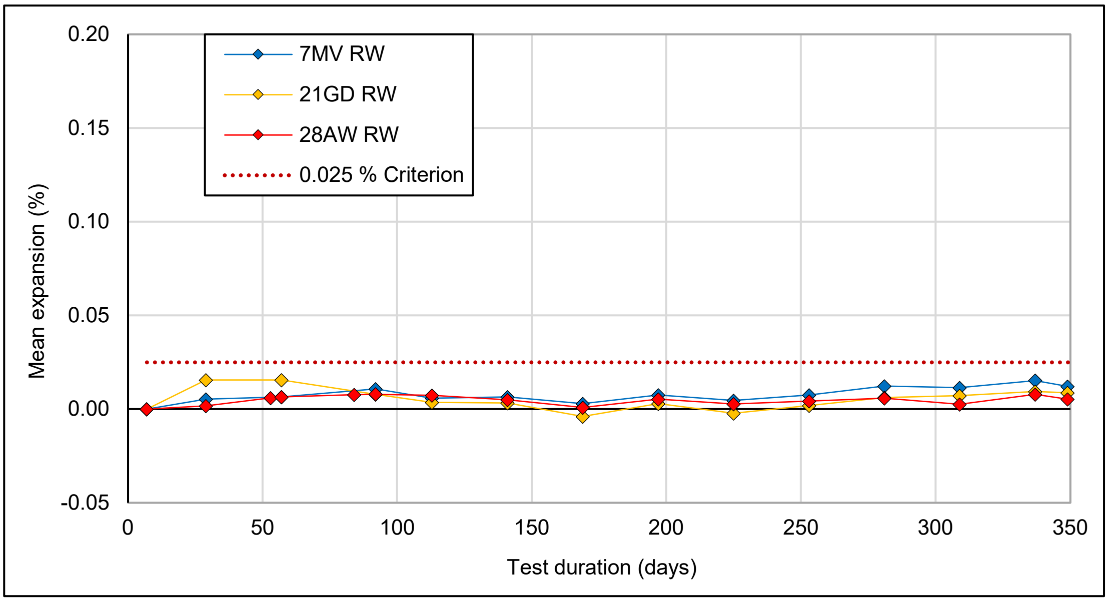


Figure 6-4 RICS The Mundic Problem Stage 3, foundation, mean expansion

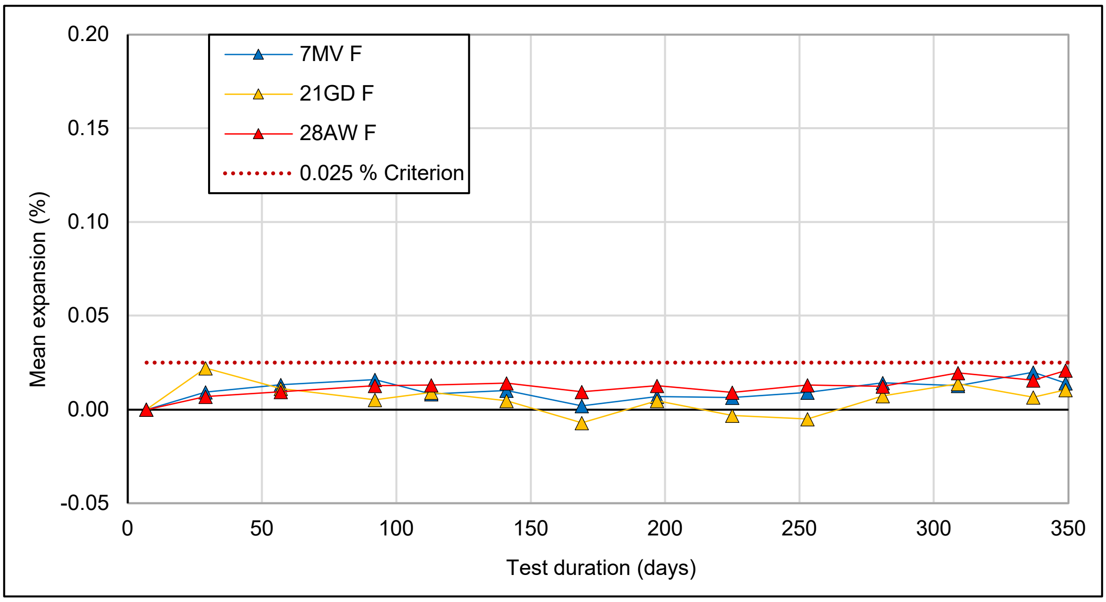


Figure 6-5 RICS The Mundic Problem Stage 3, Test property building element – mean expansion

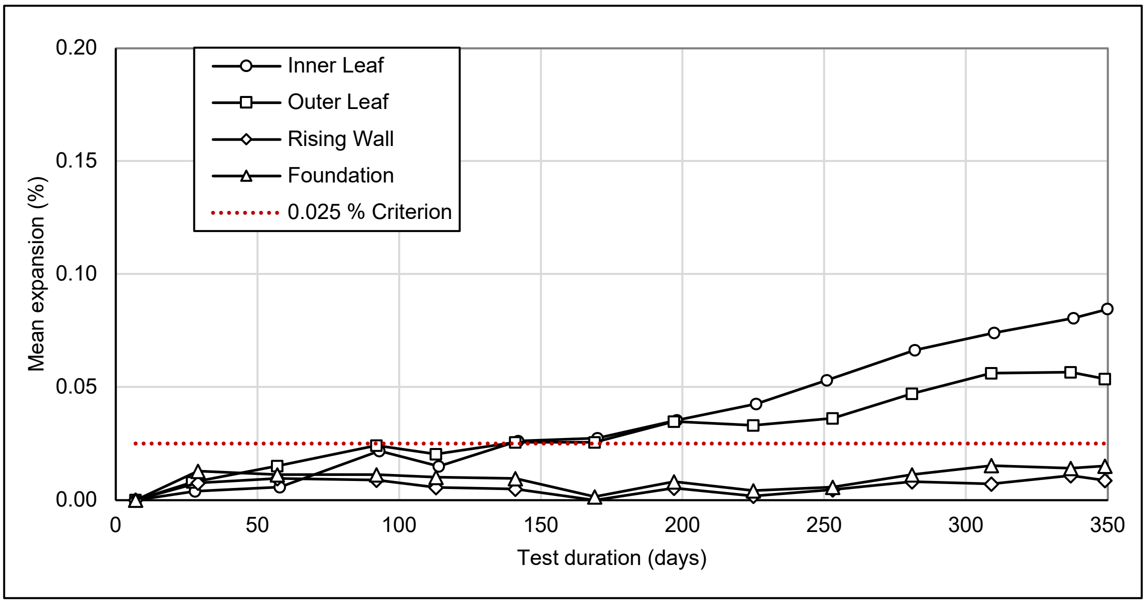
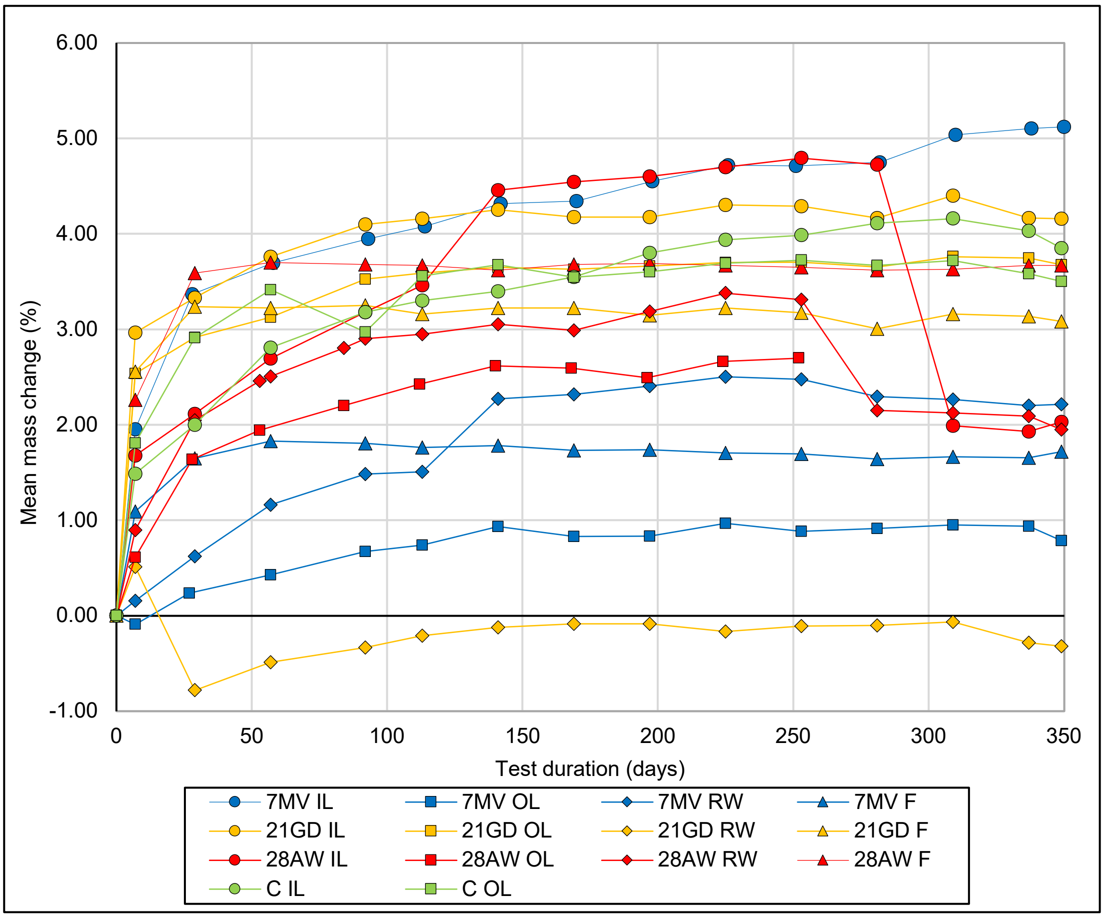


Figure 6-6 RICS The Mundic Problem Stage 3, Mean mass change

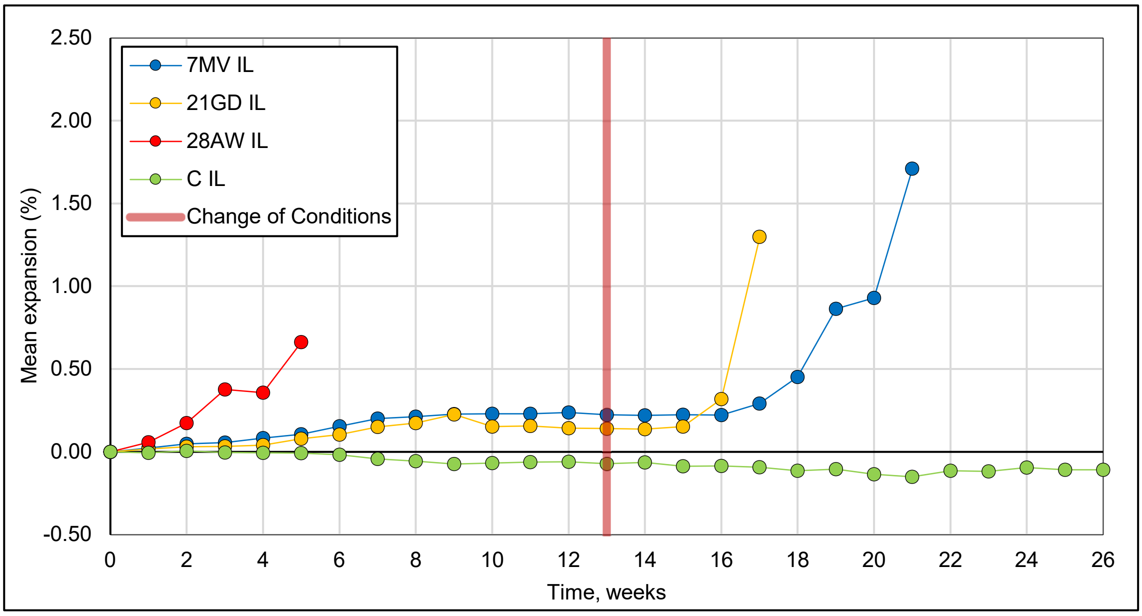


Note that mass reductions tend to represent loss of materials from samples rather than moisture loss. Note that 28AW OL was only tested for 250 days.

6.2 CSA A23.1 P3 adapted oxidisation test

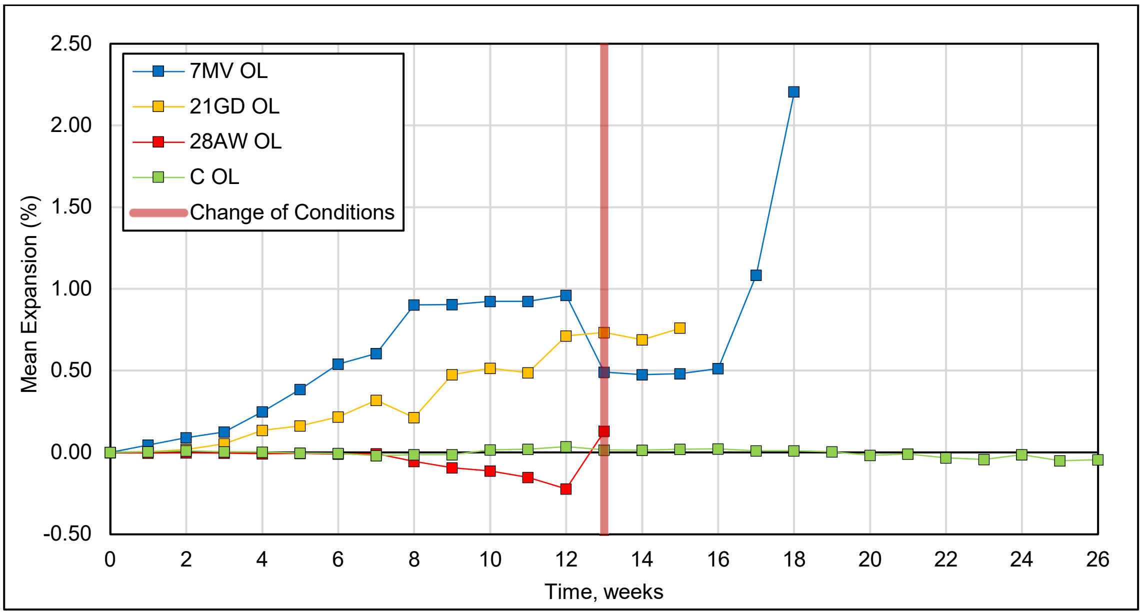
The data presented for the CSA A23.1 P3 adapted oxidisation test results is presented in a series of expansion, mass change and date-to-failure graphical representations. Further details and tabulated numerical data can be found in **Appendix C**. Note that no control property RW (absent) and F (inadequate samples) tests were conducted.

Figure 6-7 CSA A23.1 P3 Oxidisation test, Expansion – Inner Leaf



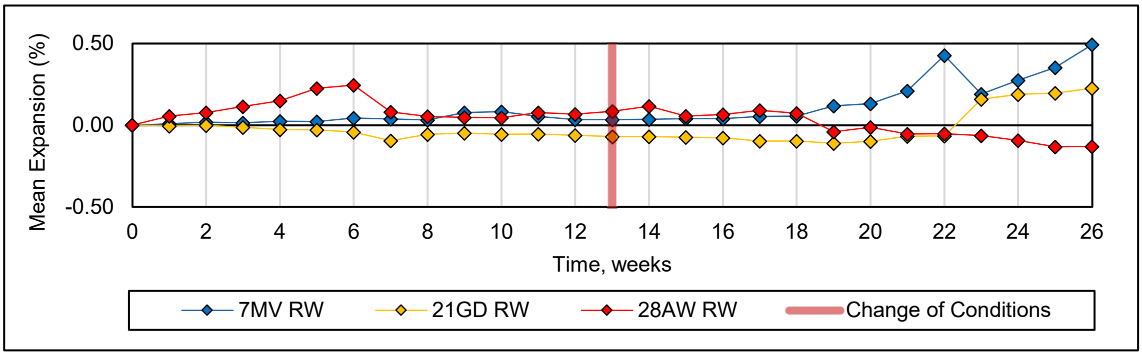
Note that values presented are means of remaining samples on test for each element. Therefore, where a set of readings ends before 26 weeks, this represents the failure of the last sub-sample. Where a sub-sample set shrinks or decreases its rate of expansion rapidly, this likely represents failure of a sub-sample that had been expanding and the mean reverting to the remaining sub-samples.

Figure 6-8 CSA A23.1 P3 Oxidisation test, Expansion – Outer Leaf



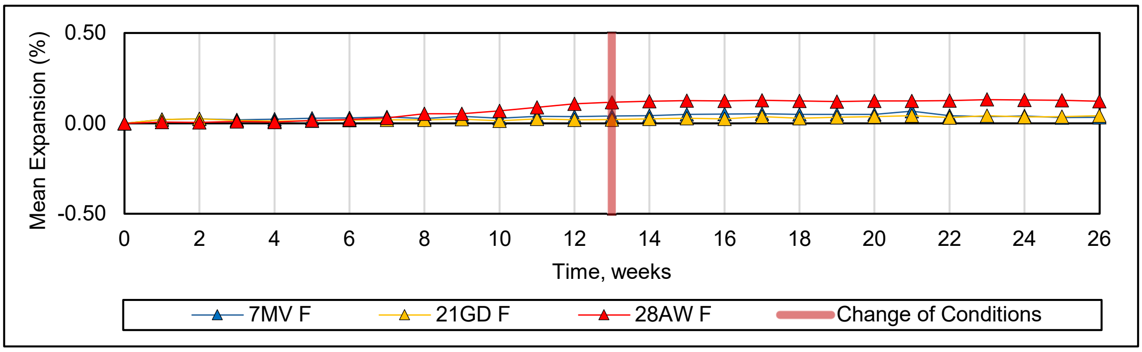
Note that values presented are means of remaining samples on test for each outer leaf. Therefore, where a set of readings ends before 26 weeks, this represents the failure of the last sub-sample. Where a sub-sample set shrinks or decreases its rate of expansion rapidly, this likely represents failure of a sub-sample that had been expanding and the mean reverting to the remaining sub-samples.

Figure 6-9 CSA A23.1 P3 Oxidisation test, Expansion – Rising Wall



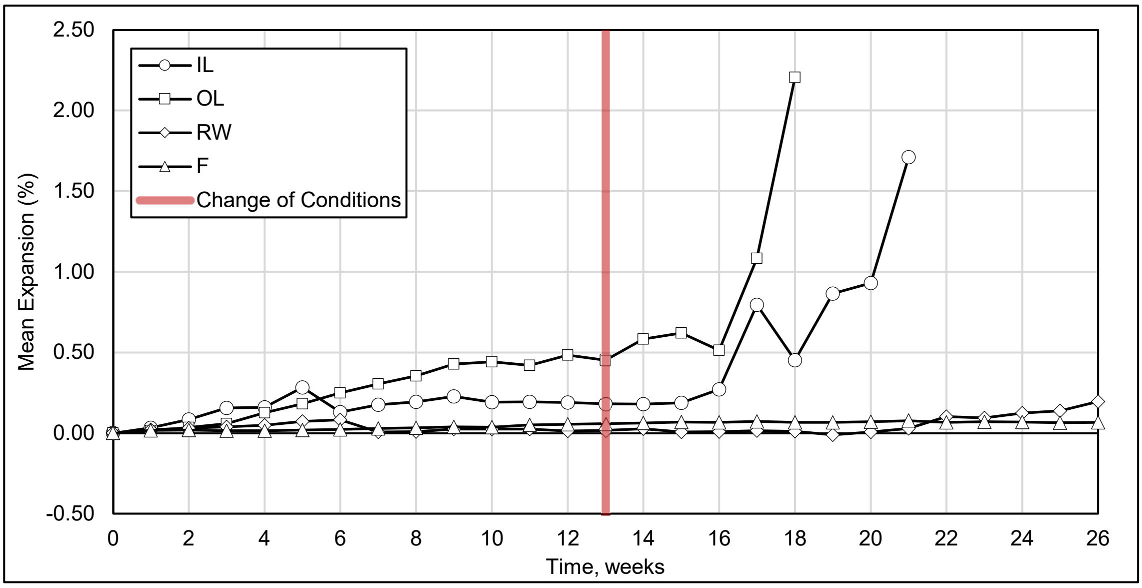
Note that values presented are means of remaining samples on test for each rising wall. Where a sub-sample set shrinks or decreases its rate of expansion rapidly, this likely represents failure of a sub-sample that had been expanding and the mean reverting to the remaining sub-samples.

Figure 6-10 CSA A23.1 P3 Oxidisation test, Expansion – Foundation



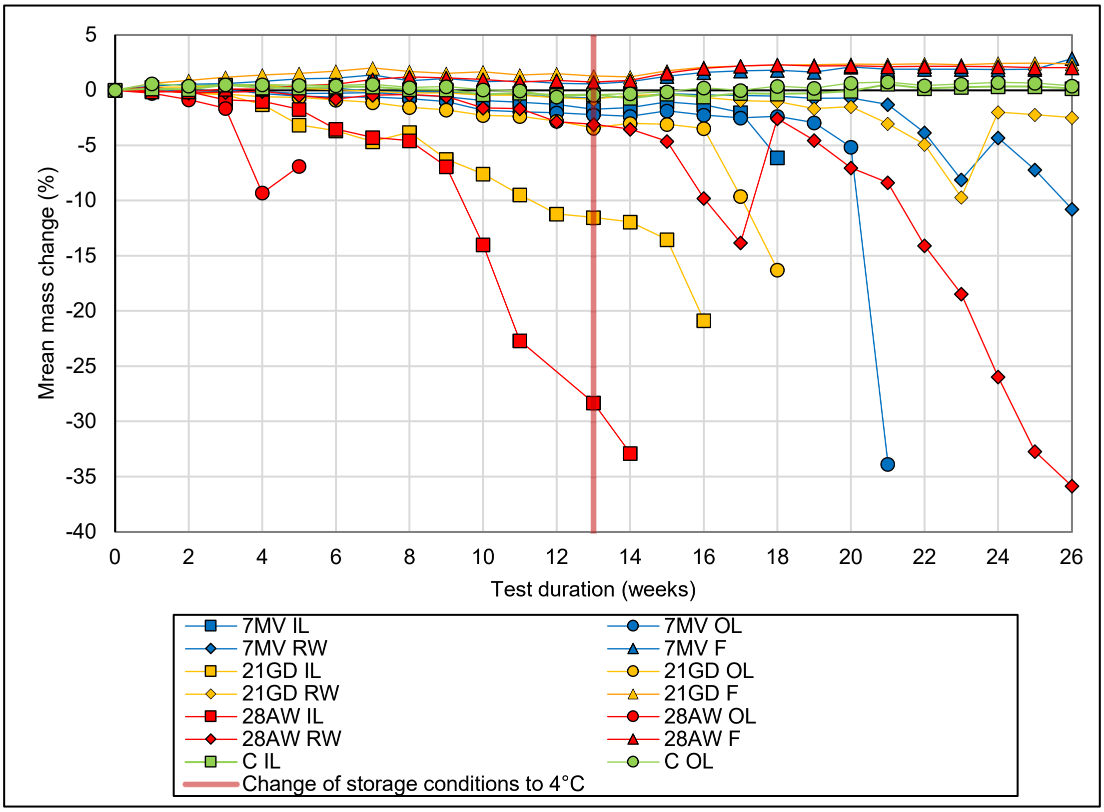
Note that values presented are means of remaining samples on test for each foundation. Where a sub-sample set shrinks or decreases its rate of expansion rapidly, this likely represents failure of a sub-sample that had been expanding and the mean reverting to the remaining sub-samples.

Figure 6-11 CSA A23.1 P3 Oxidisation test, Test property building element – Mean expansion



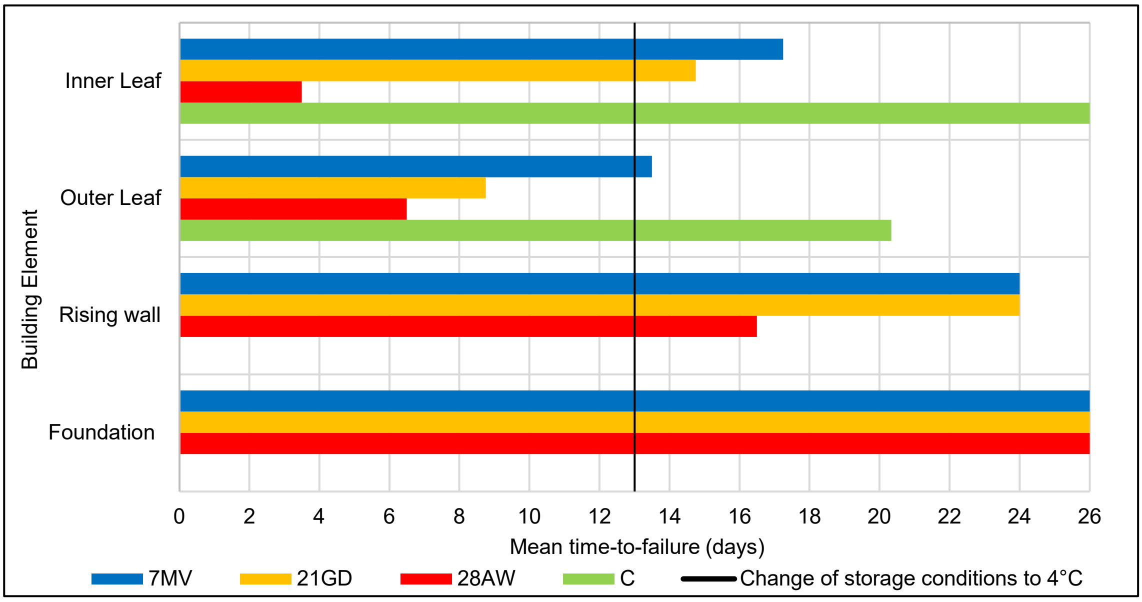
Note that values presented are means of remaining samples on test for each test property element. Therefore, where a set of readings ends before 26 weeks, this represents the failure of the last sub-sample. Where a sample set shrinks or decreases its rate of expansion rapidly, this likely represents failure of a sub-sample that had been expanding and the mean reverting to the remaining sub-samples.

Figure 6-12 CSA A23.1 P3 Oxidisation test, Mean Mass Change



Note that values presented are means of remaining samples on test for each element. Therefore, where a set of readings ends, this represents the failure of the last sub-sample or the end of the test at 26 weeks. Where a series increases in mass sharply, this represents failure of a sub-sample that had been losing mass and the mean reverting to the remaining sub-samples.

Figure 6-13 CSA A23.1 P3 Oxidisation test – Mean time-to-failure (core)



Note that 7MV F excludes core taken off test at 13 weeks for comparative purposes

6.3 Optical Microscopy

6.3.1 Sulfides

The following section consists of summaries of the petrographic data collected by OM.

Table 6-1 OM sulfide abundance

Property		7MV	21GD	28AW	C (Control)
Aggregate		PHY			SST
Pyrrhotite	Aggregate	XXX			X
	Matrix	XXX			X
Pyrite	Aggregate	XX			XX
	Matrix	XX			X
Chalcopyrite	Aggregate	X			-
	Matrix	X			-

X denotes relative abundances – XXX high abundance, XX moderate abundance, X lower abundance, - absent. Most common sulfides included only, excluded pentlandite and other rare trace minerals. Taken from RSK report 1283831-01

6.3.2 Features of Interest

Table 6-2 OM features of interest – Inner Leaf

Element	Inner Leaf							
Property	7MV		21GD		28AW		C	
Phase 2 Durability Testing	RICS - Stage 3	CSA P3 adapted	RICS - Stage 3	CSA P3 adapted	RICS - Stage 3	CSA P3 adapted	RICS - Stage 3	CSA P3 adapted
Exposure until failure	350 Days	21-22 Weeks	350 Days	26 Weeks	350 Days	4-5 Weeks	350 Days	26 Weeks
Exposure to test conditions	350 Days	26 Weeks	350 Days	26 Weeks	350 Days	4-5 Weeks	350 Days	26 Weeks
Sulfide oxidation CA	XX	XXX	XX	XXX	XXX	XXX	XX	XX
Sulfide oxidation FA	XX	XXX	XX	XXX	XXX	XXX	XX	XX
In situ sulfide oxidisation	XXX	XXX	XXX	XXX	XXX	XXX	XX	XX
Iron oxides/hydroxides	X	XX	XX	XX	XXX	XXX	X	XX
Secondary Ettringite	XX	X	X^	-†	XX^	XX^	-	-
Secondary Gypsum	-	-	X^	-†	XX	XX^	-	-
Thaumasite	-	-	-	-†	-	XX^	-	-
Weak matrix	XXX	XXX	XXX	XXX	XXX	XXX	X	-
Cement alteration	XXX	XXX	XXX	XXX	XX	XXX	-^	-^
ISA matrix conversion	X	X	X	XXX	XX	XX	-^	-^
Degree of ISA	X	XXX	X	XXX	X	XX	-	-
Secondary Portlandite	-	-	-	-	-	-	-	-
Secondary Calcite	-	-	-	-	-	-	-	-
Leaching	X	X	-	-	-	-	-	-
Chloride deposits	-	-	-	-	-	-	-	-
Secondary carbonation	X	-	-	-	-^	-	-	-
Cracking	XX	XXX	X	XXX	XX	XXX	X	-
Indicative strength	XX	X	XX	X	XX	XX	XXX	XX

X denotes relative abundances – XXX high abundance, XX moderate abundance, X lower abundance, - absent. Other deposits included secondary portlandite and other deposits, ^ = Possible/unconfirmed, † = Insufficient sample remaining for confidence.

Table 6-3 OM features of interest – Outer Leaf

Element	Outer Leaf									
Property	7MV			21GD			28AW		C	
Phase 2 Durability Testing	RICS - Stage 3	CSA P3 adapted	CSA P3 adapted	RICS - Stage 3	CSA P3 adapted	CSA P3 adapted	RICS - Stage 3	CSA P3 adapted	RICS - Stage 3	CSA P3 adapted
Exposure until failure	350 Days	13 Weeks	18 Weeks	350 Days	5 Weeks	26 Weeks	250 Days	26 Weeks	350 Days	10 & 26 Weeks
Exposure to test conditions	350 Days	13 Weeks	26 Weeks	350 Days	5 Weeks	26 Weeks	250 Days	26 Weeks	350 Days	10 & 26 Weeks
Sulfide oxidation CA	XX	XXX	XXX	XX	XXX	XXX	XX	XXX	XX	XX
Sulfide oxidation FA	XX	XXX	XXX	XX	XXX	XXX	XXX	XXX	XX	XXX
In-situ sulfide oxidisation	XXX	XXX	XXX	XXX	XXX	XXX	XX	XXX	XX	XXX
Iron oxides/hydroxides	XX	XX	XX	XX	XX	XX	X	XX	XX	XX
Secondary Ettringite	X	XX	XX	XX	X	X	XX	XX^	-^	-
Secondary Gypsum	X	-	-	-	X	X	X	XX^	-	-
Thaumasite	-	-	-	XX	-	-	XX	-	-	-
Weak matrix	XX	XXX	XXX	XX	XXX	XXX	XXX	XXX	-	-
Cement alteration	XX	XXX	XXX	XX	XXX	XXX	XXX	XXX	-	-
ISA matrix conversion	X	X	X	XX	X	X	X	XXX	-^	-^
Degree of ISA	X	X	XXX	XX	XX	XXX	XX	XXX	-^	-
Secondary Portlandite	-	-	-	-	-	-	-	-	-	-
Secondary Calcite	-	-	-	-	X	-	-	-	-^	X
Leaching	X	X	X	-	X	-	-	-	X	X
Chloride deposits	-	-	-	-	X^	X^	-	-	-	-
Secondary carbonation	-	-	-	-	-	-	-	-	-	-
Cracking	XX	XXX	XXX	X	XXX	XXX	XX	XXX	X	X
Indicative strength	XX	X	X	XX	X	X	XX	X	XXX	XX

X denotes relative abundances – XXX high abundance, XX moderate abundance, X lower abundance, - absent. Other deposits included secondary portlandite and other deposits, ^ = Possible/unconfirmed.

Table 6-4 OM features of interest – Rising Wall

Element	Rising Wall					
Property	7MV		21GD		28AW	
Phase 2 Durability Testing	RICS - Stage 3	CSA P3 adapted	RICS - Stage 3	CSA P3 adapted	RICS - Stage 3	CSA P3 adapted
Exposure until failure	350 Days	26 Weeks	350 Days	26 Weeks	350 Days	6 Weeks
Exposure to test conditions	350 Days	26 Weeks	350 Days	26 Weeks	350 Days	6 Weeks
Sulfide oxidation CA	XX	XXX	XX	XX	XX	XXX
Sulfide oxidation FA	XXX	XXX	XX	XX	XX	XXX
In-situ sulfide oxidisation	XXX	XXX	XXX	XXX	XX	XX
Iron oxides/hydroxides	XX	XX	X	XX	XX	XXX
Secondary Ettringite	XXX	XX	XX	XXX	X	XXX
Secondary Gypsum	XX	-	X	-	-	XXX
Thaumasite	-	-	-	-	-	XXX^
Weak matrix	X	XX	XXX	XXX	XX	XXX
Cement alteration	XX	XX	XXX	XXX	XX	XXX
ISA matrix conversion	XX	XX	XX	XX	XX	XXX
Degree of ISA	XX	XXX	X	XXX	X	XXX
Secondary Portlandite	XX	-	-	-	-	-
Secondary Calcite	XX	XX	X	-	X	XXX
Leaching	XX	XX	X	-	XX	XXX
Chloride deposits	-	X^	-	-	-	-
Secondary carbonation	-	-	-	-	-	XXX
Cracking	X	XXX	X	XXX	X	XXX
Indicative strength	XX	X	XXX	X	XX	X

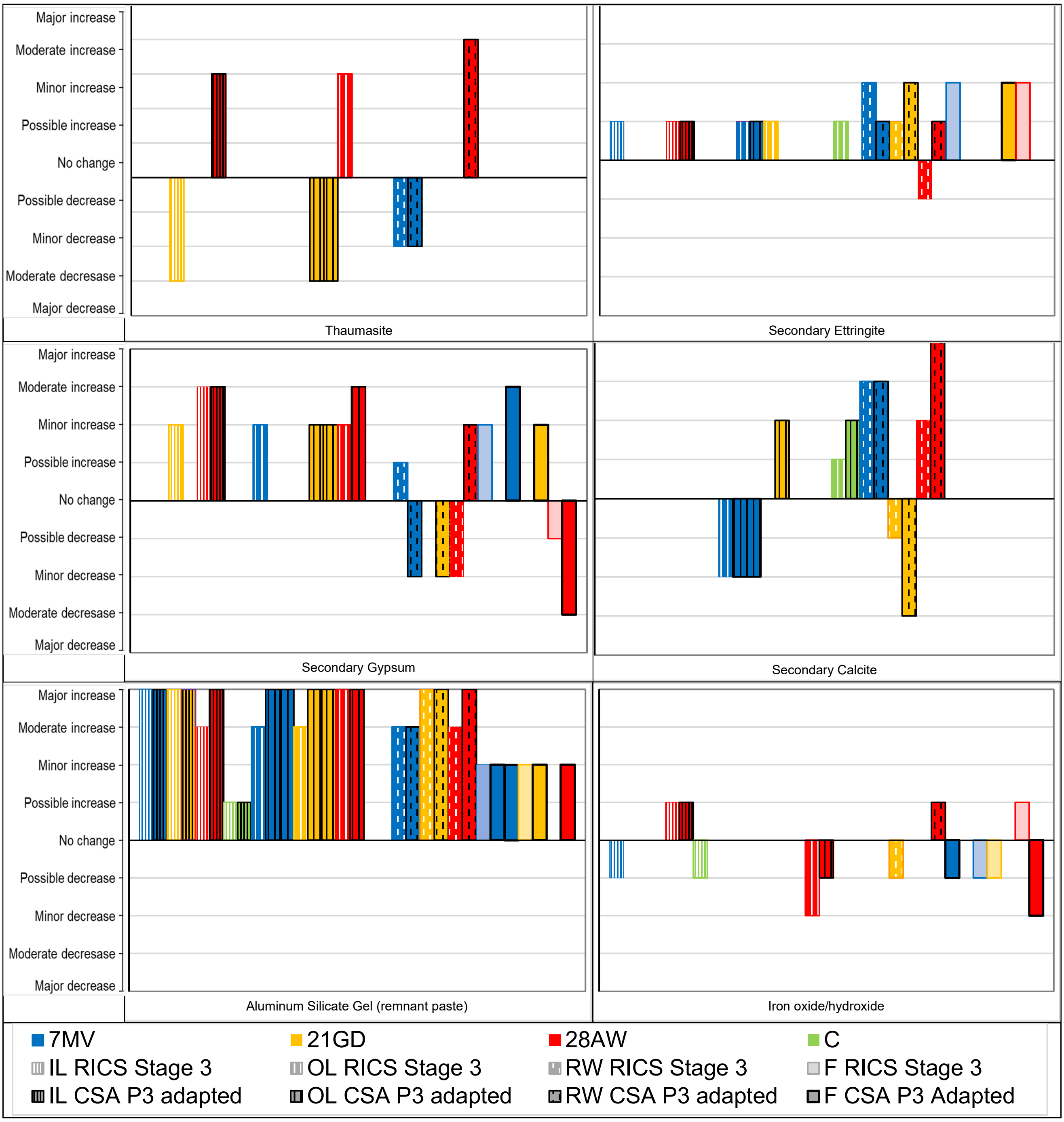
X denotes relative abundances – XXX high abundance, XX moderate abundance, X lower abundance, - absent. Other deposits included secondary portlandite and other deposits, ^ = Possible/unconfirmed.

Table 6-5 OM features of interest – Foundation

Element	Foundation						
Property	7MV			21GD		28AW	
Phase 2 Durability Testing	CSA P3 adapted	CSA P3 adapted	RICS - Stage 3	RICS - Stage 3	CSA P3 adapted	RICS - Stage 3	CSA P3 adapted
Exposure until failure	14 Weeks	26 Weeks	350 Days	350 Days	26 Weeks	350 Days	26 Weeks
Exposure to test conditions	26 Weeks	26 Weeks	350 Days	350 Days	26 Weeks	350 Days	26 Weeks
Sulfide oxidation CA	X	XX	XX	X	XX	X	X
Sulfide oxidation FA	X	XX	X	X	XX	XX	XX
In-situ sulfide oxidisation	-	-	-	X	XX	-	-
Iron oxides/hydroxides	X	XX	X	X	XX	XX	-
Secondary Ettringite	-	-	X	-	X	X	-
Secondary Gypsum	-	XX	X	-	X^	X	-
Thaumasite	-	-	-	-	-	-	-
Weak matrix	X	XX	X	XX	XX	-	-
Cement alteration	X	X	X	X	X	-	X
ISA matrix conversion	-^	-^	-^	-^	-^	-^	-^
Degree of ISA	<X^C	<X^C	<X	<X	<X	<X	<X^C
Secondary Portlandite	X	X	XX	X	X	XXX	X
Secondary Calcite	-	-	-	-	-	-	-
Leaching	X	X	X	X	X	X	X
Chloride deposits	-	XX	-	-	-	-	XXX
Secondary carbonation	-	-	X	X	X	-	-
Cracking	X	X	-	-	X	X	X
Indicative strength	XX	XX	XX	XX	XX	XX	XX

X denotes relative abundances – XXX high abundance, XX moderate abundance, X lower abundance, - absent. Other deposits included secondary portlandite and other deposits, ^ = Possible/unconfirmed, C = Chlorine affected

Table 6-6 ΔOM selected observations – Phase1-Phase 3



6.3.3 Post-durability test sample condition

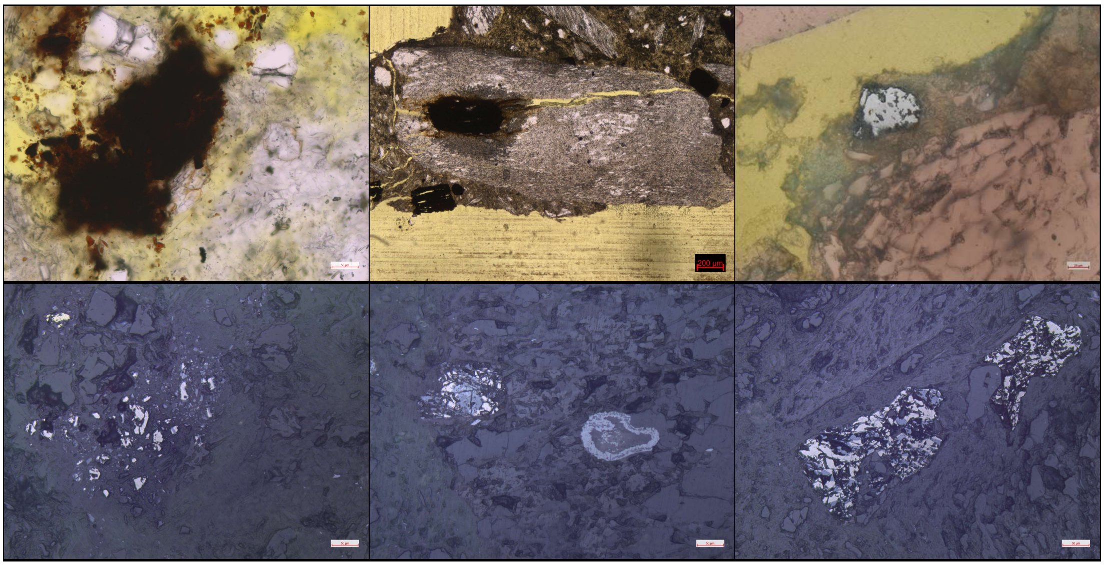
Figure 6-14 Photographs of post-durability test sample condition

Views of the post-test visual condition progressing in severity of deterioration from left (Control property) to right increasingly poor condition of test property sub-samples. Top row RICS Stage 3 and bottom row CSA P3 Adapted.

6.3.4 Photomicrographs

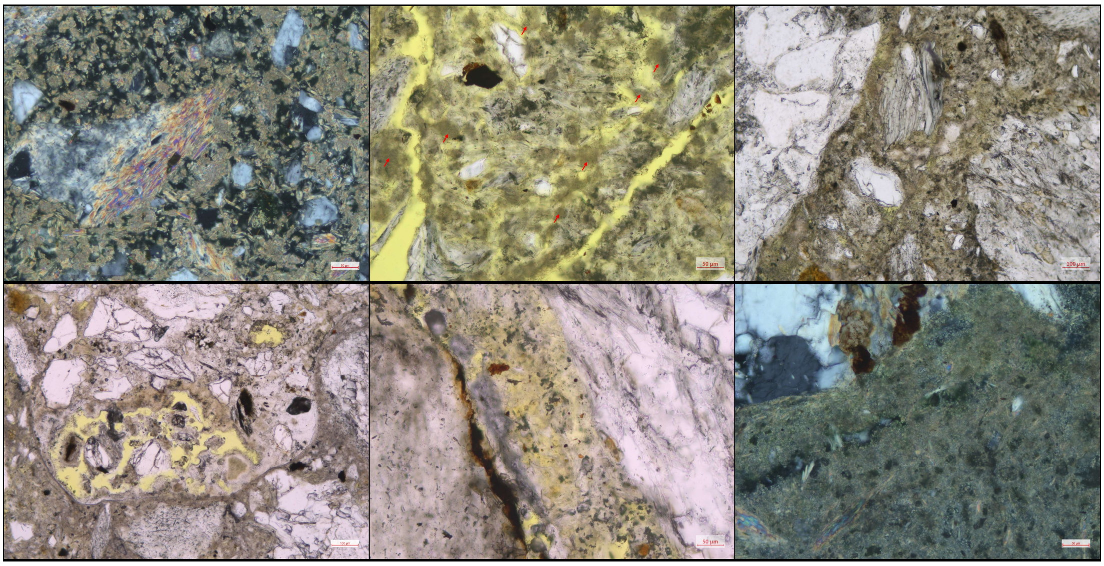
The photomicrographs presented illustrate the features of interest given in the tables above (**Table 6-2**, **Table 6-3**, **Table 6-4** and **Table 6-5**). Further details can be found in **Appendix C.**

Figure 6-15 Photomicrographs – Sulfide oxidisation



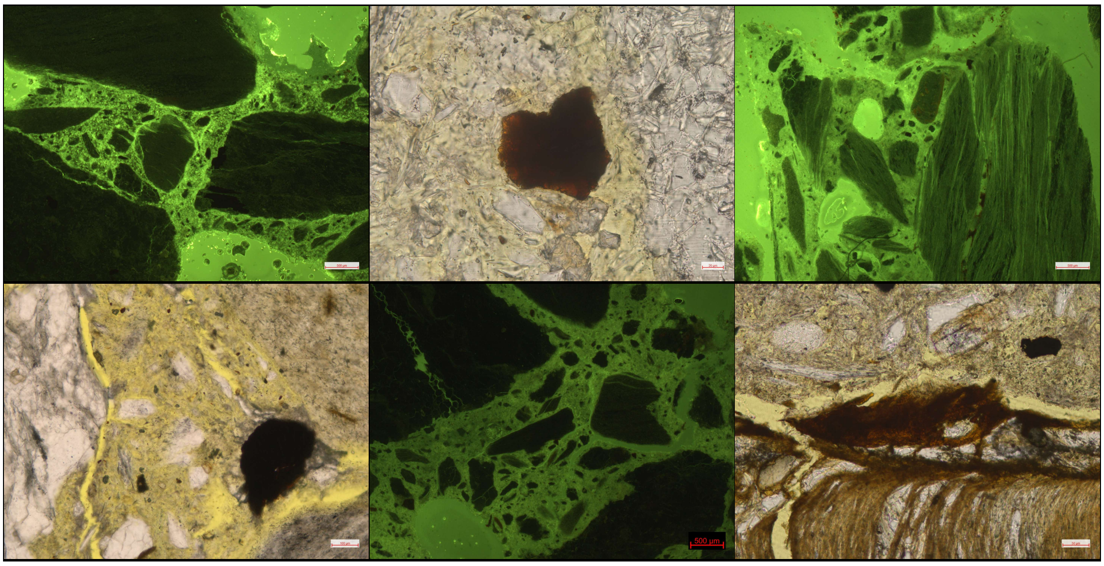
Top and bottom Left: 28AW, OL, CSA P3 adapted, PPL and RL, Near complete oxidised pyrrhotite with surrounding lack of cement matrix. Top middle: 28AW, IL, CSA P3 adapted, PPL, oxidised aggregate set pyrrhotite splitting phyllite particle apart. Lower middle: 28AW, RW, CSA P3 adapted, RL, near completely oxidised sulfides striped (left) and surface (right). Top right: C, OL, CSA P3 adapted, PPL, trace surface sulfide oxidisation. Lower right: 7MV, OL, RICS Stage 3, RL, moderate striped sulfide oxidisation

Figure 6-16 Photomicrographs – Secondary sulfates replacing the cement matrix



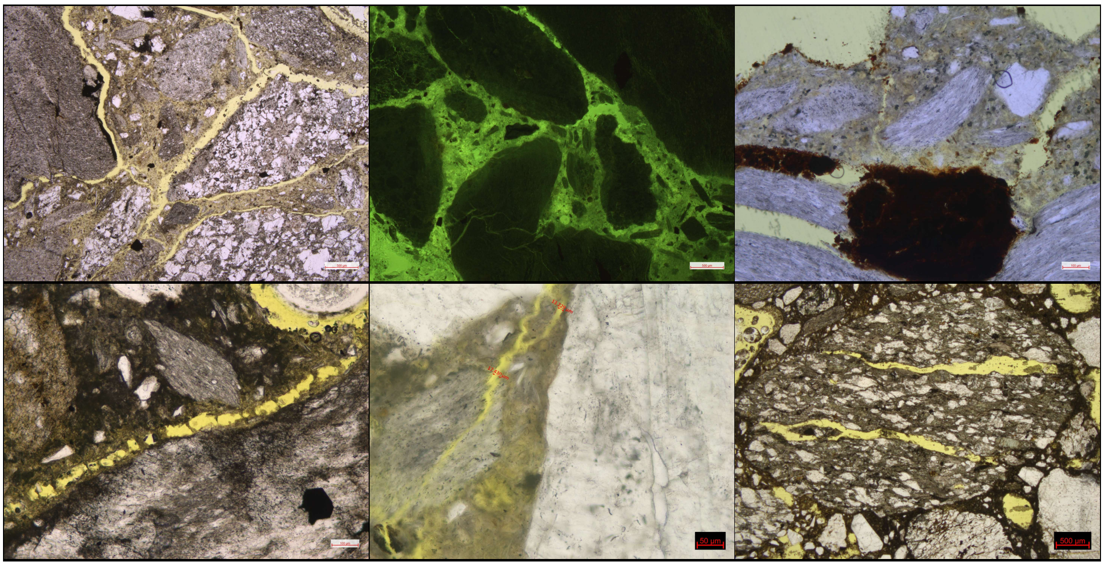
Top left: 7MV, IL, RICS Stage 3, CPL, Secondary ettringite replacing cement matrix. Bottom Left: 21GD, OL, RICS Stage 3, PPL, thaumasite replacing cement matrix at the interface with the render. Top middle: 21GD, RW, CSA P3 adapted, PPL, Brown ettringite (red arrows) replacing most of the cement matrix in a highly deteriorated sample. Bottom middle: 28AW, RW, CSA P3 adapted, PPL, Gypsum (white, centre) infilling cracking and replacing the cement matrix. Top right: 7MV, RW, RICS Stage 3, PPL, ettringite and gypsum replacing the cement matrix (white patches). Bottom right: 7MV, F, CSA P3 adapted, CPL, Secondary gypsum infilling a crack and replacing the cement matrix.

Figure 6-17 Photomicrographs – Weak cement matrix



Top left: 21GD, RW, RICS Stage 3, RF, Areas of very high microporosity weak cement matrix between dark aggregate particles. Top middle: 7MV, OL, CSA P3 adapted, PPL, Weak matrix surrounding oxidised pyrrhotite. Top right: 28AW, OL, CSA P3 adapted, RF, weak matrix in a fragmented sample. Bottom left: 28AW, RW, CSA P3 adapted, PPL, weak matrix around slightly oxidised pyrrhotite with crack. Bottom middle: 28AW, IL, CSA P3 adapted, RF, weak matrix with high microporosity. Bottom right: 7MV, IL, RICS Stage 3, PPL, Oxidised sulfides in phyllite with associated cracking and a very weak cement matrix.

Figure 6-18 Photomicrographs – Cracking



Top left: 28AW, RW, CSA P3 adapted, PPL, extensive cracking through the cement matrix and around aggregate particles. Top middle: 21GD, IL, CSA P3 adapted, RF, Cracking in cement matrix around aggregate particles. Top right: 7MV, IL, CSA P3 adapted, PPL, Oxidised pyrrhotite splitting phyllite and the surrounding cement matrix. Bottom left: 7MV, RW, CSA P3 adapted, PPL, cracking partially infilled with ettringite. Bottom middle: 28AW, IL, RICS Stage 3, PPL, cracking through fine aggregate and the cement matrix. C, IL, CSA P3 adapted, PPL, the fracturing of a coarse aggregate particle does not run into the cement matrix (pre-existing).

6.4 SEM/EDX

Table 6-7 SEM/EDX features of interest – Inner Leaf

Element	Inner Leaf							
Location	7MV		21GD		28AW		C	
Phase 2 Durability Testing	RICS - Stage 3	CSA P3 adapted	RICS - Stage 3	CSA P3 adapted	RICS - Stage 3	CSA P3 adapted	RICS - Stage 3	CSA P3 adapted
Exposure until failure	350 Days	21 Weeks	350 Days	26 Weeks	350 Days	4 Weeks	350 Days	26 Weeks
Exposure to test conditions	350 Days	26 Weeks	350 Days	26 Weeks	350 Days	4 Weeks	350 Days	26 Weeks
In situ oxidisation - degree	X	XXX	X	XXX	XX	XXX	N/a	X
In situ oxidisation - frequency	XX	XXX	XX	XXX	XX	XXX	N/a	X
Complete sulfide oxidisation	X	XX	X	XX	X	XX	N/a	-
Sulfide, surface oxidisation	X	XXX	X	XX	X	X	N/a	-
Striped oxidisation of pyrrhotite	X	XXX	X	XX	XX	XXX	N/a	-
Cracking (assoc. oxidised sulfides)	X	XXX	-	XXX	XX	XXX	-	X
Calcium depletion of cement matrix	XX	XXX	XX	XX	XX	XX	X	XX
Remnant aluminium oxide matrix		XX	XX	XX	XX	XX	X	X
Thaumasite (location)	-	X	-	-	-	-	-	-
Secondary ettringite	X^	-	-	-	-	-	-	-
Secondary gypsum	XX	-	XX	-	-	-	-	-
ISA matrix conversion	XX	XXX	X	XXX^	X	XXX	-	X^
Secondary calcite	X	XXX	X	XX	X	X	X	X
Leaching	X	-	X	X	X	X	X	X
Sodium chloride	-	-	-	-	-	-	-	X
Sodium sulfate	-	-	-	-	-	X	-	-

X denotes relative abundances – XXX high abundance, XX moderate abundance, X lower abundance, - absent. Other deposits included secondary portlandite and other deposits, ^ = Possible/unconfirmed.

Table 6-8 SEM/EDX features of interest – Outer Leaf

Element	Outer Leaf							
Location	7MV		21GD		28AW		C	
Phase 2 Durability Testing	RICS - Stage 3	CSA P3 adapted	RICS - Stage 3	CSA P3 adapted	RICS - Stage 3	CSA P3 adapted	RICS - Stage 3	CSA P3 adapted
Exposure until failure	350 Days	13 Weeks	350 Days	26 Weeks	350 Days	26 Weeks	350 Days	26 Weeks
Exposure to test conditions	350 Days	13 Weeks	350 Days	26 Weeks	350 Days	26 Weeks	350 Days	26 Weeks
In situ oxidisation - degree	X	XXX	XX	XXX	XXX	XXX	X	X
In situ oxidisation - frequency	XX	XXX	XXX	XXX	XX	XXX	X	X
Complete sulfide oxidisation	X	XX	XX	XX	XX	XX	-	-
Sulfide, surface oxidisation	XX	XXX	X	XX	XX	X	X	X
Striped oxidisation of pyrrhotite	XX	XXX	XX	XXX	XX	XXX	X	-
Cracking (assoc. oxidised sulfides)	-	XXX	X	XXX	X	XXX	-	-
Calcium depletion of cement matrix	XX	XXX	XXX	XXX	XXX	XX	X	X
Remnant aluminium oxide matrix	X	XX	XX	XX	XX	XX	X	XX
Thaumasite	-	XX	X^	XX	XX	wm	-	-
Secondary ettringite	-	-	XX	-	XX	wm	-	-
Secondary gypsum	XX	-	XX	-	X	wm	X	-
ISA matrix conversion	XX	XXX	XXX	XXX	XX	XXX	-	-
Secondary calcite	XX	XX	XX	X	XX	XX	X	XX
Leaching	X	X	XX	X	XX	X	X	XX
Sodium Chloride	-	-	-	-	XXX	X	-	-

X denotes relative abundances – XXX high abundance, XX moderate abundance, X lower abundance, - absent. Other deposits included secondary portlandite and other deposits, ^ = Possible/unconfirmed, wm = matrix too weak to retain sulfate replacement.

Table 6-9 SEM/EDX features of interest – Rising Wall

Element	Rising Wall					
Location	7MV		21GD		28AW	
Phase 2 Durability Testing	RICS - Stage 3	CSA P3 adapted	RICS - Stage 3	CSA P3 adapted	RICS - Stage 3	CSA P3 adapted
Exposure until failure	350 Days	26 Weeks	350 Days	26 Weeks	350 Days	6 Weeks
Exposure to test conditions	350 Days	26 Weeks	350 Days	26 Weeks	350 Days	6 Weeks
In situ oxidisation - degree	XX	XX	X	X	XX	XXX
In situ oxidisation - frequency	XX	XXX	XX	XX	XX	XXX
Complete sulfide oxidisation	X	XX	X	X	X	XX
Sulfide, surface oxidisation	-	-	X	X	-	XXX
Striped oxidisation of pyrrhotite	XX	XXX	XX	XX	XX	XXX
Cracking (assoc. oxidised sulfides)	XX	XX	X	XX	X	XXX
Calcium depletion of cement matrix	XXX	XX	X	XX	XX	XXX
Remnant aluminium oxide matrix	-	-	-	XX	-	XX
Thaumasite (location)	X^	X^	-	-	X^	-
Secondary ettringite	XXX	XX	-	XXX	XX	-
Secondary gypsum	-	XX	-	-	-	-
ISA matrix conversion	X	X	-	X	X	XXX
Secondary calcite	-	XX	XX	XX	XX	XXX
Leaching	XX	X	X	X	X	XX
Sodium Chloride	-	X	-	-	-	-
Sodium sulfate	-	-	-	-	-	X
Background sulfur	-	-	X	-	-	-

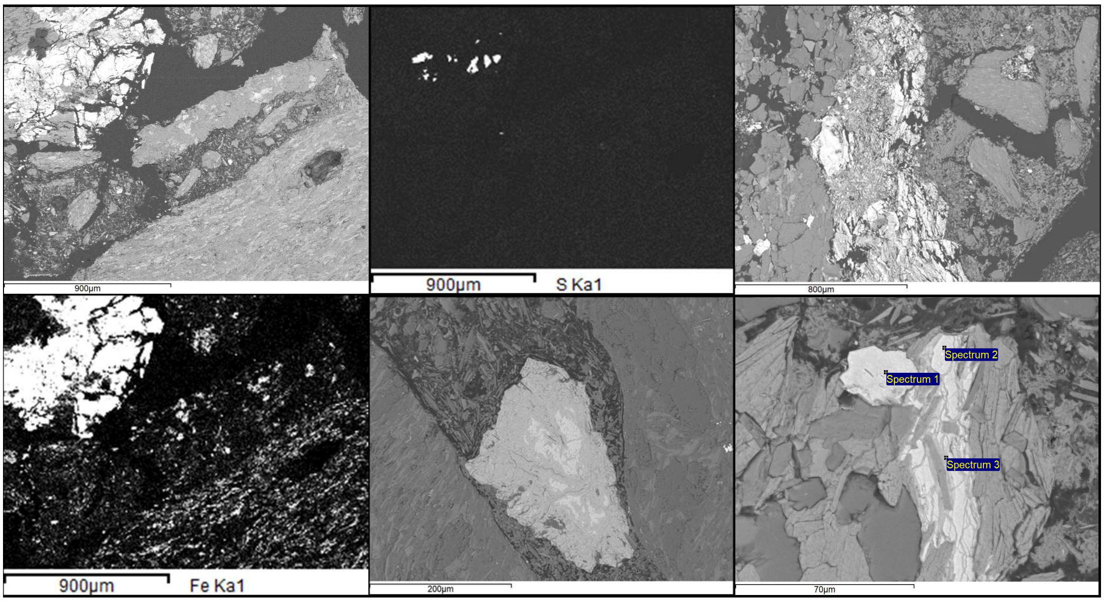
X denotes relative abundances – XXX high abundance, XX moderate abundance, X lower abundance, - absent. Other deposits included secondary portlandite and other deposits, ^ = Possible/unconfirmed.

Table 6-10 SEM/EDX features of interest – Foundation

Element	Foundation						
Location	7MV			21GD		28AW	
Phase 2 Durability Testing	CSA P3 adapted	CSA P3 adapted	RICS - Stage 3	RICS - Stage 3	CSA P3 adapted	RICS - Stage 3	CSA P3 adapted
Exposure until failure	13 Weeks	26 Weeks	350 Days	350 Days	26 Weeks	350 Days	26 Weeks
Exposure to test conditions	13 Weeks	26 Weeks	350 Days	350 Days	26 Weeks	350 Days	26 Weeks
In situ oxidisation - degree	X	X	X	X	X	X	X
In situ oxidisation - frequency	XX	XX	XX	XX	XX	XX	XX
Complete sulfide oxidisation	X	X	X	X	X	X	X
Sulfide, surface oxidisation	X	X	X	X	X	X	X
Striped oxidisation of pyrrhotite	X	X	XX	X	X	X	X
Cracking (assoc. oxidised sulfides)	-	XC	-	-	-	-	-
Calcium depletion of cement matrix	XX	X	XX	X	XX	XX	X
Remnant aluminium oxide matrix	-	-	-	-	-	-	-
Thaumasite (location)	-	-	X^	X^	X^	X^	X
Secondary ettringite	-	-	X	X^	X^	X^	-
Secondary gypsum	-	-	-	-	-	-	-
ISA matrix conversion	XC	XC	X^	X^	XC	X^	XC
Secondary calcite	X	X	X	X	X	X	X
Leaching	X	X	X	X	X	-	X
Sodium Chloride	-	X	-	-	-	-	-
Sodium sulfate	-	-	-	-	-	-	-
Background sulfur	-	-	X	X	X	X	X

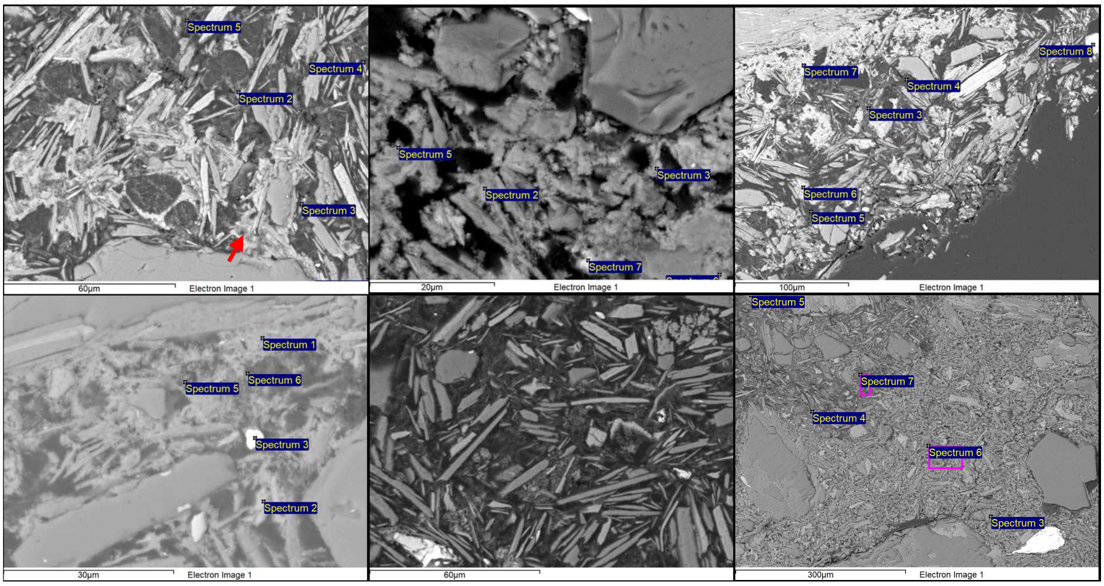
X denotes relative abundances – XXX high abundance, XX moderate abundance, X lower abundance, - absent. Other deposits included secondary portlandite and other deposits, ^ = Possible/unconfirmed, C =Chlorine affected.

Figure 6-19 Photomicrographs – Sulfide oxidisation



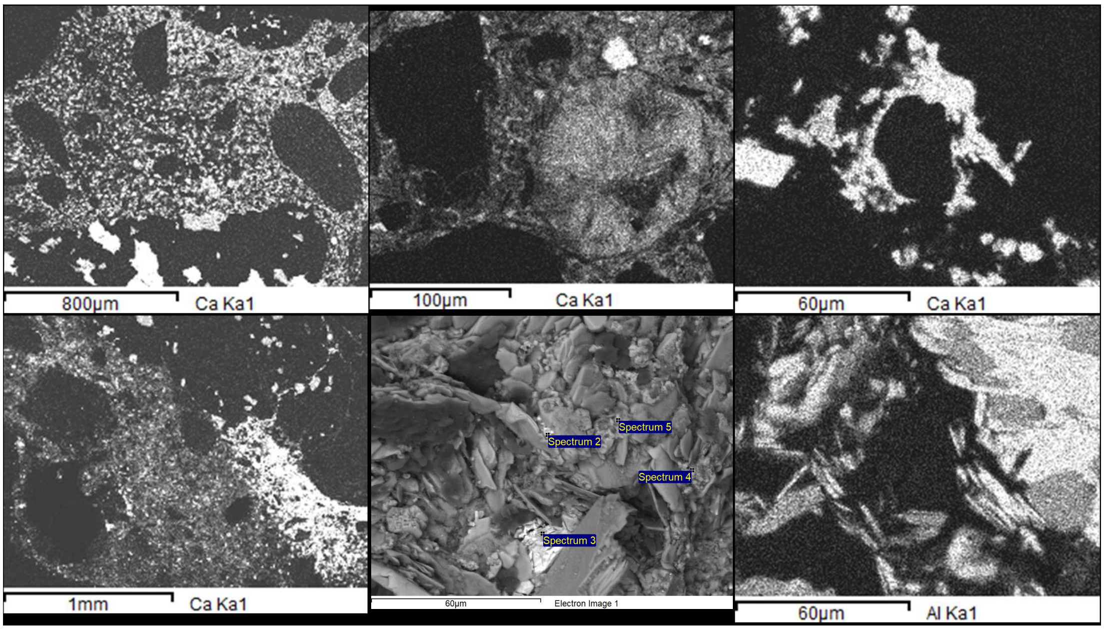
Top and bottom left and top middle: 7MV, IL, CSA P3 adapted, BSE top left, elemental maps, Fe bottom left and S top middle, Near complete oxidised fractured pyrrhotite with surrounding lack of cement matrix. Lower middle: 21GD, OL, CSA P3 adapted, BSE, near completely surface oxidised pyrite. Top right: 21GD, OL, CSA P3 adapted, PPL, Near complete oxidation of pyrrhotite with extensive staining. Lower right: C, OL, RICS Stage 3, BSE, possible partially striped sulfide oxidisation in control sample.

Figure 6-20 Photomicrographs – Secondary sulfates replacing the cement matrix



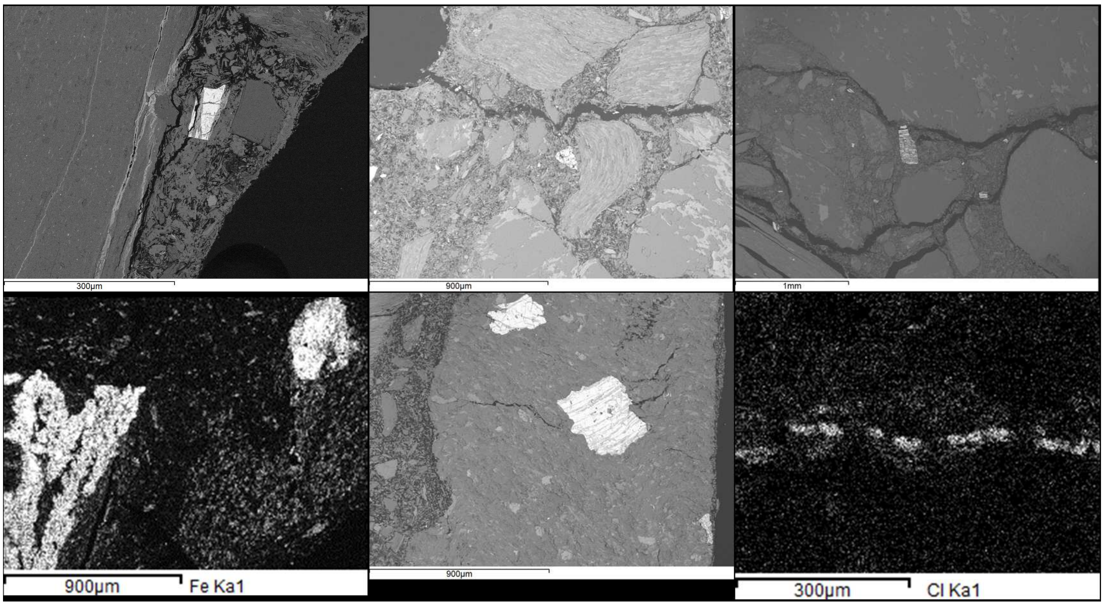
All BSE images. Top left: 7MV, IL, RICS Stage 3, BSE, Secondary gypsum (red arrow) partially replacing cement matrix. Bottom Left: 21GD, IL, RICS Stage 3, BSE, gypsum replacing cement matrix. Top middle: 21GD, OL, CSA P3 adapted, BSE, possible thaumasite replacing cement matrix. Bottom middle: 28AW, RW, CSA P3 adapted, BSE, clustering of calcium and remnant matrix of possible aluminium-silica. Top right: 7MV, IL, CSA P3 adapted, BSE, Ca depleted matrix and possible thaumasite adjacent to the coarse aggregate particle. Bottom right: 21GD, RW, CSA P3 adapted, BSE, depletion of calcite and possible formation of aluminium-sulfates within the cement matrix.

Figure 6-21 SEM/EDX – Weak cement matrix



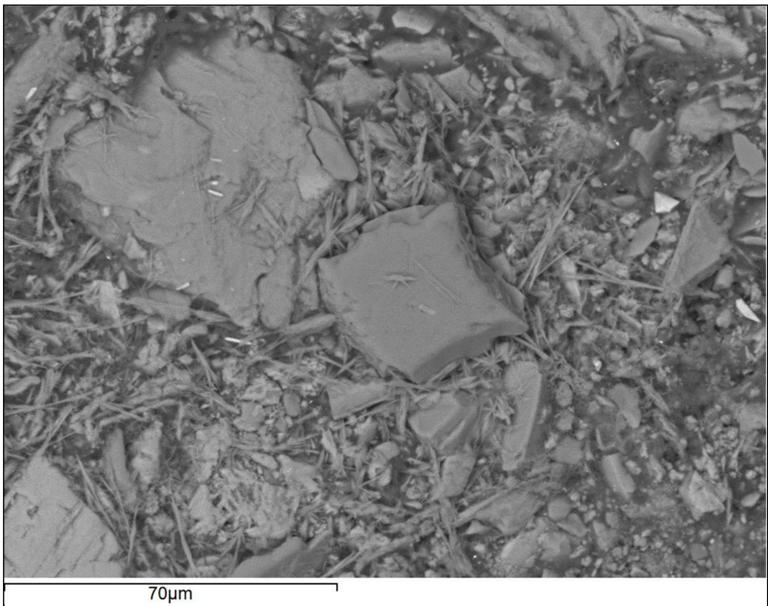
Top left: 7MV, IL, RICS Stage 3, Elemental Ca map, dark areas of Ca depleted weak cement. Top middle: 7MV, RW, RICS Stage 3, Elemental Ca map, depleted calcium within cement matrix. Top and bottom right: 28AW, IL, RICS Stage 3, elemental maps, showing depletion of Ca and remnant Al. Bottom left: 28AW, OL, CSA P3 adapted, Elemental Ca map, poor preservation of cement matrix with low Ca. Bottom middle: 28AW, OL, CSA P3 adapted, BSE, matrix with uneven, platy, mineral-rich texture with rare small patches of cement paste.

Figure 6-22 SEM - Cracking

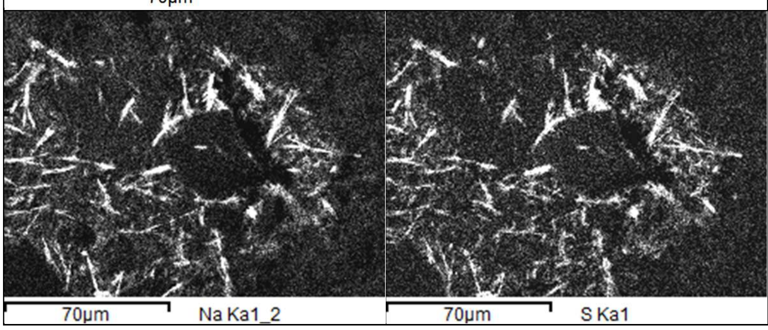


Top left: 28AW, IL, CSA P3 adapted, BSE, extensive cracking through the cement matrix and around aggregate particles. Top middle: 21GD, IL, CSA P3 adapted, BSE, Cracking in cement matrix around aggregate particles. Top right: 7MV, RW, CSA P3 adapted, BSE. Partially oxidised pyrrhotite with associated cracking of cement matrix. Bottom left: 7MV, OL, CSA P3 adapted, Elemental Fe map, highly fractured, heavily oxidised aggregate-bound sulfides. Bottom middle: 21GD, OL, RICS Stage 3, BSE, cracking associated with aggregate bound partially oxidised pyrrhotite. Bottom right: 21GDC, RW, CSA P3 adapted, Elemental Cl map, crack infilled with chlorine-rich secondary ettringite.

Figure 6-23 SEM/EDX Sodium sulfate deposits



2AW IL, CSA P3 adapted BSE image top of secondary sodium sulfate deposits formed on top of the polished surface since preparation.



Left and Right: Elemental EDX maps showing the sodium sulfate deposits.

6.5 XRD Semi-Quantitative Compositional data

The semi-quantitative XRD data is summarised and presented in **Table 6-11**.

Table 6-11 XRD Semi-quantitative compositional results – Phase 3

Property	Element	Durability testing	Composition %																	
			Chlorite	Muscovite	Quartz	Albite	Calcite	Calcium Magnesium Carbonate	Ettringite	Dolomite	Rutile	Anortho-clase	Halite	Gypsum	Paragonite	Calcium phosphate	Zircon	Portland-ite	Hydro-calumite	Calcium Aluminium Oxide Carbonate Hydrate
7MV	IL	RICS Stage 3	38.1	39.1	13.3	6.2	1.2	0.9	0.2	0.2	0.2	--	--	--	--	--	--	0.1	--	--
		CSA P3 adapted	38.1	38.4	13.5	5.9	1.2	1.7	0.3	0.2	0.2	--	0.2	--	--	--	--	--	--	--
	OL	RICS Stage 3	40.6	40.1	11.6	4.7	1.0	1.0	0.3	0.2	0.2	--	--	0.3	--	--	--	--	--	--
		CSA P3 adapted	45.3	34	10.7	2.2	1.0	2.9	--	1.0	0.2	--	--	--	0.8	--	--	--	--	--
	RW	RICS Stage 3	43.3	33.3	13.8	6.2	0.4	1.2	0.6	0.2	0.2	--	--	0.2	--	--	--	0.3	--	--
		CSA P3 adapted	48.9	29.7	11.5	5.6	1.2	1.9	0.3	0.2	0.2	--	0.2	--	--	--	--	--	--	--
	F	CSA P3 adapted	36.2	26.4	24.1	3.7	1.2	6.6	0.4	0.2	0.3	--	--	--	--	--	--	--	0.9	--
		CSA P3 adapted	34.9	28.6	27.1	4.7	1.3	1.4	0.4	0.4	0.2	--	--	--	--	--	--	--	1	--
		RICS Stage 3	34.8	30.2	26.5	3.2	0.4	1.7	0.6	1	0.2	--	--	--	--	--	--	0.4	--	1
21GD	IL	RICS Stage 3	38.2	38.5	13.5	5.9	1.2	1.7	0.3	0.3	0.2	--	--	0.1	--	--	--	--	--	--
		CSA P3 adapted	37.9	39.3	13.4	5.8	1.2	1.4	0.4	0.2	0.2	--	0.2	--	--	--	--	--	--	--
	OL	RICS Stage 3	41.9	36.0	12.7	6.4	1.0	0.9	0.4	0.1	0.2	--	--	0.2	--	--	--	--	--	--
		CSA P3 adapted	43.4	40.5	9.6	2.3	0.9	1.2	--	--	--	1.6	--	--	0.6	--	--	--	--	--
	RW	RICS Stage 3	38.2	40.7	11.7	6.2	0.4	1.4	0.4	0.2	0.2	--	--	--	--	--	--	--	--	--
		CSA P3 adapted	41.9	35.0	14.8	5.2	1.0	1.0	0.3	0.1	0.2	--	0.3	--	--	--	--	--	--	--
	F	RICS Stage 3	35.5	46.2	11.5	3.3	0.9	--	0.6	--	0.3	--	--	--	--	--	--	0.7	--	1
		CSA P3 adapted	33.5	38.4	18.3	5.3	1.1	1.6	0.4	--	0.3	--	--	--	--	--	--	--	1.1	--
28AW	IL	RICS Stage 3	40.3	44.0	8.0	4.3	1.2	1.1	0.3	0.3	0.2	--	--	0.2	--	--	--	--	--	--
		CSA P3 adapted	43.1	39.2	10.5	4.0	0.8	--	--	0.5	--	0.9	--	--	0.9	--	--	--	--	--
	OL	RICS Stage 3	40.8	39.8	11.0	3.9	0.7	2.9	0.4	0.3	0.1	--	--	--	--	--	0.1	--	--	--
		CSA P3 adapted	33.4	34.7	23.3	5.5	1.4	--	--	--	0.3	--	0.5	--	--	0.8	0.1	--	--	--
	RW	RICS Stage 3	43.0	38.7	11.2	4.4	0.6	1.0	0.8	0.1	0.2	--	--	--	--	--	--	--	--	--
		CSA P3 adapted	43.1	40.3	10.4	4.4	0.6	0.3	0.3	0.1	0.2	--	0.3	--	--	--	--	--	--	--
	F	RICS Stage 3	37.3	26.8	22.8	5.9	1	3.7	0.7	--	0.3	--	--	--	--	--	--	0.3	--	1.1
		CSA P3 adapted	36.1	32.6	23.2	3.2	2.7	--	0.5	--	0.4	--	--	--	--	--	--	--	1.3	--
C	IL	RICS Stage 3	47.1	23.6	19.6	7.7	1.2	--	0.3	0.2	0.3	--	--	0.1	--	--	--	--	--	--
		CSA P3 adapted	40.3	15.2	31.9	9.0	3.0	--	0.2	--	0.2	--	0.1	--	--	--	--	--	--	--
	OL	RICS Stage 3	47.8	20.7	21.6	7.3	1.2	--	0.2	0.3	0.2	--	--	0.1	--	--	--	--	--	--
		CSA P3 adapted	43.0	14.7	28.6	10.5	2.6	--	0.2	0.1	0.2	--	0.1	--	--	--	--	--	--	--

Table 6-12 XRD Semi-quantitative compositional results – Phase 3 (2)

Property	Element	Durability testing	Composition %		
			Iron Sulfide/ Pyrite (FeS_2)	Szomolnokite ($FeSO_4 H_2O$)	Iron Oxide (Fe_2O_3)
7MV	IL	RICS Stage 3	0.3	0.1	--
		CSA P3 adapted	0.5	--	--
	OL	RICS Stage 3	0.1	--	--
		CSA P3 adapted	--	--	--
	RW	RICS Stage 3	0.1	--	--
		CSA P3 adapted	--	--	0.3
	F	CSA P3 adapted	--	--	--
		CSA P3 adapted	--	--	--
		RICS Stage 3	--	--	--
21GD	IL	RICS Stage 3	0.1	--	--
		CSA P3 adapted	0.1	--	--
	OL	RICS Stage 3	0.1	--	--
		CSA P3 adapted	--	--	--
	RW	RICS Stage 3	0.4	0.2	--
		CSA P3 adapted	0.2	--	--
	F	RICS Stage 3	--	--	--
		CSA P3 adapted	--	--	--
28AW	IL	RICS Stage 3	0.1	--	--
		CSA P3 adapted	--	--	--
	OL	RICS Stage 3	0.1	--	--
		CSA P3 adapted	0.1	--	--
	RW	RICS Stage 3	0.1	--	--
		CSA P3 adapted	--	--	0.1
	F	RICS Stage 3	--	--	--
		CSA P3 adapted	--	--	--
C	IL	RICS Stage 3	--	--	--
		CSA P3 adapted	--	--	--
	OL	RICS Stage 3	--	--	0.5
		CSA P3 adapted	--	--	--

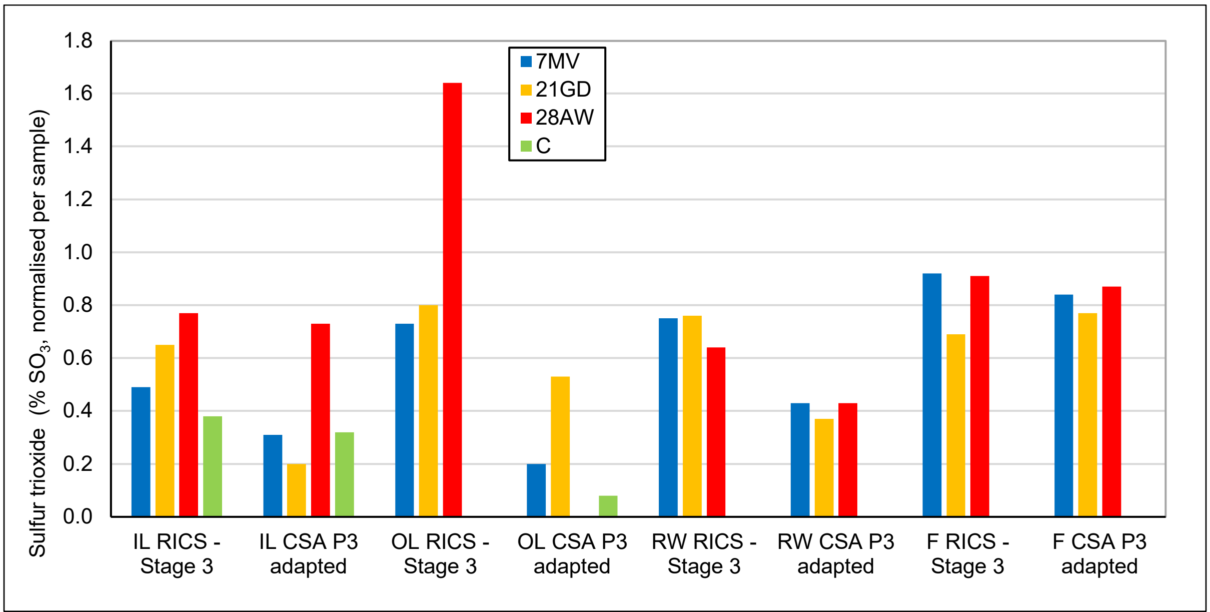
6.6 XRF

Table 6-13 XRF Semi-quantitative compositional results

Location	Element	Durability testing	Sodium oxide – Na2O	Magnesium oxide – MgO	Aluminium oxide – Al2O3	Silicon dioxide – SiO2	Phosphorous pentoxide – P2O5	Sulfur trioxide – SO3	Chlorine – Cl	Potassium oxide – K2O	Calcium oxide – CaO	Titanium dioxide – TiO2	Manganese oxide – MnO	Iron oxide – Fe2O3	Sum of Concentrations before normalisation
7MV	IL	RICS - Stage 3	1.10	1.63	23.24	58.13	0.14	0.49	-	3.34	5.02	0.62	0.16	6.00	89.90
		CSA P3 adapted	2.02	1.50	23.17	55.95	0.21	0.31	0.74	3.60	5.16	0.61	0.14	6.44	86.14
	OL	RICS - Stage 3	0.93	1.63	24.32	55.05	0.14	0.73	<<	3.90	5.74	0.72	0.16	6.53	86.80
		CSA P3 adapted	1.93	1.61	24.91	52.97	0.20	0.20	0.66	4.09	5.36	0.71	0.14	7.05	85.60
	RW	RICS - Stage 3	1.06	1.60	23.53	56.12	0.17	0.75	<<	3.35	6.25	0.65	0.15	6.23	89.10
		CSA P3 adapted	2.41	1.57	21.07	54.83	0.17	0.43	0.89	2.89	7.50	0.64	0.17	7.30	79.00
	F	CSA P3 adapted	2.54	1.41	18.59	51.01	0.12	0.85	1.96	2.56	13.97	0.60	0.16	6.15	78.90
		CSA P3 adapted	2.55	1.43	18.97	50.16	0.12	0.84	2.08	2.61	14.50	0.58	0.20	5.90	81.10
		RICS - Stage 3	0.99	1.75	20.12	52.44	0.13	0.92	0.06	2.63	13.02	0.61	0.25	7.00	79.60
21GD	IL	RICS - Stage 3	0.99	1.61	23.97	56.44	0.18	0.65	<<	3.61	5.50	0.65	0.13	6.15	89.20
		CSA P3 adapted	2.28	1.54	23.79	53.80	0.14	0.20	1.00	3.56	6.04	0.69	0.13	6.67	87.20
	OL	RICS - Stage 3	1.04	1.68	24.67	55.38	0.19	0.80	<<	3.66	5.31	0.65	0.15	6.34	89.80
		CSA P3 adapted	1.92	1.60	23.05	54.78	0.25	0.53	0.68	3.48	5.86	0.65	0.16	6.91	85.60
	RW	RICS - Stage 3	0.98	1.68	24.50	54.33	0.15	0.76	<<	3.78	6.17	0.63	0.16	6.72	88.30
		CSA P3 adapted	2.18	1.59	23.39	53.34	0.19	0.37	1.13	3.65	6.57	0.68	0.15	6.61	81.00
	F	RICS - Stage 3	1.03	1.71	21.21	52.99	0.12	0.69	0.06	2.93	12.19	0.63	0.18	6.19	79.50
		CSA P3 adapted	2.30	1.44	19.85	49.85	0.11	0.77	1.77	2.90	14.09	0.63	0.11	6.05	80.00
28AW	IL	RICS - Stage 3	0.82	1.72	0.82	54.27	0.15	0.77	<<	4.13	5.47	0.66	0.16	6.32	90.00
		CSA P3 adapted	2.11	1.57	2.11	52.26	0.13	0.73	1.09	3.78	6.17	0.68	0.13	6.85	85.70
	OL	RICS - Stage 3	1.35	2.25	25.83	50.91	0.15	1.64	0.179	5.21	6.47	0.85	0.11	4.837	97.75
		CSA P3 adapted	3.05	2.02	23.93	53.28	0.11	-	1.43	4.06	4.96	0.65	0.15	6.23	88.00
	RW	RICS - Stage 3	0.89	1.67	0.89	54.49	0.10	0.64	<<	4.12	5.42	0.68	0.15	6.27	89.00
		CSA P3 adapted	1.78	1.55	1.78	52.35	0.16	0.43	0.77	4.20	3.86	0.69	0.13	7.52	85.80
	F	RICS - Stage 3	0.86	1.65	23.54	49.32	0.11	0.91	0.07	3.77	11.52	0.73	0.18	7.17	78.60
		CSA P3 adapted	6.06	1.49	20.44	43.66	0.08	0.87	5.40	3.26	11.41	0.64	0.17	6.38	82.00
C	IL	RICS - Stage 3	1.49	2.47	1.49	62.88	0.15	0.38	<<	1.70	6.62	0.72	0.13	7.36	83.70
		CSA P3 adapted	2.52	2.68	2.52	58.12	0.18	0.32	0.54	1.48	9.07	0.85	0.16	9.46	71.20
	OL	RICS - Stage 3	1.44	3.12	1.44	61.88	0.17	-	0.04	-	6.78	0.77	0.14	8.02	84.90
		CSA P3 adapted	2.22	2.80	2.22	62.02	0.16	0.08	0.41	1.65	6.08	0.76	0.11	7.58	86.20

p **= pending, << trace below limit of quantification. All elemental data presented as oxides, normalised to 100% of the sample and ignoring elements lighter than F.**

Figure 6-24 XRF Sulfur reported as sulfate, normalised



Note that results for C OL RICS Stage 3 and 28AW OL CSA P3 adapted were 0, and no C RW and F sub-samples were tested in Stage 2 and 3 of the project. All S data presented as SO_3, normalised to 100% of each separate sample and ignoring elements lighter than F.

6.7 Physical testing

A summary of chemistry testing is presented in **Table 6-12** and **Table 6-13** and followed by graphical representations of each test and any relevant information as required. Note that in all graphical representations, there was no rising wall in C, and no samples tested for the foundation of C.

Each set of results is presented separately for the two different Phase 2 durability test sub-samples (i.e. RICS testing separate from CSA testing) to be directly comparable.

Table 6-14 Physical test data – RICS Stage 3

Location		7MV	7MV	7MV	7MV	21GD	21GD	21GD	28AW	28AW	28AW	28AW	C	C
Element		IL	OL	RW	F	IL	OL	RW	IL	RW	RW	F	IL	OL
RSK Sample Ref		20511/B2	20511/B4	20511/C6	20511/C7	20511/B14	20511/B16	20511/C25	20511/B8	20511/C18	20954/C4	20954/C5	20511/B6	20511/B7
RSK Sub-sample Ref		G	H	A	E	B	C	E	A	E	A	A	F	F
Exposure length	*days*	350	350	350	350	350	350	350	350	350	250	250	350	350
C/S	*N/mm²*	6.5	6.0	7.4	17.8	2.8	2.4	0.7	2.0	4.1	7.3	13.4	8.4	9.4
Previous C/S	*N/mm²*	7.9	8.6	12.8	29.9	9.1	10.1	5.9	8.6	5.9	5.9	22.2	14.1	12.8
ΔC/S	*N/mm²*	-1.4	-2.6	-5.4	-12.1	-6.3	-7.7	-5.2	-6.6	-1.8	1.4	-8.8	-5.7	-3.4
Density	*kg/m³*	2420	2150	2230	2280	2200	2120	2000	2220	2230	2210	2420	2230	2310
Previous density	*kg/m³*	2170	2150	2190	2240	2170	1990	2090	2370	2050	2050	2260	2060	2270
ΔDensity	*kg/m³*	250	0	40	40	30	130	-90	-150	180	160	160	170	40

Increases in density may be related to precipitation of secondary minerals incorporating material from outside the sample originally.

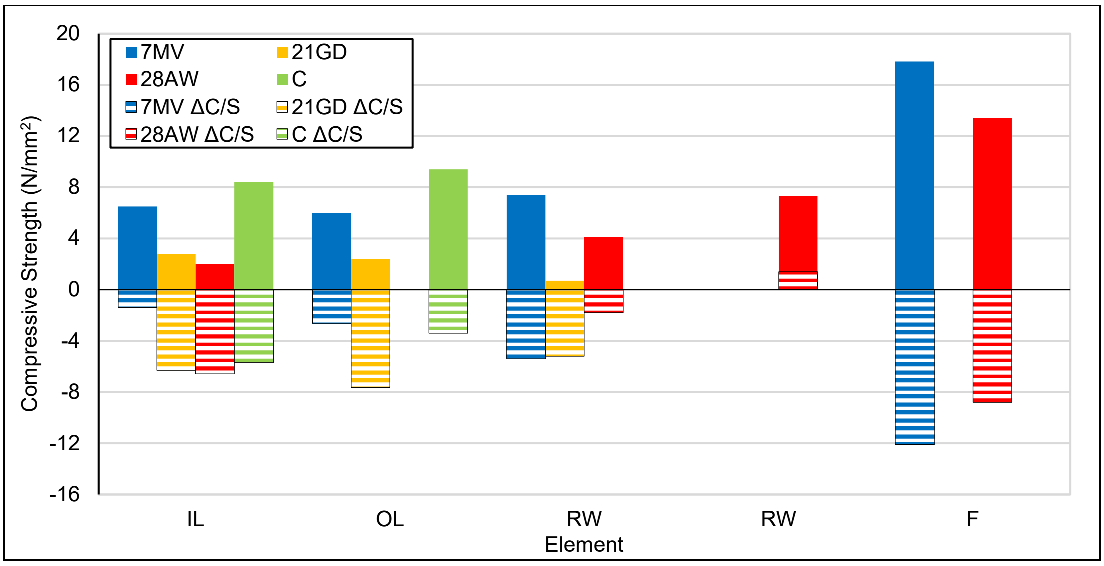
Table 6-15 Physical test data CSA P3 adapted

Location		7MV	7MV	7MV	21GD	21GD	21GD	28AW	C	C
Element		IL	OL	F	IL	OL	RW	F	IL	OL
RSK Sample Ref		20511/B3	20511/B5	20511/C9	20511/B15	20511/B17	20511/C26	20954/C5	20511/B6	20511/B7
RSK Sub-sample Ref		A	D	C	A	D	B	B	I	J
Exposure length	*weeks*	8	8	26	10	8	26	26	26	26
C/S	*N/mm²*	1.8	1.0	11.9	0.8	2.8	2.2	16.7	5.2	7.4
Previous C/S	*N/mm²*	7.9	8.6	29.9	9.1	10.1	5.9	22.2	14.1	12.8
ΔC/S	*N/mm²*	-6.1	-7.6	-18.0	-8.3	-7.3	-3.7	-5.5	-8.9	-5.4
Density	*kg/m³*	1970	1900	2380	1750	2050	2150	2060	2180	2300
Previous density	*kg/m³*	2170	2150	2240	2170	1990	2090	2260	2060	2270
ΔDensity	*kg/m³*	-200	-250	140	-420	60	60	-200	120	30

6.7.1 Compressive Strength

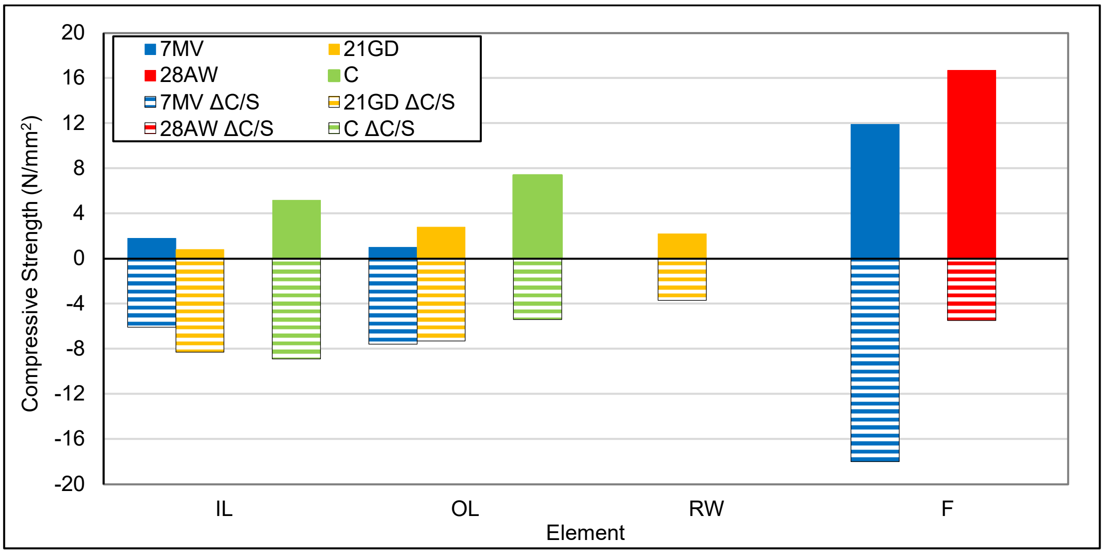
The limited suite of compressive strength results is presented below. For more details on methodology and measurements see **4.7** and **Appendix C**. Compressive strength results are presented as core compressive strengths, highlighting the changes from Phase 1 results.

Figure 6-25 Compressive strength data – RICS Stage 3



Solid columns represent Phase 3 results, whilst horizontally striped columns represent the change (usually loss) in compressive strength between Phase 1 and Phase 3 testing. There are two RW elements present to accommodate more than one sample tested for 28AW RW. No 28AW OL, 21GD F and C RW or F samples were tested. Samples tested in accordance with BS EN 12504-1:2019[32].

Figure 6-26 Compressive strength data – CSA P3 adapted



Solid columns represent Phase 3 results, whilst horizontally striped columns represent the change (usually loss) in compressive strength between Phase 1 and Phase 3 testing. . No 28AW IL, OL, RW, 21GD F or C RW and C F samples were tested. Samples tested in accordance with BS EN 12504-1:2019[33].

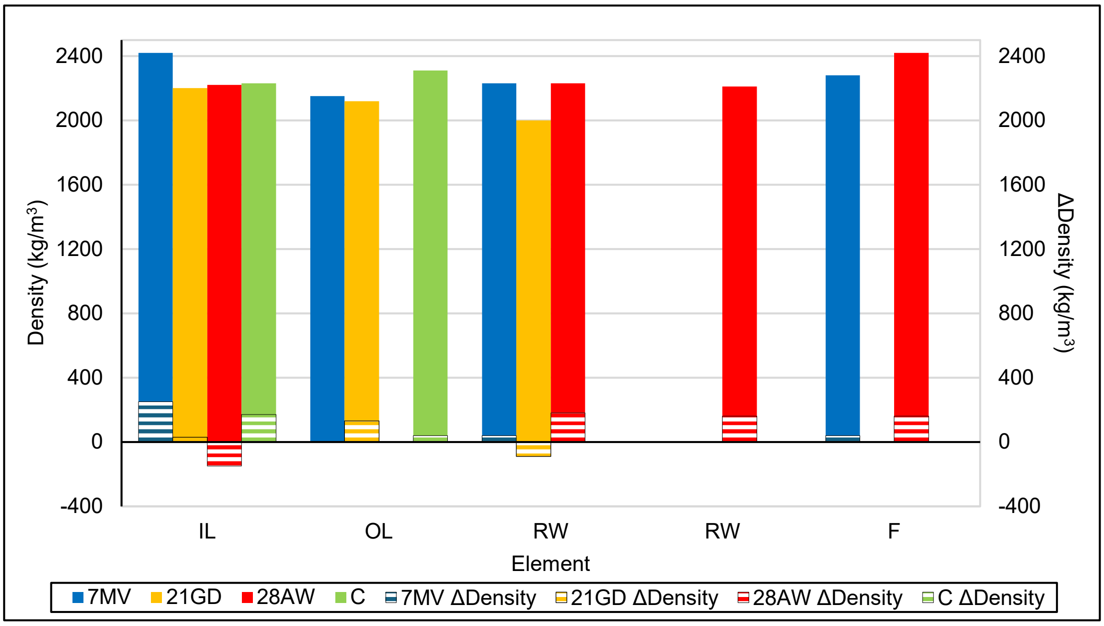
[32] BS EN 12504-1:2019. See [20]

[33] BS EN 12504-1:2019. See [20]

6.7.2 Density

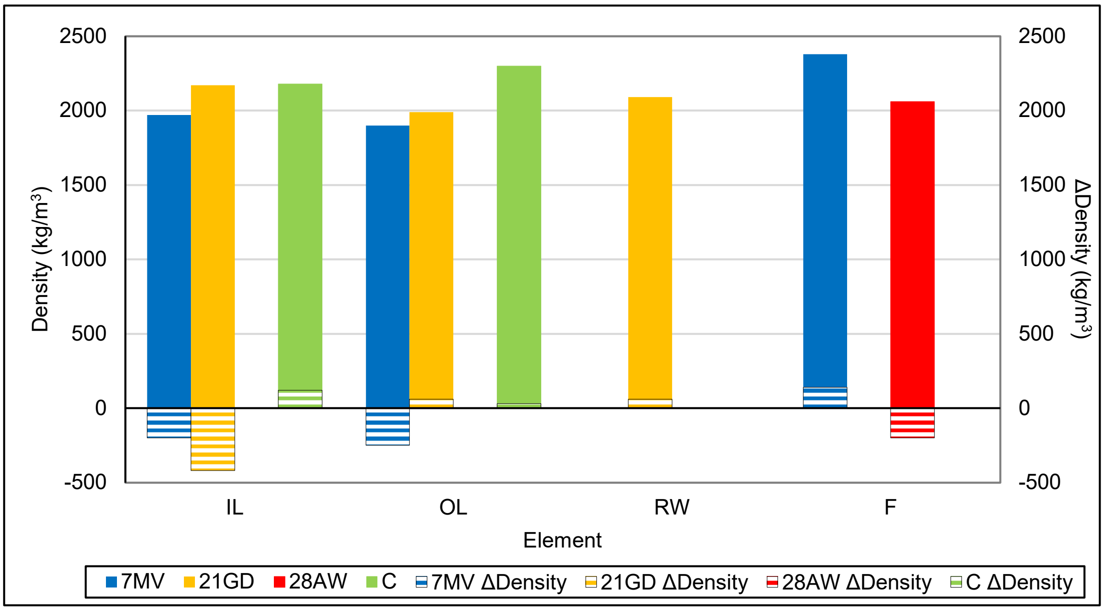
The limited suite of compressive strength results is presented below, for more details on methodology and measurements see **4.8** and **Appendix C**. Density results highlight the changes from Phase 1 results.

Figure 6-27 Density data – RICS Stage 3



Solid columns represent Phase 3 results, whilst horizontally striped columns represent the in density between Phase 1 and Phase 3 testing. There are two RW elements present to accommodate more than one sample tested per element per property. No 28AW OL, 21GD F or C RW or F samples were tested. Phase 3 samples tested in accordance with BS EN 12390-7:2019+AC:2020.

Figure 6-28 Density data – CSA P3 adapted



Solid columns represent Phase 3 results, whilst horizontally striped columns represent the change in density between Phase 1 and Phase 3 testing. No 7MV RW, 28AW IL, OL, RW or 21GD F or C RW or F samples were tested. Phase 3 samples tested in accordance with BS EN 12390-7:2019+AC:2020.

6.8 Chemical testing

A summary of chemistry testing is presented in **Table 6-12** and **Table 6-13** and followed by graphical representations of each test and any relevant information as required. Note that in all graphical representations, there was no rising wall in C, and no samples tested for the foundation of C.

Each set of results is presented separately for the two different durability sub-samples to be directly comparable.

Where multiple results are present for the same test on the same element the greatest value is graphically represented unless multiple entries of the same element are stated.

Table 6-16 Chemical data – Phase 3 – RICS Stage 3

Standard	Test	*Location*	7MV	7MV	7MV	7MV	7MV	21GD	21GD	21GD	21GD	28AW	28AW	28AW	28AW	28AW	28AW	C	C
		Element	IL	OL	RW	F	F	IL	OL	RW	F	IL	OL	RW	RW	F	F	IL	OL
		Exposure length, days	350	350	350	350	350	350	350	350	350	350	250	350	250	350	250	350	350
EN 1744-1	Total sulfur – Acid digestion (TS)	*S % mass (sample)*	0.4	0.4	0.2	0.2	-	0.6	0.5	0.2	0.2	0.5	0.4	0.2	0.2	-	0.2	0.1	0.1
		S % mass (aggregate)	0.3	0.3	0.1	0	-	0.5	0.4	0.1	0.0	0.4	0.3	0.1	0.1	-	0.0	0.0	0.0
	Total sulfur – HTC (TS)	*S % mass (sample)*	0.58	0.60	0.52	0.61	0.58	1.11	0.72	-	0.54	0.80	0.72	-	0.77	0.51	0.54	0.09	-
		S % mass (aggregate)	0.48	0.50	0.42	0.41	0.38	1.01	0.62	-	0.34	0.70	0.62	-	0.67	0.31	0.34	0.00	-
	Acid soluble sulfate (ASS)	*SO_4 % mass (sample)*	0.3	0.3	0.3	0.5	-	0.2	0.4	0.3	0.5	0.2	0.3	0.3	0.4	-	0.3	0.2	0.2
	Water soluble sulfate (WSS)	*SO_3 % mass (sample)*	0.15	0.12	-	0.00	-	0.12	0.25	0.01	0.00	0.12	0.18	0.01	0.01	-	0.01	0.12	0.12
TRL447, Table 8.1.	Total potential sulfate - HTC (TPS)	*SO_4 % mass (aggregate)*	1.44	1.50	1.26	1.23	1.14	3.03	1.86	-	1.02	2.10	1.86	-	2.01	0.93	1.02	<0.03	-
	Oxidisable sulfides (OS)	*SO_4 % mass (sample)*	1.14	1.20	0.96	0.73	-	2.83	1.46	-	0.52	1.90	1.56	-	1.61	-	0.72	0.00	-
	Equivalent pyrite content	*FeS_2 % mass (sample)*	0.71	0.75	0.60	0.46	-	1.77	0.91	-	0.33	1.19	0.98	-	1.01	-	0.45	-	-
	Equivalent pyrrhotite content	*FeS % mass (sample)*	1.05	1.10	0.88	0.67	-	2.59	1.34	-	0.48	1.74	1.43	-	1.48	-	0.66	-	-
EN 196-2	Sulfide content	*S^{2-} % mass (sample)*	0.45	0.56	0.54	0.05	-	0.55	0.44	0.59	0.02	0.58	0.52	0.62	0.28	-	0.55	0.05	0.05
		S^{2-} % mass (aggregate)	0.58	0.72	0.70	0.07	-	0.72	0.53	0.73	0.03	0.75	0.63	0.75	0.34	-	0.73	0.06	0.07
	Acid-soluble sulfate content	*SO_4 % mass*	0.36	0.29	0.31	0.49	-	0.29	0.46	0.18	0.47	0.28	0.40	0.25	0.43	-	0.32	0.17	0.24
BS 1881-124	Cement content	*kg/m^3*	120	150	180	360	-	110	110	100	290	90	110	160	140	-	270	110	90
BRE SD1 Suite D	pH	*(Concrete)*	9.01	10.63	11.92	12.47	-	9.16	9.95	11.59	12.3	9.08	11.03	11.65	11.51	-	12.44	9.05	8.8
		(Leachate)	7.10	6.35	6.50	9.27	-	6.22	7.87	6.43	10.33	6.49	-	8.30	-	7.01	-	6.78	6.93
	Acid soluble sulfate - ICP-OES (ASS)	*SO_4 % mass (sample)*	0.52	1.91	0.82	1.65	-	0.45	1.49	1.85	0.68	0.64	3.36	0.68	3.00	-	0.02	0.22	0.26
	Water soluble sulfate - Colorimetry (WSS)	*SO_4 % mass (sample)*	0.22	0.12	0.13	0.03	-	0.19	0.28	0.26	0.01	0.20	0.19	0.16	0.10	-	0.01	0.14	0.19
		SO_4 mg/l (sample)	1120	610	634	132	-	967	1420	1290	37	1010	950	810	523	-	58	695	949
		SO_4 mg/l (leachate)	3850	2630	2290	1770	-	2940	2890	2350	483	2830	-	2150	-	1910	-	2840	3620
	Total sulfur – ICP-OES (TS)	*S % mass (sample)*	0.49	0.65	0.85	0.53	-	0.48	0.67	0.68	0.57	0.73	0.74	0.66	0.71	-	0.50	0.09	0.11
		S % mass (aggregate)	0.39	0.55	0.75	0.33	-	0.38	0.57	0.58	0.37	0.63	0.64	0.56	0.61	-	0.30	0.00	0.01
TRL447, Table 8.1.	Total potential sulfate - ICP-OES (TPS)	*SO_4 % mass*	1.17	1.65	2.25	0.99	-	1.14	1.71	1.74	1.11	1.89	1.92	1.68	1.83	-	0.90	0.00	0.03
	Oxidisable sulfides (OS)	*SO_4 % mass*	0.65	0.00	1.43	0.00	-	0.69	0.22	-0.11	0.43	1.25	-	1.00	-	-	0.88	0.00	0.00
	Equivalent pyrite content	*FeS_2 % mass*	0.41	-	0.89	-	-	0.43	0.14	-0.07	0.27	0.78	-	0.63	-	-	0.55	-	-
	Equivalent pyrrhotite content	*FeS % mass*	0.60	-	1.31	-	-	0.63	0.20	-0.10	0.39	1.15	-	0.92	-	-	0.81	-	-

Table 6-17 Chemical data – Phase 3 – CSA P3 adapted

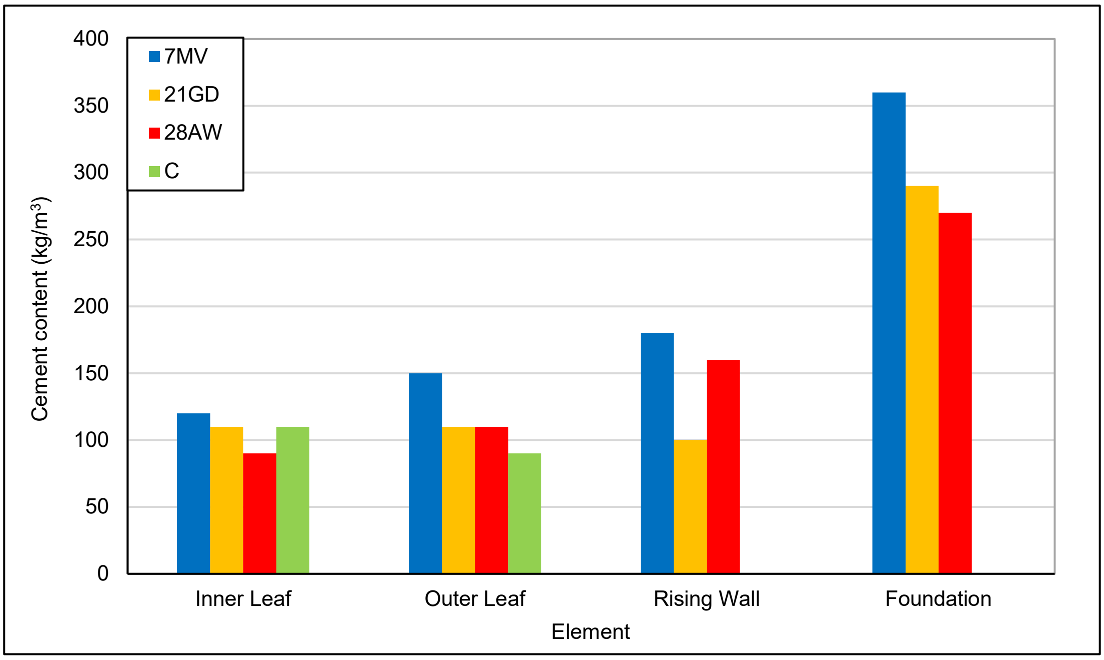
Standard	Test	*Location*	7MV	7MV	7MV	7MV	7MV	7MV	7MV	7MV	21GD	21GD	21GD	21GD	21GD	21GD	28AW	28AW	28AW	28AW	28AW	C	C	C
		Element	IL	IL	OL	OL	RW	F	F	F	IL	OL	OL	RW	RW	F	IL	OL	RW	F	F	IL	OL	OL
		Exposure length, weeks	26	26	26	26	26	13	26	26	26	7	26	26	26	26	4	26	26	26	26	26	26	26
		Previous Testing	CSA P3 adapted																					
EN 1744-1	Total sulfur – Acid digestion (TS)	*S % mass (sample)*	-	0.1	-	0.3	0.2	-	-	0.2	0.1	0.4	-	0.2	-	0.2	0.3	0.1	0.2	-	0.2	<0.1	-	<0.1
		S % mass (aggregate)	-	0.0	-	0.2	0.1	-	-	0.0	0.0	0.3	-	0.1	-	0.0	0.2	0.0	0.1	-	0.0	0.0	-	0.0
	Total sulfur – HTC (TS)	*S % mass (sample)*	0.42	-	0.14	-	0.04	0.43	0.46	0.55	0.13	0.57	0.28	-	0.27	0.62	0.32	0.12	0.28	0.39	0.50	0.01	0.24	-
		S % mass (aggregate)	0.32	-	0.04	-	0.00	0.23	0.26	0.35	0.03	0.47	0.18	-	0.17	0.42	0.22	0.02	0.18	0.19	0.30	0.00	0.14	-
	Acid soluble sulfate (ASS)	*SO_4 % mass (sample)*	-	0.2	-	0.3	0.5	-	-	0.6	0.1	0.6	-	0.3	-	0.4	0.4	0.2	0.2	-	0.4	0.0	-	0.1
	Water soluble sulfate (WSS)	*SO_3 % mass (sample)*	-	0.05	-	0.05	0.12	-	-	0.03	0.03	0.41	-	0.14	-	0.07	0.22	0.03	0.06	-	0.04	0.01	-	0.00
TRL447, table 8.1.	Total potential sulfate - HTC (TPS)	*SO_4 % mass (aggregate)*	0.96		0.12		0.00	0.69	0.78	1.05	0.09	1.41	0.54	0.51		1.26	0.66	0.06	0.54	0.57	0.90	0.00	0.42	
	Oxidisable sulfides (OS)	*SO_4 % mass (sample)*	0.76		0.00		-	0.00	0.00	0.45	0.00	0.81	0.00	0.21		0.86	0.26	0.00	0.34	0.00	0.50	-	0.32	
	Equivalent pyrite content	*FeS_2 % mass (sample)*	0.48		-	-	-	-	-	0.28	-	0.51	-	0.13		0.54	0.16	-	0.21	-	0.31	-	0.20	
	Equivalent pyrrhotite content	*FeS % mass (sample)*	0.70		-	-	-	-	-	0.41	-	0.74	-	0.19		0.79	0.24	-	0.31	-	0.46	-	0.29	
EN 196-2	Sulfide content	*S^{2-} % mass (sample)*	-	0.17	-	0.40	0.41	-	-	0.03	0.13	0.36	-	0.50	-	0.01	0.26	0.16	0.11	-	0.18	0.03	-	0.05
		S^{2-} % mass (aggregate)	-	0.22	-	0.51	0.53	-	-	0.04	0.17	0.43	-	0.62	-	0.01	0.34	0.19	0.13	-	0.24	0.04	-	0.07
	Acid-soluble sulfate content	*SO_4 % mass*	-	0.16	-	0.23	0.35	-	-	0.59	0.07	0.65	-	0.31	-	0.54	0.40	0.12	0.22	-	0.43	0.05	-	0.04
BS 1881-124	Cement content	*kg/m^3*	-	100	-	130	180	-	-	360	90	100	-	170	-	290	160	140	160	-	270	90	-	70
BRE SD1 Suite D	pH	*(Concrete)*	-	10.72	-	11.29	11.72	-	-	11.85	10.75	9.91	-	10.79	-	11.80	9.88	10.54	10.46	-	-	10.88	-	10.74
	Acid soluble sulfate - ICP-OES (ASS)	*SO_4 % mass (sample)*	-	0.18		0.20	0.36	-	-	0.71	0.14	0.95	-	1.16	-	0.47	0.53	0.19	0.17	-	-	0.05	-	0.07
	Water soluble sulfate - Colorimetry (WSS)	*SO_4 % mass (sample)*	-	0.06	-	0.07	0.16	-	-	0.21	0.04	0.64	-	0.27	-	0.13	0.30	0.06	0.08	-	-	0.01	-	0.01
		SO_4 mg/l (sample)	-	317	-	340	783	-	-	1040	207	3210	-	1370	-	675	1510	285	387	-	-	53	-	56
	Total sulfur – ICP-OES (TS)	*S % mass (sample)*	-	0.25	-	0.36	0.55	-	-	0.54	0.11	0.82	-	0.81	-	0.61	0.47	0.16	0.24	-	-	0.06	-	0.03
		S % mass (aggregate)	-	0.15	-	0.26	0.45	-	-	0.34	0.01	0.72	-	0.71	-	0.41	0.37	0.06	0.14	-	-	0.00	-	0.00
TRL447, table 8.1.	Total potential sulfate - ICP-OES (TPS)	*SO_4 % mass*	-	0.45	-	0.78	1.35	-	-	1.02	0.03	2.16	-	2.13	-	1.23	1.11	0.18	0.42	-	-	0.00	-	0.00
	Oxidisable sulfides (OS)	*SO_4 % mass*	-	0.27	-	0.58	0.99	-	-	0.31	0.00	1.21	-	0.97	-	0.76	0.58	0.00	0.25	-	-	-	-	-
	Equivalent pyrite content	*FeS_2 % mass*	-	0.17	-	0.36	0.62	-	-	0.19	-	0.76	-	0.61	-	0.48	0.36	-	0.16	-	-	-	-	-
	Equivalent pyrrhotite content	*FeS % mass*	-	0.25	-	0.53	0.91	-	-	0.28	-	1.11	-	0.89	-	0.70	0.53	-	0.23	-	-	-	-	-

Data aligned over two columns utilises data from both columns.

6.8.1 Cement content

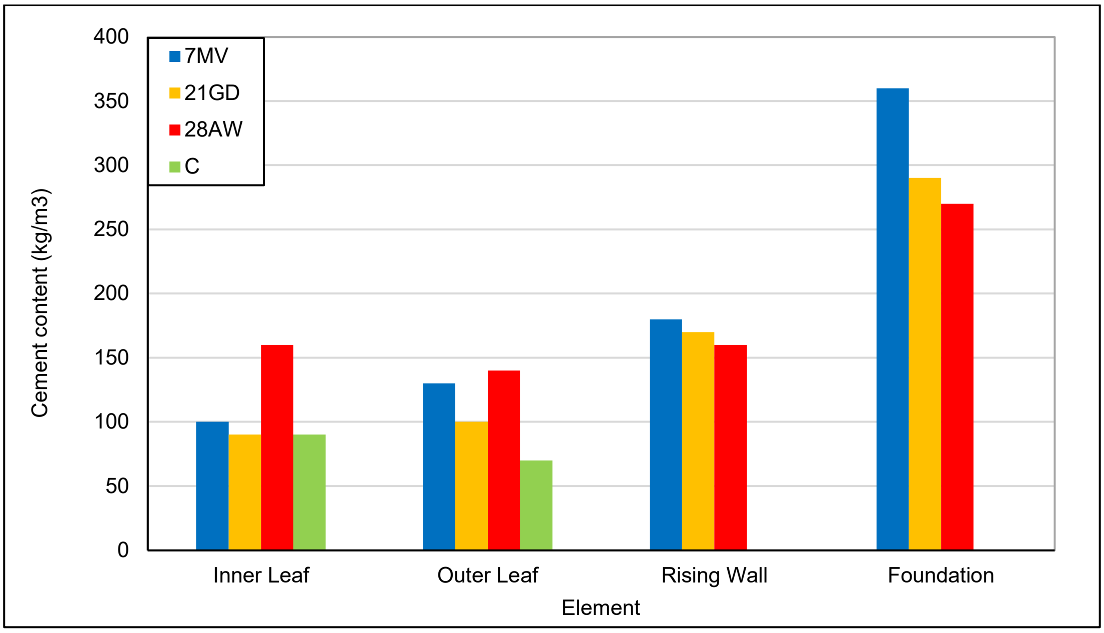
The results of cement content and calculated differences with Phase 1 results testing are presented below. For more details on methodology and measurements please see **Section 4.9** and **Appendix C**.

Figure 6-29 Cement content data – after RICS Stage 3



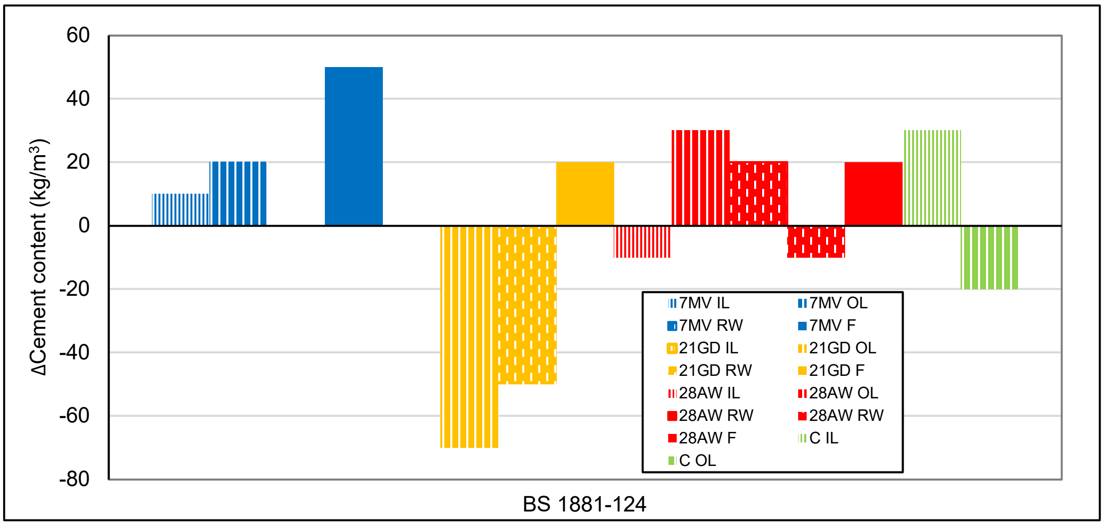
28AW RW 140 kg/m³ not represented. C RW did not exist and C F test was not conducted.

Figure 6-30 Cement content data – after CSA P3 adapted



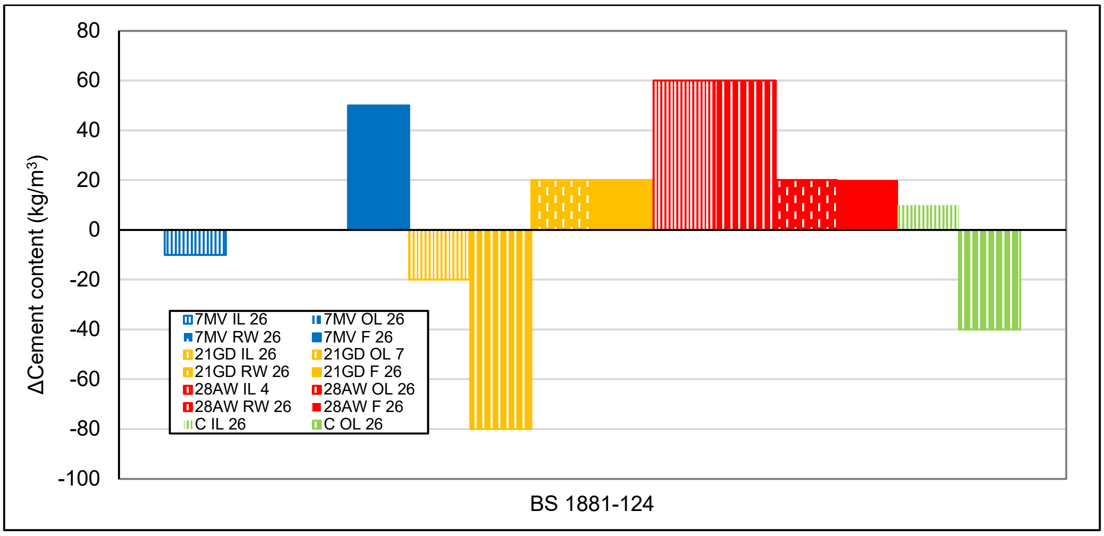
C RW did not exist and C F test was not conducted.

Figure 6-31 ΔCement content – RICS Stage 3



Elements where no test data was available are not presented. Results of 0 indicate no change in results for 7MV RW and 21GD IL.

Figure 6-32 ΔCement content – CSA P3 adapted

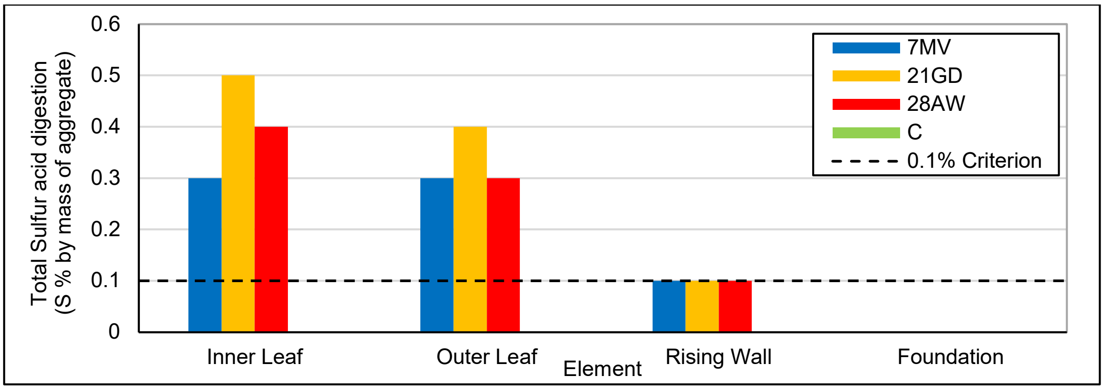


Elements where no test data was available are not presented. Results of 0 indicate no change in results for 7MV OL and RW. Numbers in legend indicate test exposure duration (weeks).

6.8.2 Total sulfur (TS)

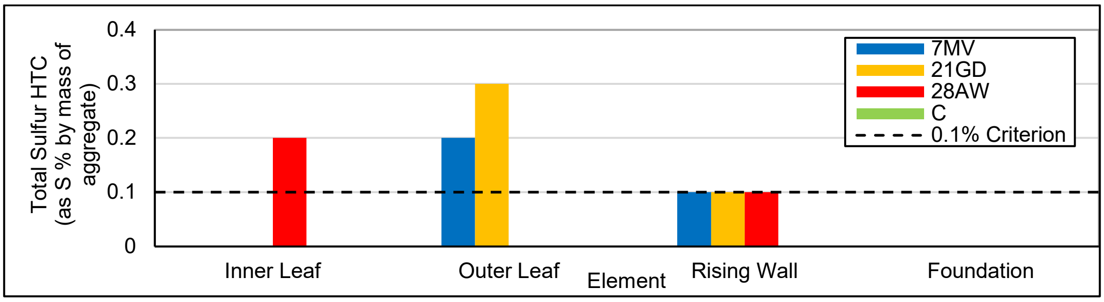
For more details on methodology and measurements please see sections **4.10** and **4.16** and **Appendix C**. All TS results presented have a 0.1 % S mass reduction applied to the data to account for the contribution of sulfur from the cement present within a typical standard Irish concrete block as noted within I.S.465:2018+A1:2020, E3.[34] A reduction for the foundation mass concrete TS values of 0.2 % mass S was similarly applied, calculated using a typical cement content of 14 % for general strip foundations.[35]

Figure 6-33 Total sulfur data (aggregate equivalent) – Acid digestion – RICS Stage 3



Samples tested in accordance with EN 1744-1:2009+A1:2012 Clause 11.1.[36] N=1. Note a 0.1 % S reduction has been applied to results determined for the whole concrete sample to account for the sulfur contributed by cement in a typical standard Irish concrete block as per I.S.465:2018+A1:2020.[37]. A 0.2 % S reduction has been applied in a similar method but for typical mass concrete foundations. The applied reduction has resulted in some 0.00 S % mass results, this does not indicate a lack of results apart from C Rising wall (non-existent) or C Foundation (no sample). The 0.1 % by dry mass of aggregate maximum total sulfur criterion for aggregates containing pyrrhotite is taken from EN 12620:2002+A1:2008[38].

Figure 6-34 Total sulfur data (aggregate equivalent) – Acid digestion – CSA P3 adapted



Samples tested in accordance with EN 1744-1:2009+A1:2012 Clause 11.1.[39] N=1. Note a 0.1 % S reduction has been applied to results determined for the whole concrete sample to account for the sulfur contributed by cement in a typical standard Irish concrete block as per I.S.465:2018+A1:2020.[40] A 0.2 % S reduction has been applied in a similar method but for typical mass concrete foundations. The applied reduction has resulted in some 0.0 S % mass results, this does not indicate a lack of results apart from C Rising wall (non-existent) or C Foundation (no sample). The 0.1 % by dry mass of aggregate maximum total sulfur criterion for aggregates containing pyrrhotite is taken from EN 12620:2002+A1:2008.[41]

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[34] I.S. 465:2018+A1:2020, See [2]

[35] NHBC Standards 2024, Site mixed concrete grade ST3

[36] BS EN 1744-1, See [23]

[37] I.S. 465:2018+A1:2020, See [2]

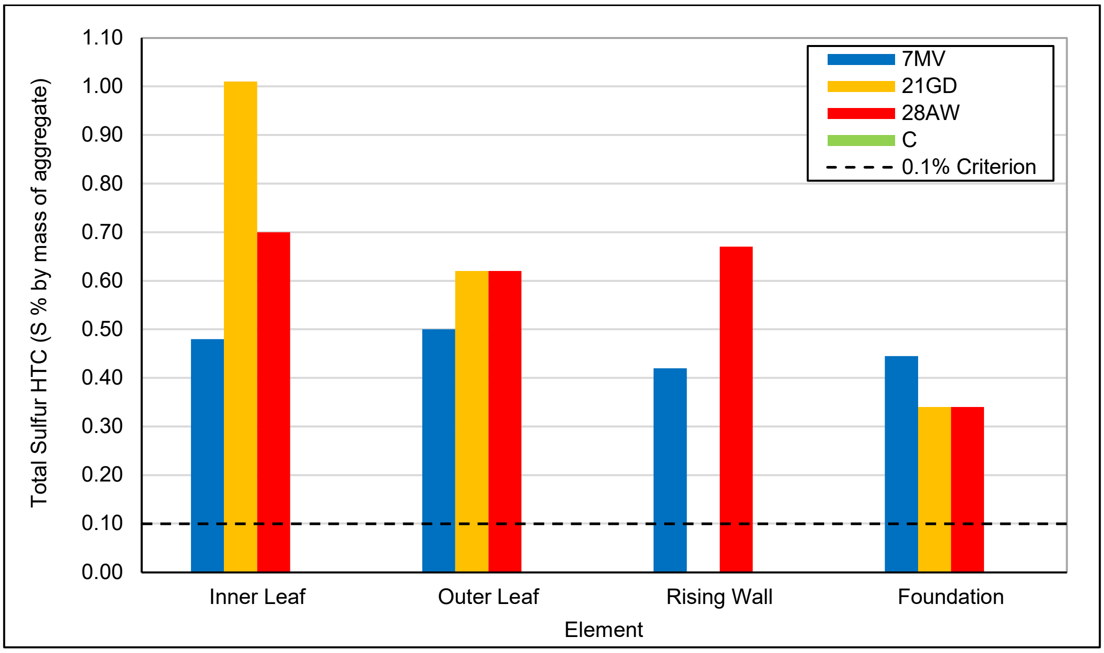
[38] EN 12620:2002+A1:2008 Aggregates for concrete. CEN, Brussels

[39] BS EN 1744-1, See [23]

[40] I.S. 465:2018+A1:2020, See [2]

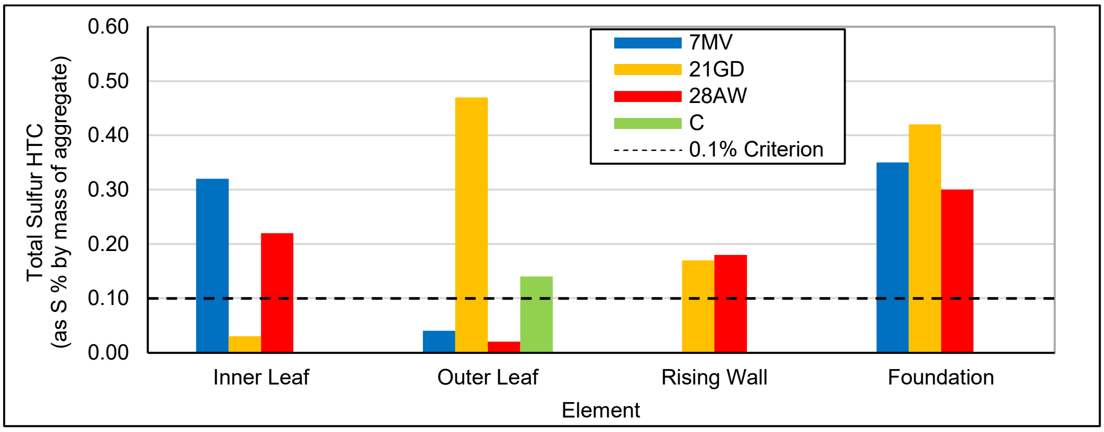
[41] EN 12620:2002+A1:2008 Aggregates for concrete. CEN, Brussels

Figure 6-35 Total sulfur data (aggregate equivalent) – HTC – RICS Stage 3



Samples tested in accordance with EN 1744-1:2009+A1:2012 Clause 11.2.[42] N=1. Note a 0.1 % S reduction has been applied to results determined for the whole concrete sample to account for the sulfur contributed by cement in a typical standard Irish concrete block as per I.S.465:2018+A1:2020.[43] A 0.2 % S reduction has been applied in a similar method but for typical mass concrete foundations. No 21GD RW, C OL, RW and F samples were tested. The 0.1 % by dry mass of aggregate maximum total sulfur criterion for aggregates containing pyrrhotite is taken from EN 12620:2002+A1:2008.[44]

Figure 6-36 Total sulfur data (aggregate equivalent) – HTC – CSA P3 Adapted



Samples tested in accordance with EN 1744-1:2009+A1:2012 Clause 11.2[45] N=1. Note a 0.1 % S reduction has been applied to results determined for the whole concrete sample to account for the sulfur contributed by cement in a typical standard Irish concrete block as per I.S.465:2018+A1:2020.[46] A 0.2 % S reduction has been applied in a similar method but for typical mass concrete foundations. Note that no C RW and F samples were tested. The 0.1 % by dry mass of aggregate criterion for aggregates containing pyrrhotite is taken from EN 12620:2002+A1:2008.[47]

[42] BS EN 1744-1, See [23]

[43] I.S. 465:2018+A1:2020, See [2]

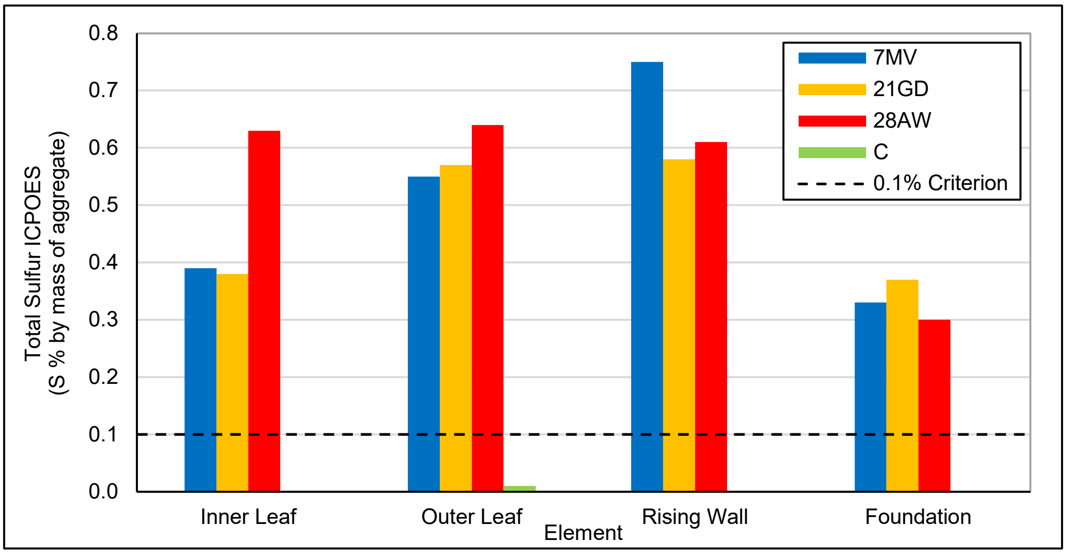
[44] EN 12620:2002+A1:2008 Aggregates for concrete. CEN, Brussels

[45] BS EN 1744-1, See [23]

[46] I.S. 465:2018+A1:2020, See [2]

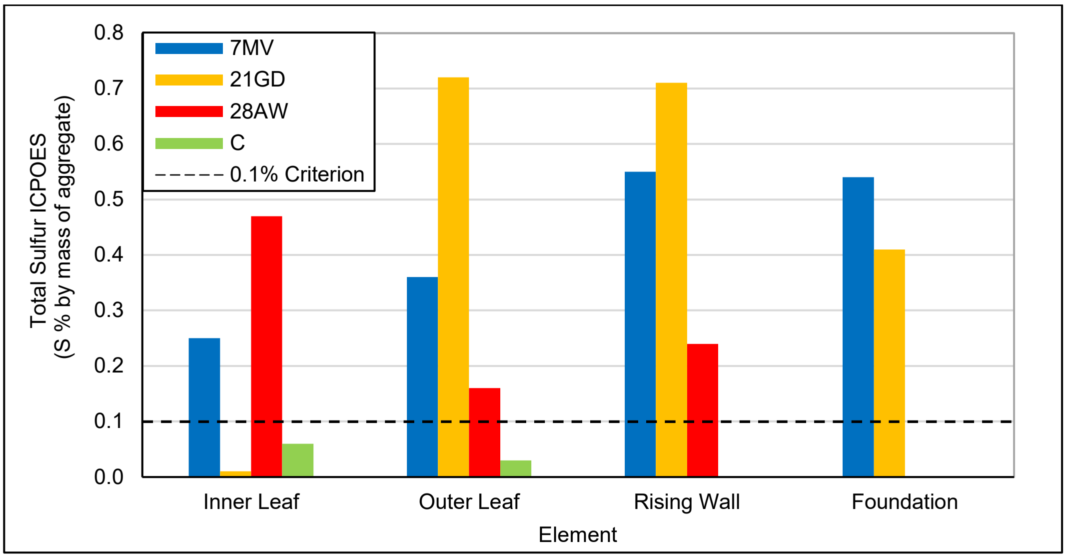
[47] EN 12620:2002+A1:2008 Aggregates for concrete. CEN, Brussels

Figure 6-37 Total sulfur data (aggregate equivalent) – ICP-OES – RICS Stage 3



Samples tested in accordance with BRE SD1 Brownfield Suite by ICP-OES.[48] N=1. Note a 0.1 % S reduction has been applied to results determined for the whole concrete sample to account for the sulfur contributed by cement in a typical standard Irish concrete block as per I.S.465:2018+A1:2020.[49] A 0.2 % S reduction has been applied in a similar method but for typical mass concrete foundations. Note that no C RW and F samples were tested. The 0.1 % by dry mass of aggregate criterion for aggregates containing pyrrhotite is taken from EN 12620:2002+A1:2008.[50]

Figure 6-38 Total sulfur data (aggregate equivalent) – ICP-OES – CSA P3 adapted



Samples tested in accordance with BRE SD1 Brownfield Suite by ICP-OES.[51] N=1. Note a 0.1 % S reduction has been applied to results determined for the whole concrete sample to account for the sulfur contributed by cement in a typical standard Irish concrete block as per I.S.465:2018+A1:2020.[52] A 0.2 % S reduction has been applied in a similar method but for typical mass concrete foundations. Note that no 28AW F or C, RW and F samples were tested. The 0.1 % by dry mass of aggregate criterion for aggregates containing pyrrhotite is taken from EN 12620:2002+A1:2008.[53]

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[48] BRE Special Digest 1: 2005, See [31]

[49] I.S. 465:2018+A1:2020, See [2]

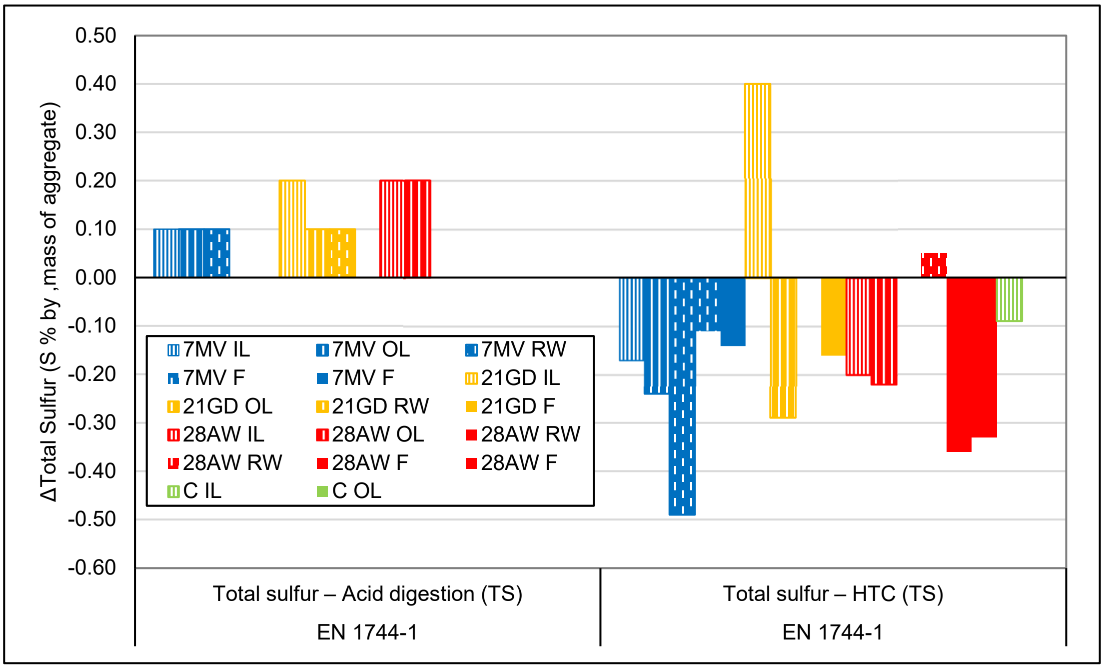
[50] EN 12620:2002+A1:2008 Aggregates for concrete. CEN, Brussels

[51] BRE Special Digest 1: 2005, See [31]

[52] I.S. 465:2018+A1:2020, See [2]

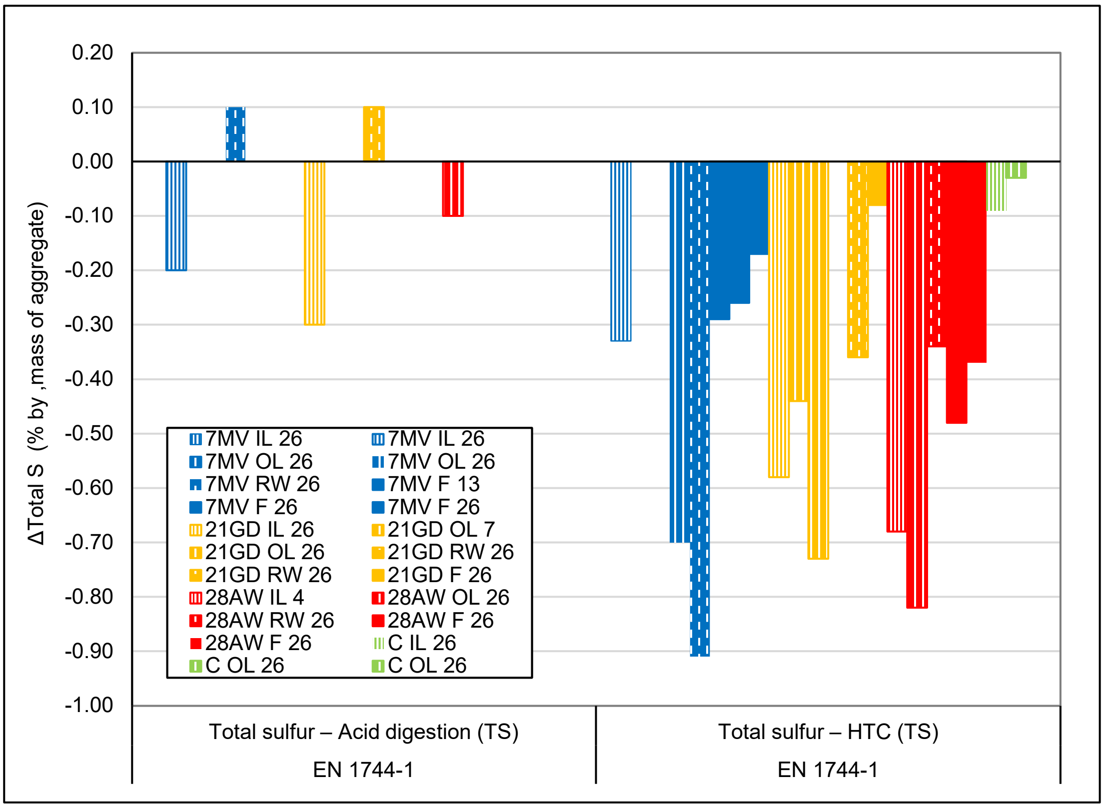
[53] EN 12620:2002+A1:2008 Aggregates for concrete. CEN, Brussels

Figure 6-39 ΔTotal S EN 1744-1 Acid digestion and HTC – RICS Stage 3



Elements where no test data was available are not presented unless they were tested in the alternate methodology. No testing for acid digestion, 7MV F and 28AW F (2nd samples) samples and HTC, 21GD RW, 28 AW RW (1st sample) and C OL samples. Results of 0 % indicate no change in results.

Figure 6-40 ΔTotal S EN 1744-1 Acid digestion and HTC – CSA

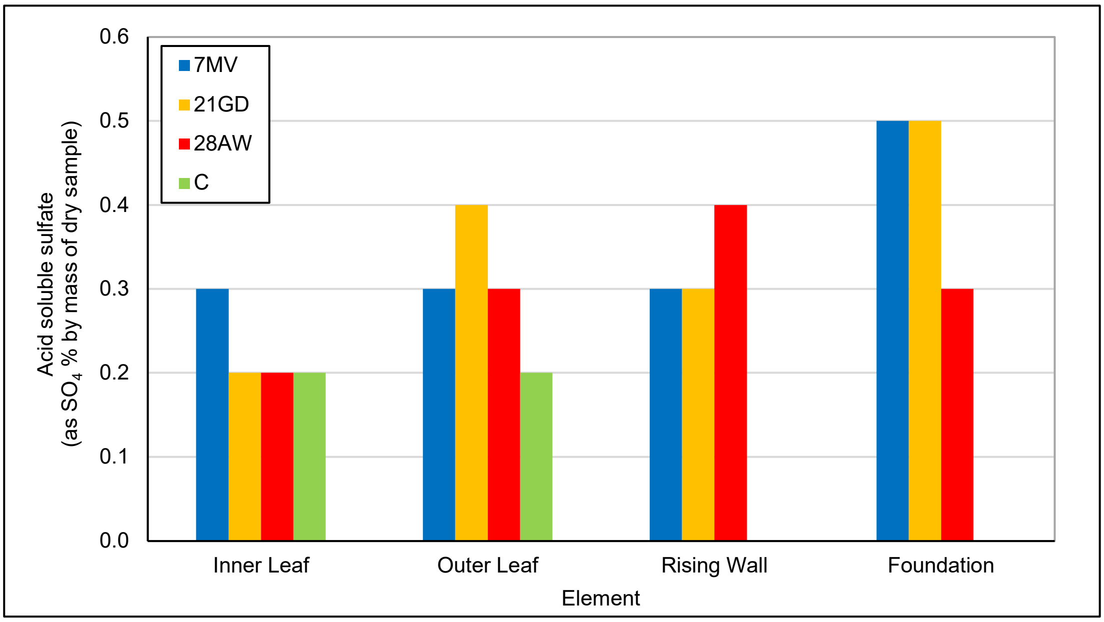


Elements where no test data was available are not presented. Results of 0 % indicate no change in results. No test for Acid digestion 7MV IL 1st sample, 7MV OL 2nd sample, 7MV F 1st and 2nd samples, 21GD OL 2nd sample, 21GD RW 2nd sample, 28AW F 1st sample and C OL 1st sample. Numbers in legend indicate test exposure duration.

6.8.3 Acid soluble sulfate (ASS)

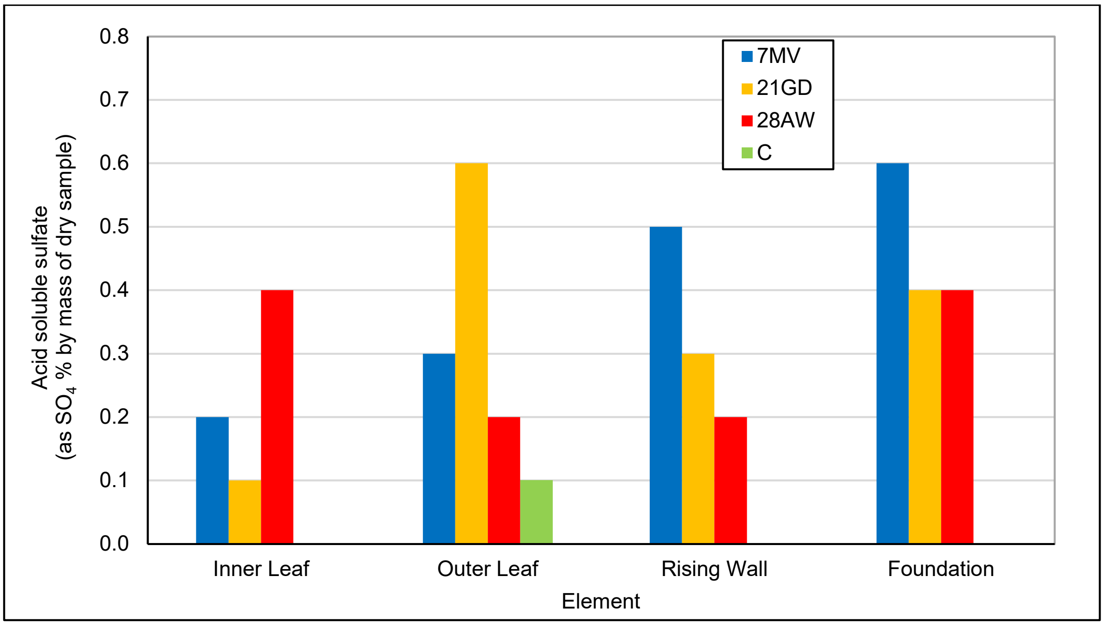
Results of the employed methods of acid-soluble sulfate content testing are presented. For more details on methodology and measurements please see **4.11**, **4.14**, **4.16** and **Appendix C**. Note that EN 1744-1 and EN 196-2 data have been converted from SO_3 to SO_4 (by multiplication by a factor of 1.2) to allow comparison to criteria given in the aggregate standards.

Figure 6-41 Acid soluble sulfate data – EN 1744-1 – RICS Stage 3



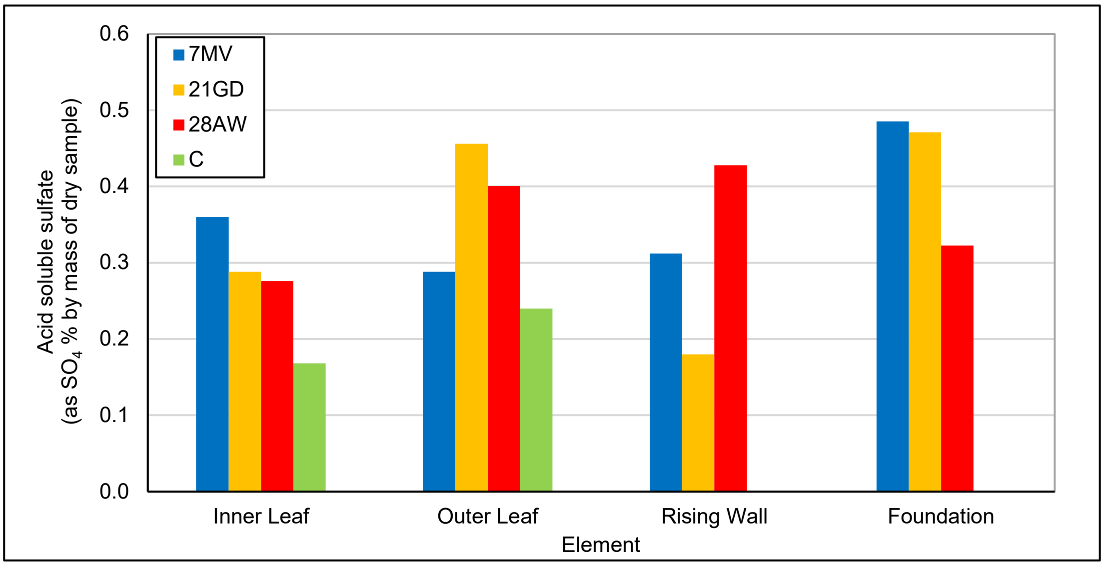
No C RW sample existed or C F sample tested.

Figure 6-42 Acid soluble sulfate data – EN 1744-1 – CSA P3 adapted



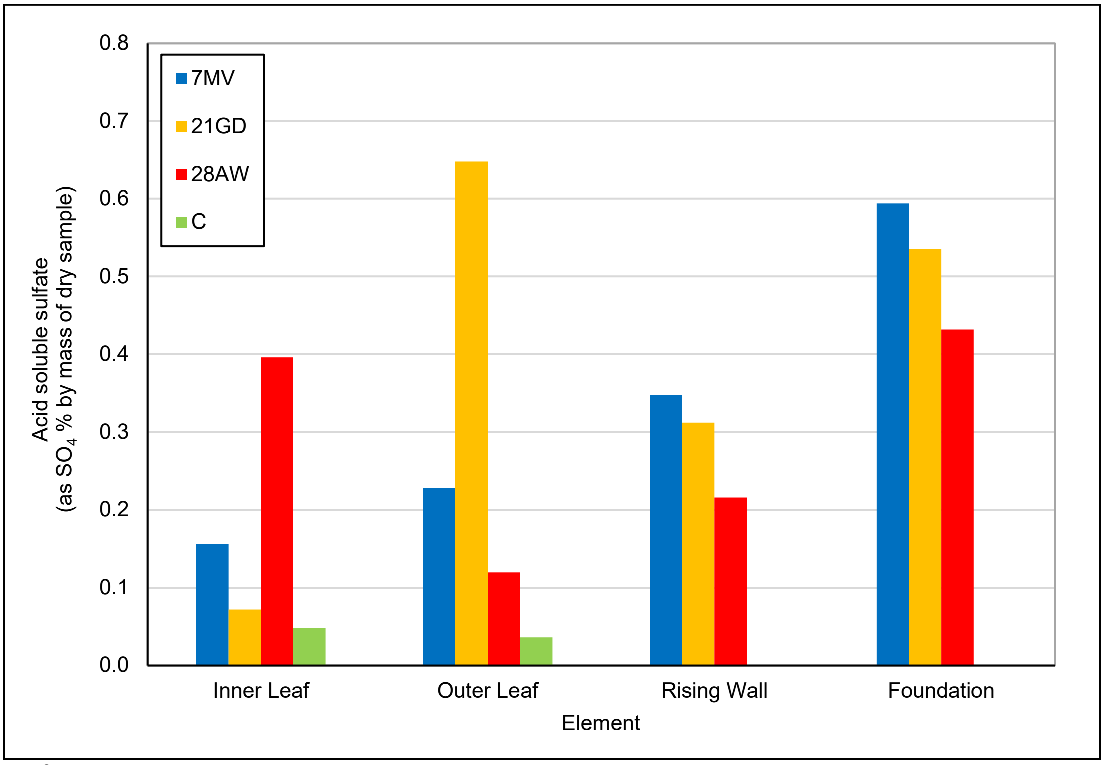
No C RW sample existed or C F sample tested.

Figure 6-43 Acid soluble sulfate data – EN 196-2 – RICS Stage 3



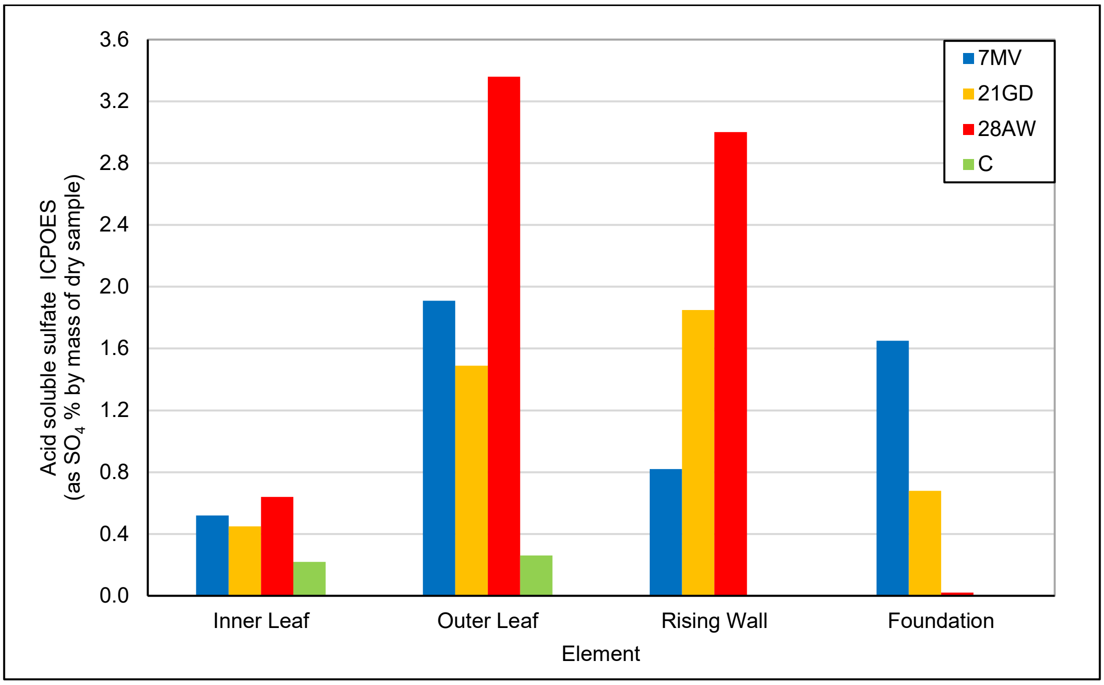
No C RW or F samples were tested.

Figure 6-44 Acid soluble sulfate data – EN 196-2 – CSA P3 adapted



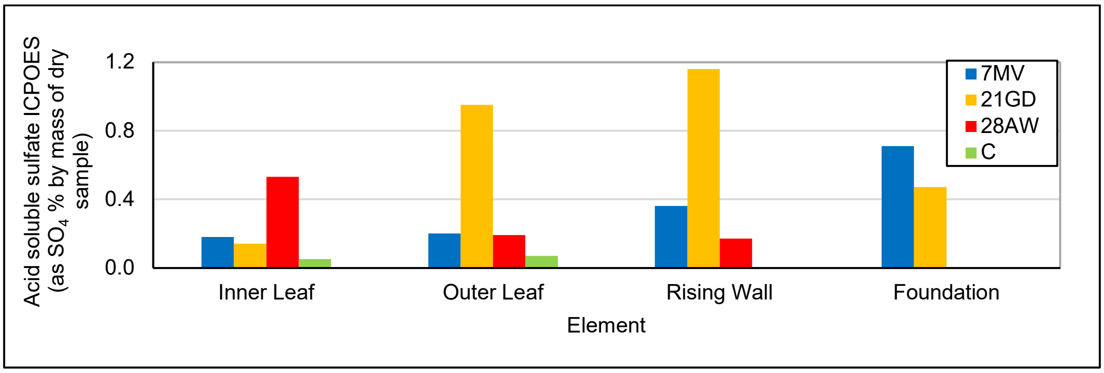
No C RW and F samples were tested.

Figure 6-45 Acid soluble sulfate data – ICP-OES – RICS Stage 3



No C RW and F samples were tested.[54]

Figure 6-46 Acid soluble sulfate data – ICP-OES – CSA P3 adapted

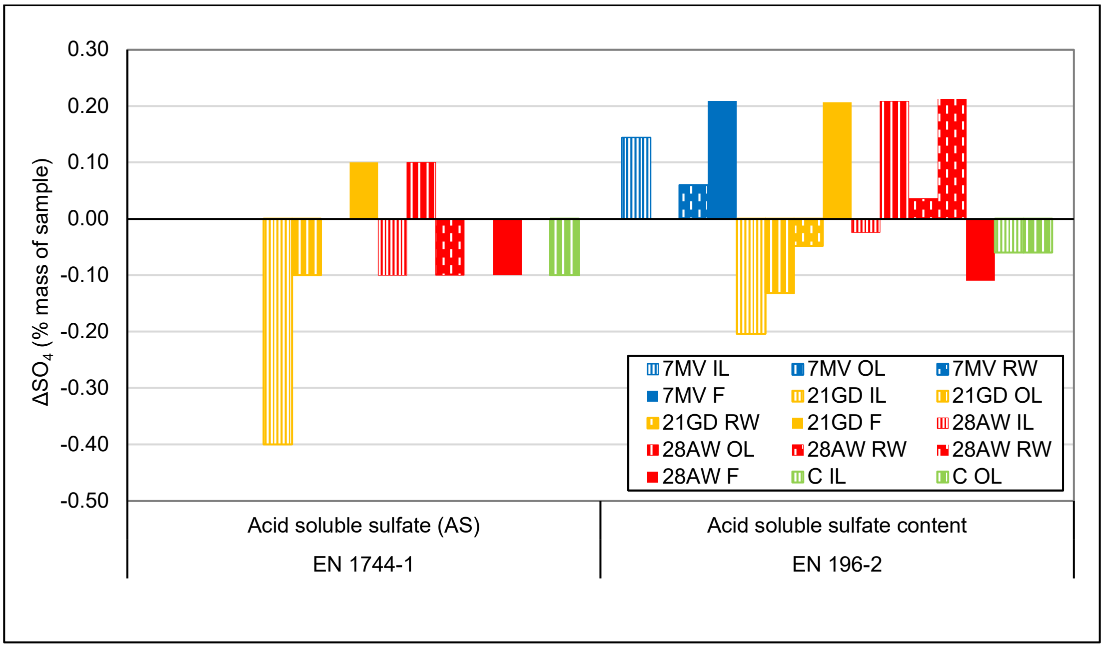


No 28AW F or C RW and F samples were tested.[55]

[54] BRE Special Digest 1: 2005, See [31]

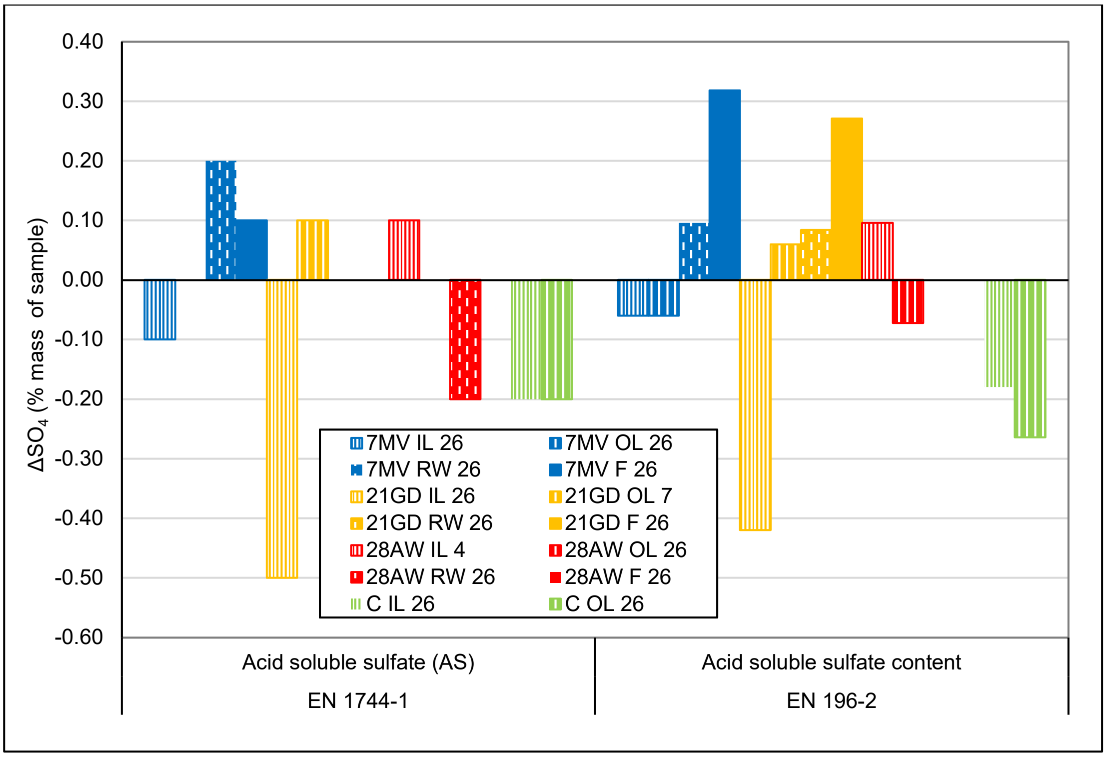
[55] BRE Special Digest 1: 2005, See [31]

Figure 6-47 ΔAcid soluble sulfate content – EN 1744-1 and EN 196-2 – RICS Stage 3



Elements where no test data was available are not presented. Results of 0 % indicate no change in results.

Figure 6-48 ΔAcid soluble sulfate content – EN 1744-1 and EN 196-2 – CSA P3 adapted

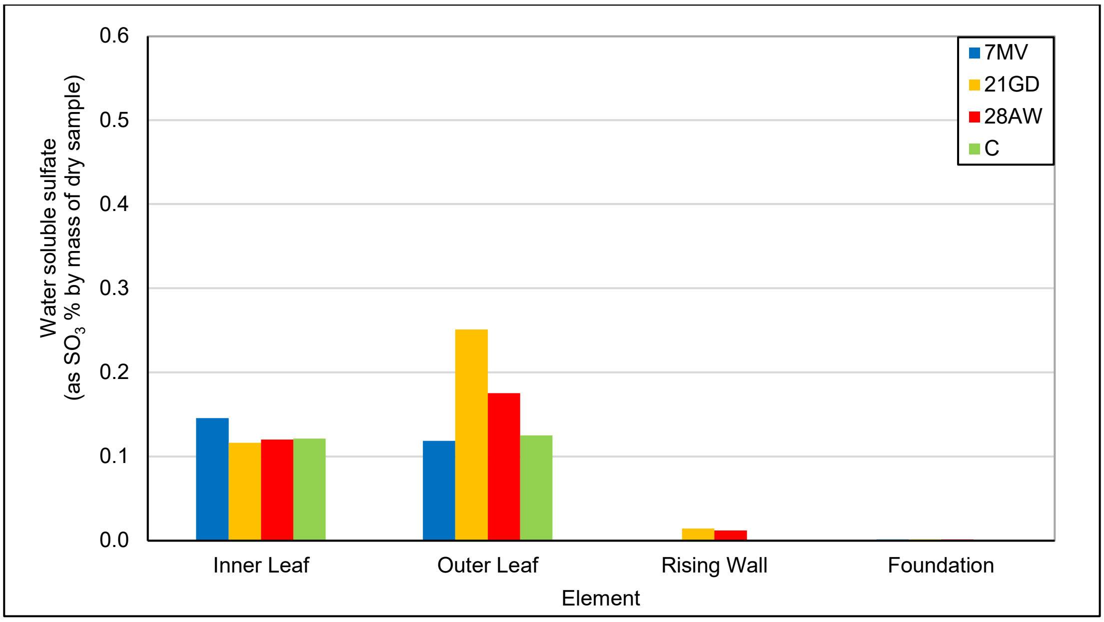


Elements where no test data was available are not presented. Results of 0 % indicate no change in results. Numbers in legend represent TTF.

6.8.4 Water soluble sulfate content (WSS)

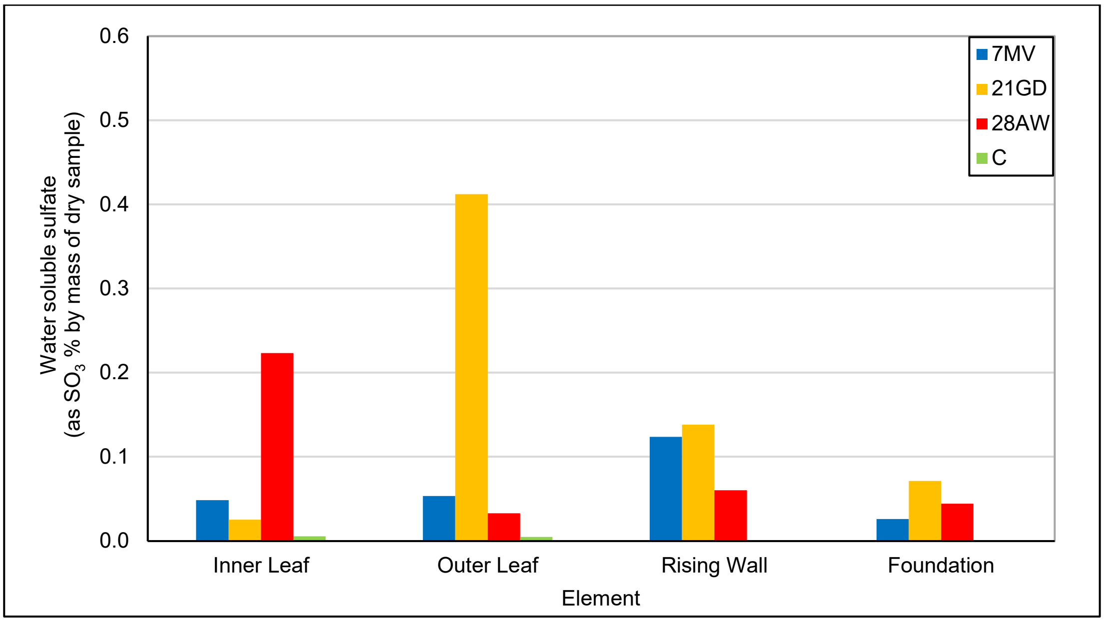
Results of the employed methods of water-soluble sulfate content testing are presented. For more details on methodology and measurements please see **4.12** and **4.16** and **Appendix C**.

Figure 6-49 Water soluble sulfate data – EN 1744-1 – RICS Stage 3



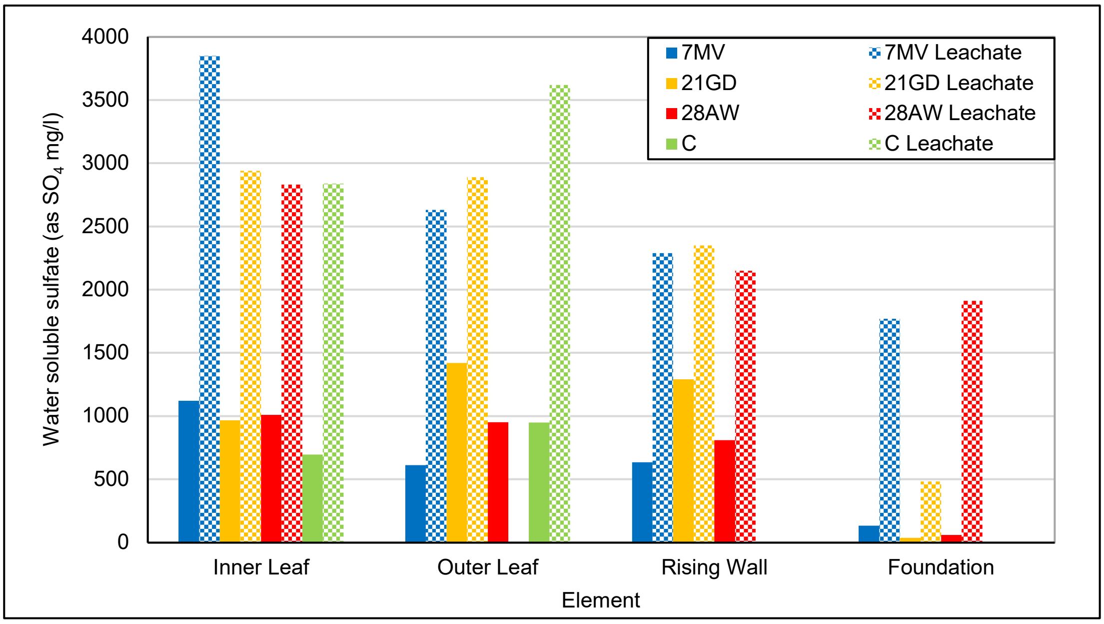
No test for 7MV RW, C RW, C F.

Figure 6-50 Water soluble sulfate data – EN 1744-1 – CSA P3 adapted



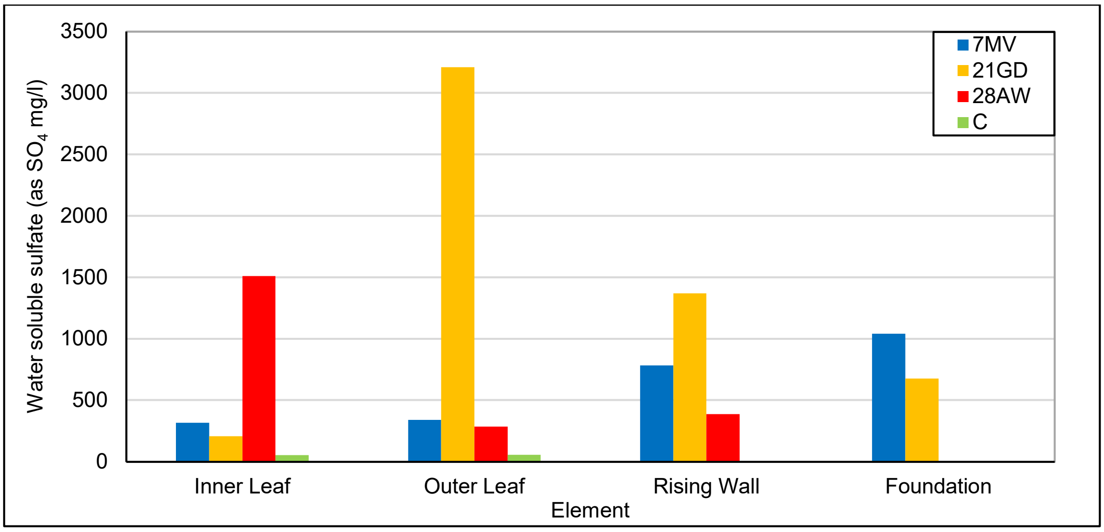
No test for C RW or C F.

Figure 6-51 Water soluble sulfate data – Colorimetry – RICS Stage 3



Both crushed concrete (solid) and leachate (check) results are presented.[56] 28AW OL Leachate no data. No C RW or F tests were conducted. Note that the results are presented in mg/l so that leachate (water) samples are comparable.

Figure 6-52 Water soluble sulfate data – Colorimetry – CSA P3 adapted

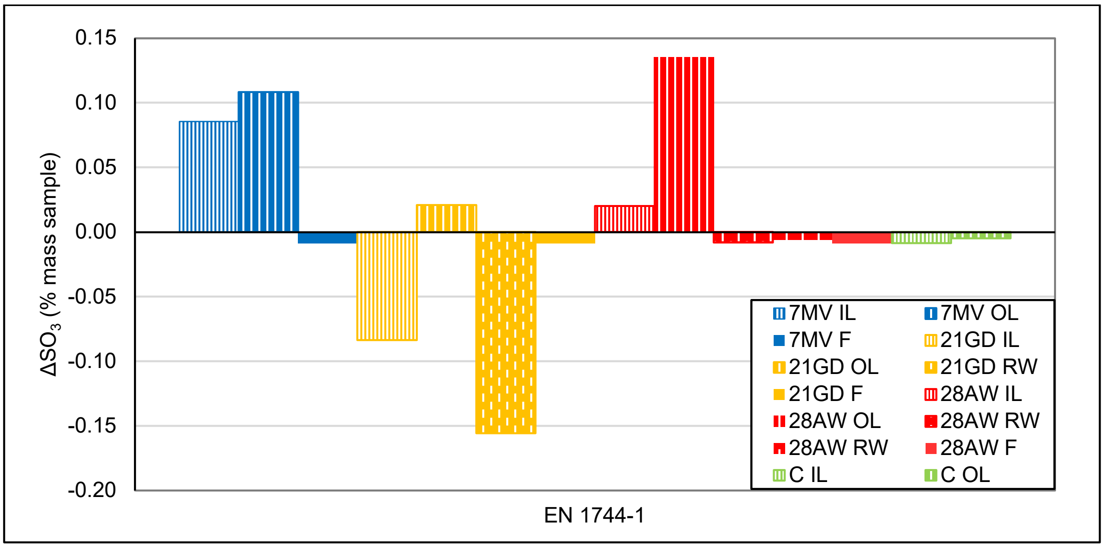


No 28AW F and C RW or F tests were conducted.[57]

[56] BRE Special Digest 1: 2005, See [31]

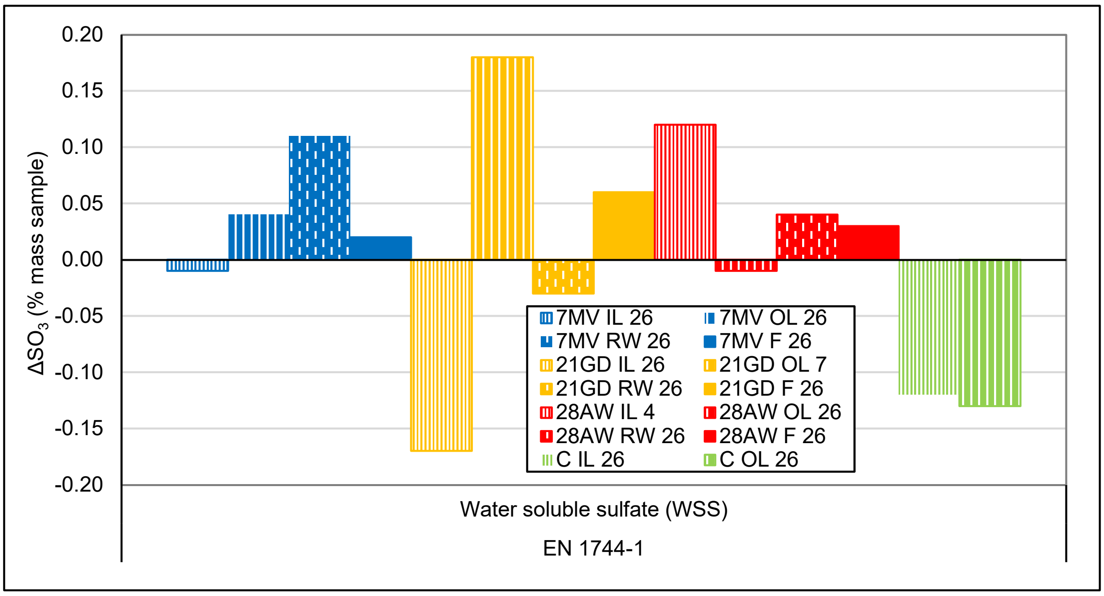
[57] BRE Special Digest 1: 2005, See [31]

Figure 6-53 ΔWater soluble sulfate data – EN 1744-1 – RICS Stage 3



Elements where no test data was available are not presented.

Figure 6-54 ΔWater soluble sulfate data – EN 1744-1 – CSA P3 adapted

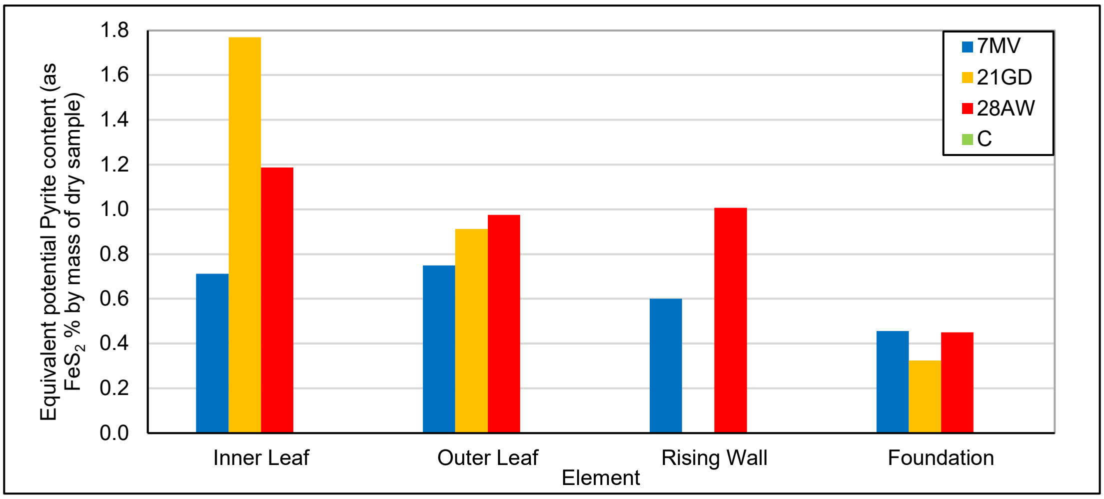


Elements where no test data was available are not presented. Numbers in legend represent TTF.

6.8.5 Pyrite and pyrrhotite content

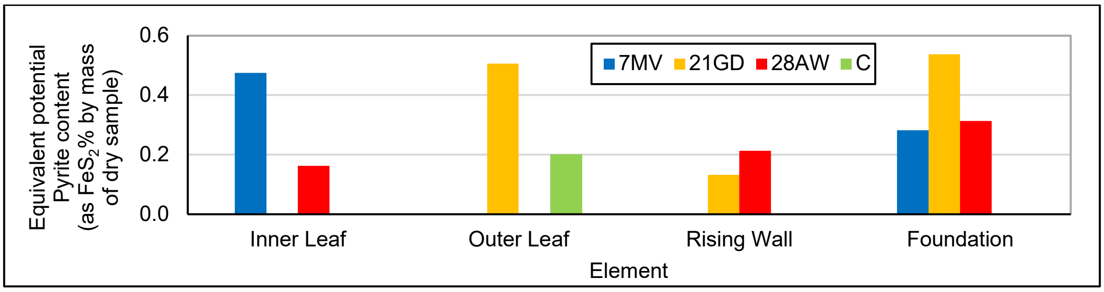
The calculated maximum possible pyrite and pyrrhotite content represent values assuming all the oxidisable sulfides present were purely one mineral species. In this case, we know this not to be the case, as petrographic examinations have identified all of pyrrhotite, pyrite and chalcopyrite in some samples, but values are provided here for relative comparison. The values presented are calculated using TS (HTC) and ASS determined values (**Table 6-12** and **Table 6-13**). By calculating total potential sulfate (TPS, 3 × TS) and subtracting ASS, oxidisable sulfides (OS) as SO_4 is derived. OS can then be converted into the sulfide mineral species (i.e. pyrrhotite or pyrite) by simple conversion of molecular masses. The values presented have been calculated using the total sulfur values that account for the contribution of sulfur from the cement, as opposed to the calculated values presented within **Appendix C**. Note that only samples where TPS is more than the value of ASS will allow calculation of any OS by this method and with any samples where OS is calculated to be less than zero being represented as zero results below[58].

Figure 6-55 Calculated pyrite content data – RICS Stage 3



Based on calculations as described in TRL 447.[59] N=1. The results represent the maximum possible mineral content based on TS (HTC) and ASS results assuming all sulfides are pyrite of FeS_2 composition. No results for 21GD RW or C OL, RW or F calculated. ASS substituted from similar element sub-samples where necessary.

Figure 6-56 Calculated pyrite content data – CSA P3 adapted

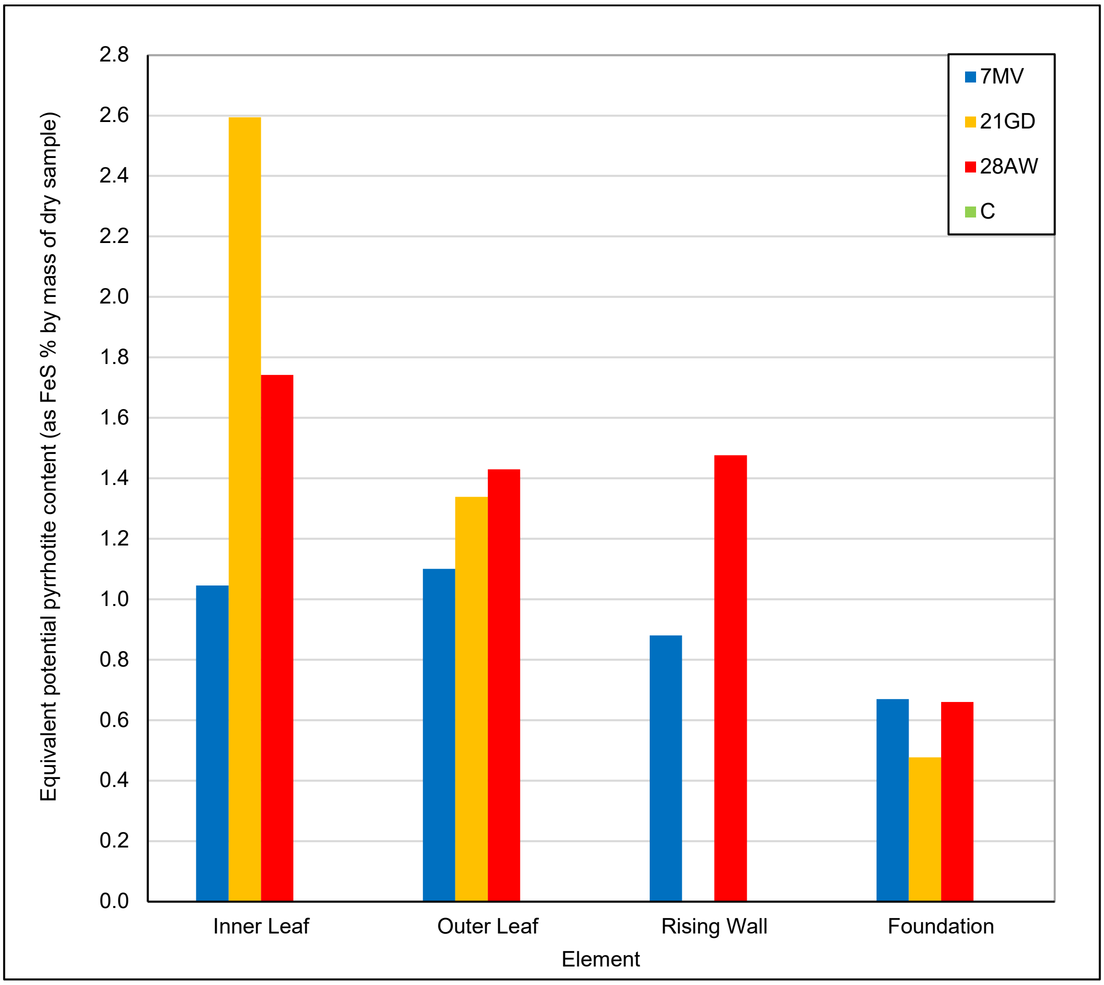


[58] As the standard methods used to determine TS and ASS are different, this occasionally occurs in instances where the sulfur is essentially all oxidized, when the difference between the calculated values is less than the analytical error in the methods.

[59] TRL Report TRL447. See [27]

Based on calculations as described in TRL 447.[60] N=1. The results represent the maximum possible mineral content based on TS (HTC) and ASS results assuming all sulfides are pyrite of FeS_2 composition. Zero values, 7MV OL, RW, 21GD IL, 28AW OL, C IL No results for C RW or F calculated. ASS substituted from similar element sub-samples where necessary

Figure 6-57 Calculated pyrrhotite content data – RICS Stage 3

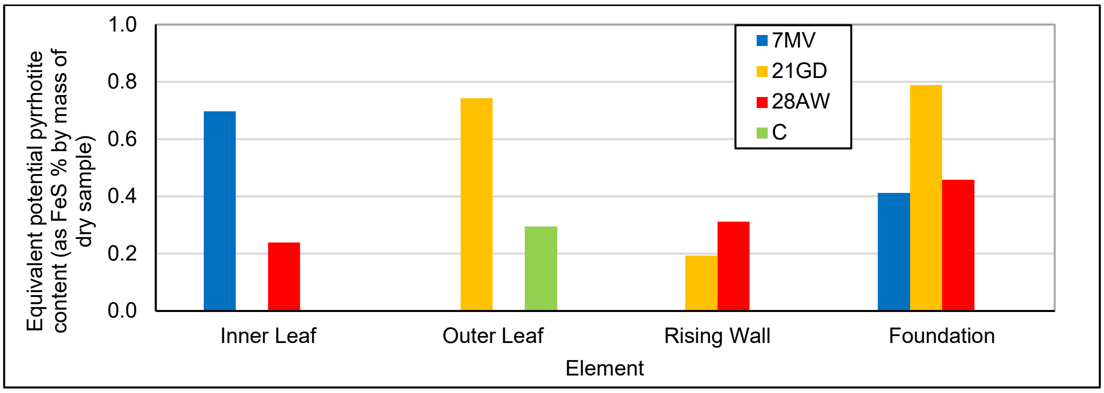


Based on calculations as described in TRL 447.[61] N=1. The results represent the maximum possible mineral content based on TS (HTC) and ASS results assuming all sulfides are pyrrhotite of FeS composition. No results for 21GD RW or C OL, RW or F calculated. ASS substituted from similar element sub-samples where necessary.

[60] TRL Report TRL447. See [27]

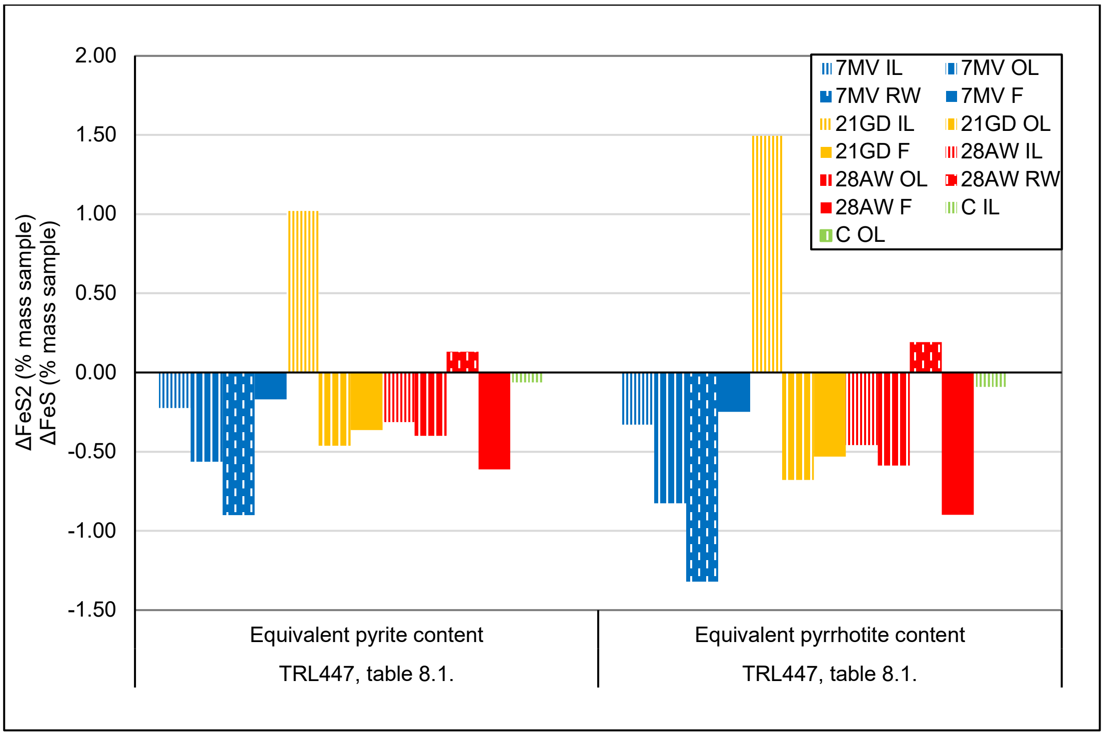
[61] TRL Report TRL447. See [27]

Figure 6-58 Calculated pyrrhotite content data – CSA P3 adapted



Based on calculations as described in TRL 447.[62] N=1. The results represent the maximum possible mineral content based on TS (HTC) and ASS results assuming all sulfides are pyrrhotite of FeS composition. Zero values, 7MV OL, RW, 21GD IL, 28AW OL, C IL No results for C RW or F calculated. ASS substituted from similar element sub-samples where necessary.

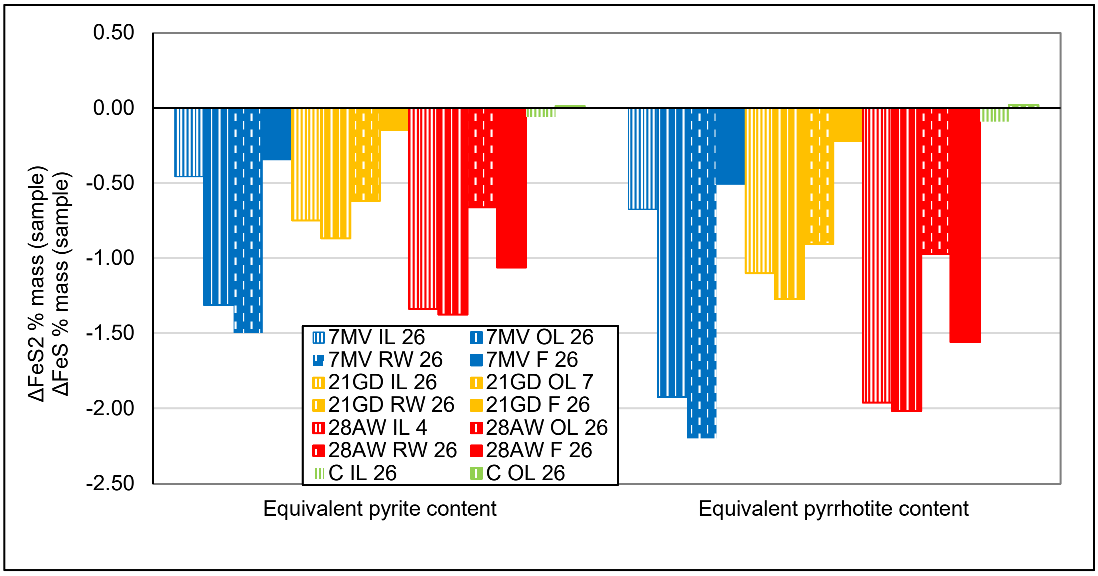
Figure 6-59 ΔCalculated pyrite and pyrrhotite content data – RICS Stage 3



Elements where no test data was available are not presented.

[62] TRL Report TRL447. See [27]

Figure 6-60 ΔCalculated pyrite and pyrrhotite content data – CSA P3 adapted

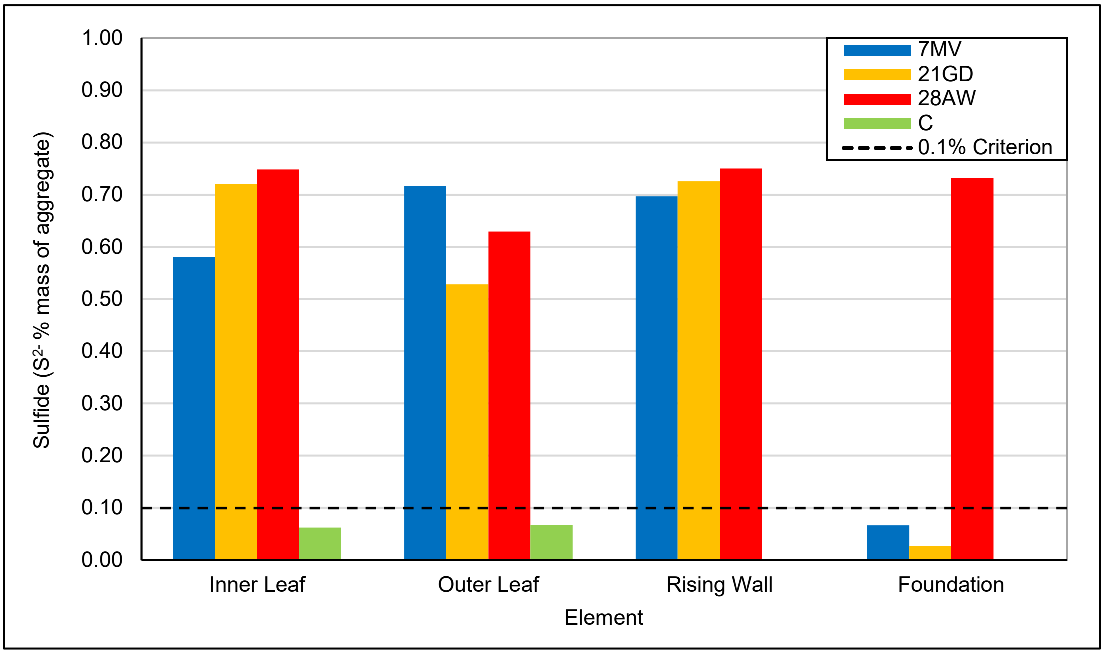


Elements where no test data was available are not presented. Numbers in legend represent TTF.

6.8.6 Sulfide content

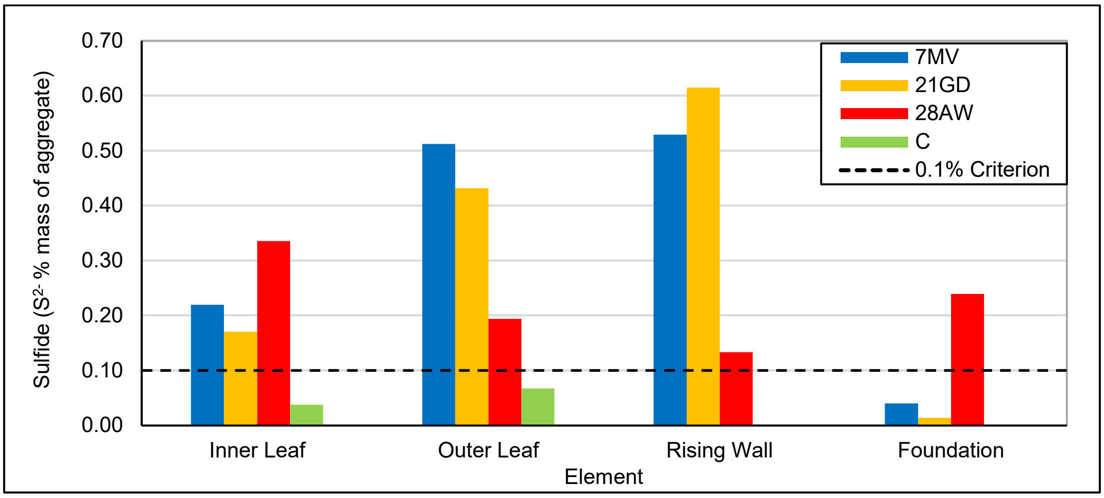
The results of sulfide content testing are presented. For more details on methodology and measurements please see **4.15** and **Appendix C**.

Figure 6-61 Sulfide content data – EN 196-2 – RICS Stage 3



Samples tested in accordance with BS EN 196-2:2013.[63] N=1. No C RW and F tests were conducted. Note that a conversion factor based on sample density was applied to the results to represent the aggregate rather than the whole sample, see Appendix C.

Figure 6-62 Sulfide content data – EN 196-2 – CSA P3 adapted

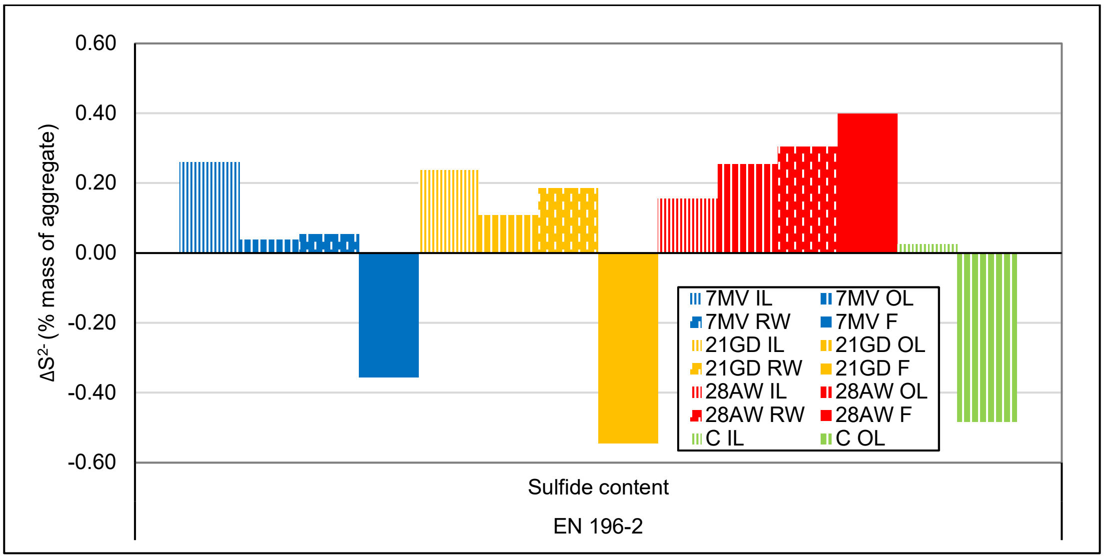


Samples tested in accordance with BS EN 196-2:2013.[64] N=1.No C RW or F tests were conducted. Note that a conversion factor based on sample density was applied to the results to represent the aggregate rather than the whole sample, see Appendix C.

[63] BS EN 196-2, See [28]

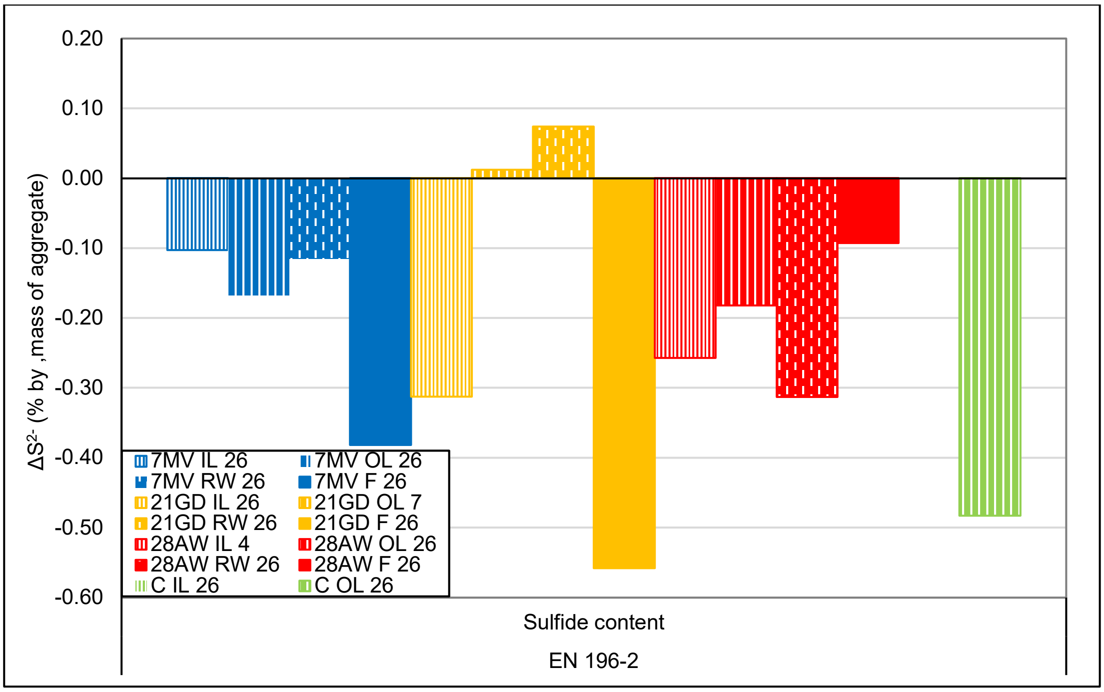
[64] BS EN 196-2, See [28]

Figure 6-63 ΔSulfide content – EN 196-2 – RICS Stage 3



Elements where no test data was available are not presented. Numbers in legend represent TTF.

Figure 6-64 ΔSulfide content – EN 196-2 – CSA P3 adapted

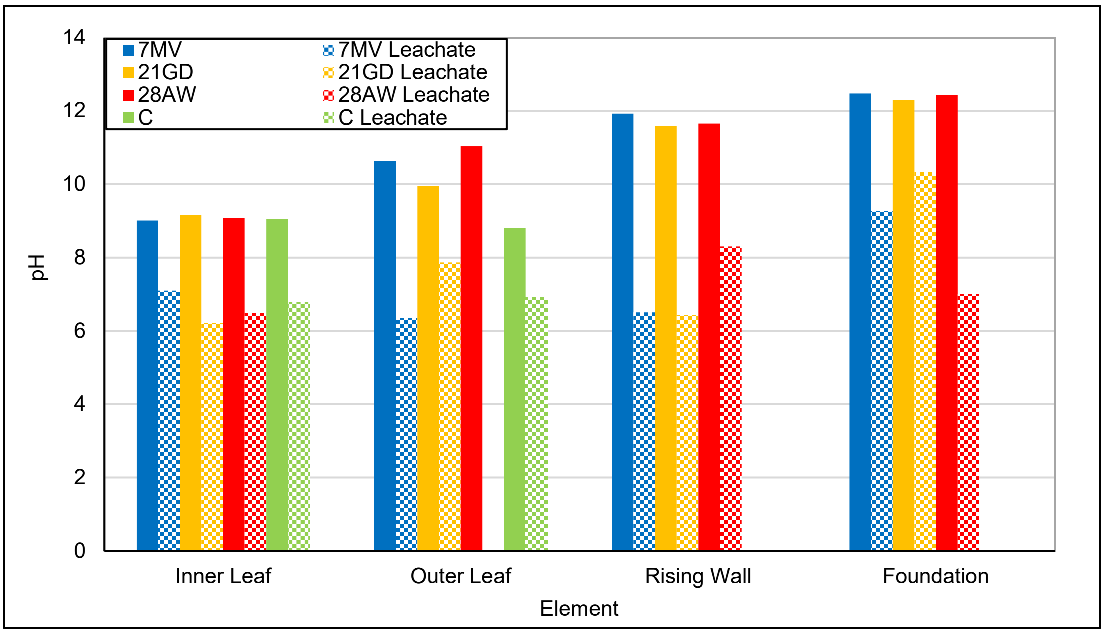


Elements where no test data was available are not presented. 0 % results represent no change.

6.8.7 pH

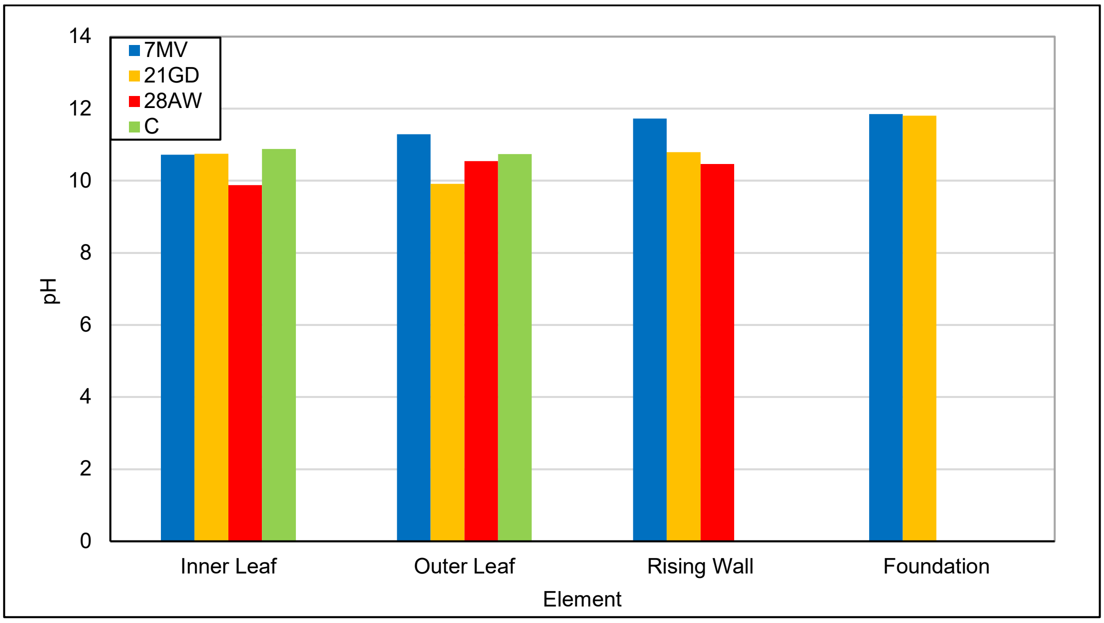
The results of sulfide content testing are presented. For more details on methodology and measurements please see **4.15** and **Appendix C**.

Figure 6-65 pH – RICS Stage 3 – concrete and leachate samples



Both crushed concrete (solid) and leachate (check) results are presented.[65]. No 28AW OL (leachate), C RW or F tests were conducted.

Figure 6-66 pH – CSA P3 adapted – concrete samples



No 28AW F and C RW or F tests were conducted.[66]

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[65] BRE Special Digest 1: 2005, See [31]

[66] BRE Special Digest 1: 2005, See [31]

7 DISCUSSION

The objectives of the following discussion are to evaluate the relative performance of the investigated building elements during Stage 2 durability testing and the subsequently determined Stage 3 testing results.

Discussion of Phase 1 results is presented in RSK report 1283831-01 for all elements and discussed again in 1283831-03 for the investigated foundations.

Phase 3 discussion of foundations is presented in RSK report 1283831-03 and is therefore not discussed in detail below but is chiefly referred to for relative comparison only with regards to the performance of mass concrete relative to blockwork concrete and to the differing environments of use.

A relatively brief discussion of the test properties Phase 1 condition is provided as a brief reference for comparison.

7.1 Phase 1 – Initial Characterisation

The three test properties that exhibited evidence of damage to the superstructural blockwork walls are understood to all have been constructed between circa 1998 and 2008, with the 'control' property 'Carrowmore' being constructed during the 1980s. It is understood that the design life of a residential dwelling in the Republic of Ireland is specified to be 50 years, although clearly many houses will comfortably exceed this; suitable, adequately produced and built concrete blockwork would be required to perform acceptably for this duration (under normal circumstances and subject to no significant changes in the local environment) with no maintenance for this duration. As such, the test properties, are 1/4 and 1/3 of the way through their design life and exhibit significant failures in certain elements.

The control Carrowmore property, at approximately 40 years from construction is beyond the mid-point of its service life; the visual examination suggests this is a relatively poor-quality concrete, however, it did not exhibit significant deterioration, sulfide oxidisation, or any ISA, even though it contains iron sulfides including rare traces of pyrrhotite and some potentially reactive framboidal pyrite.

The test property concrete blockwork and mass concrete all exhibited a similar composition, with sulfide-bearing (pyrrhotite, pyrite and chalcopyrite) PHY aggregates bound by a CEM I binder. The sulfide content and oxidisation with subsequent varied secondary sulfate mineralisation (including secondary ettringite, gypsum and thaumasite), as-received condition and other deterioration factors were found to be generally worse in the test property concrete blockwork than in the foundation mass concrete, particularly the outer and inner leaf blocks. It was clear that the concrete blockwork composition has resulted in damaging localised internal sulfate attack (ISA) reactions primarily in the outer leaf, particularly where exposed to moisture, the inner leaf when moisture had penetrated, but less so within the rising wall where more permanently saturated conditions had restricted ISA to some degree. Even though high 'free' muscovite mica content (13-40% of binder volume) was observed within the test properties, this was deemed a passive rather than active contributory factor in the observed concrete deterioration.

In other instances of internal sulfate attack on concrete associated with pyrrhotite oxidisation in aggregate, the affected concrete has typically been in a setting with the construction that is subject to wetting and drying cycles and the presence of atmospheric oxygen. For both the cases in Quebec and Connecticut, the most vulnerable concrete elements have been in a zone from immediately below ground level up to approximately 0.5 m above ground level. In Quebec, the concrete in this zone, when constructed using the most reactive aggregates, generally exhibited damage by cracking visible as the concrete surface within the first 3 to 5 years after construction (and there have been very few cases where significant damage has started to manifest after more than about 5 years).[67]

However, in the cases in Connecticut, the rate of development of damage was significantly more variable and in many cases slower, with obvious cracking to the concrete occurring for anything between about 5 and 35 years after construction.

In this case, the concrete blockwork is vulnerable to wetting and drying cycles, particularly in the outer leaf and exposed rising walls. This has been observed to have progressed to inner leaves where the outer leaves and external render have failed.

Additionally, the vulnerability of the outer leaf blocks may be influenced by the performance of the render on the outer face, wherein potentially susceptible blocks appear to perform adequately while the render is robust, but may deteriorate rapidly once the render ceases to offer adequate protection.[68]

7.2 Phase 2 – Accelerated Durability Tests

It should be noted that both the conducted RICS Mundic Stage 3 expansion and CSA P3 adapted mortar bar tests were conducted on samples that in many cases had already undergone some degree of deterioration. Therefore it follows that changes in both determined values and properties, and tests performance are relative to their previous condition at the time of sampling, rather than their ‘as new’ condition.

7.2.1 RICS The Mundic Problem Stage 3 expansion test

The RICS Mundic Stage 3 expansion is specifically designed to test precast concrete masonry blocks and their sensitivity to moist conditions needed for ISA. However as previously noted the interpretations strictly apply to the areas of southwest England specified within the testing procedure, as such the results are cautiously interpreted in terms of relative performance, particularly against the control property.

The Mundic Stage 3 expansion testing includes a cut-off of 0.025% average expansion over the period between 7 days and 250 days (or 350 days as in this case), with concrete falling below this being classed as Class A_3 (stable group 2 aggregate). All the test property IL and OL concrete block samples subject to this test exceeded this criterion apart from 7MV IL (see **6.1**), suggesting that the PHY aggregates when present within

[67] The reviewer has acted as an Expert Witness in relation to the major pyrrhotite-concrete case in Trois Rivieres, and has undertaken extensive reviews of documentation in support of various of the legal claims.

[68] The reviewer has undertaken investigations elsewhere in Ireland where blocks made with reactive pyrite-bearing aggregates have undergone localised deterioration associated with cracks within the render, where adjacent blocks of similar composition but adequately protected by render have shown no deterioration, noting that in many instances the failure or worsening condition of the render may be associated with the block deterioration behind this.

concrete blocks were of a composition that could potentially cause expansion and that the investigated test property OL and IL samples still contained enough reactive sulfides to cause significant expansion through the RICS Mundic Stage 3 test.

The test property RW and F concrete blockwork samples and the control property IL and OL samples did not exceed the 0.025 % average expansion criteria, indicating no potential future expansion of the concrete blockwork in warm moist conditions containing the SST aggregate, PHY-bearing mass concrete or permanently saturated PHY bearing concrete blocks. However, the test property RWs were known to have significant increases in factors indicative of further ISA related deterioration having occurred during the RICS Stage 3 testing. Based on the author's experience and that of ISA-related deterioration of concrete building elements in Connecticut,[69] it may be that the particular type of ISA occurring within the sample requires a significantly longer time to develop in the RICS Stage 3 test conditions than 350 days.

All Mundic Stage 3 expansion test sub-sample sets returned generally consistently increasing mean mass % change that plateaued after approximately 50 to 200 days from original readings (see **Figure 6-6**). A few instances of sudden drops in mass were associated with material spalling from sub-samples during consistent expansion.

Leaching of the samples during the RICS Stage 3 test conditions (as per WSS of leachate) may have limited the progression of any ISA-related secondary mineral precipitation and associated cracking. In this instance, the cement matrix will weaken, ultimately to the point of failure of the sample concrete.

The test appeared to be suitable for application to the investigated concrete blockwork samples, with only 1 of 43 core sub-samples considered a failure of the test procedure, noted due to mean shrinkage (this result being disregarded from mean expansion calculations). It should be noted that two main criticisms of the testing could be made. Firstly, ideally, four sub-samples should have been tested for each element rather than the one to three sub-samples tested where sampling or test programme design precluded more sub-sample provision. Secondly, all sub-samples would have been tested for the same length of time i.e. 350 days rather than 250 days for consistent comparison, however in this case the 250 days exposure for 28AW OL already exceeded the 0.025% average expansion precluding the need to extend the testing length.

7.2.2 CSA A23.1 P3 Oxidisation test

The Canadian Standard oxidisation mortar bar test (AMBT) is run over two phases, with the first phase intended to promote accelerated oxidisation of any potentially reactive iron sulfide minerals, and the second phase to promote expansion if the concrete is susceptible to damaging thaumasite attack. The testing protocol CSA A23.1 P11.6[70] for the quality control of sulfide-bearing aggregates suggests an expansion difference limit for the CSA A23.1 P3 Oxidisation AMBT test[71] between mean expansion after 90 days (P3 Phase 1) and the stable mean expansion of the next 90 days (P3 Phase 2) of 0.10 %. The P3 Phase 2 mean expansion criterion (See **Figure 7-1**) is exceeded by cores in 7MV IL, 21GD IL, 7MV OL, 7MV RW and 21GD RW. However, a large proportion of

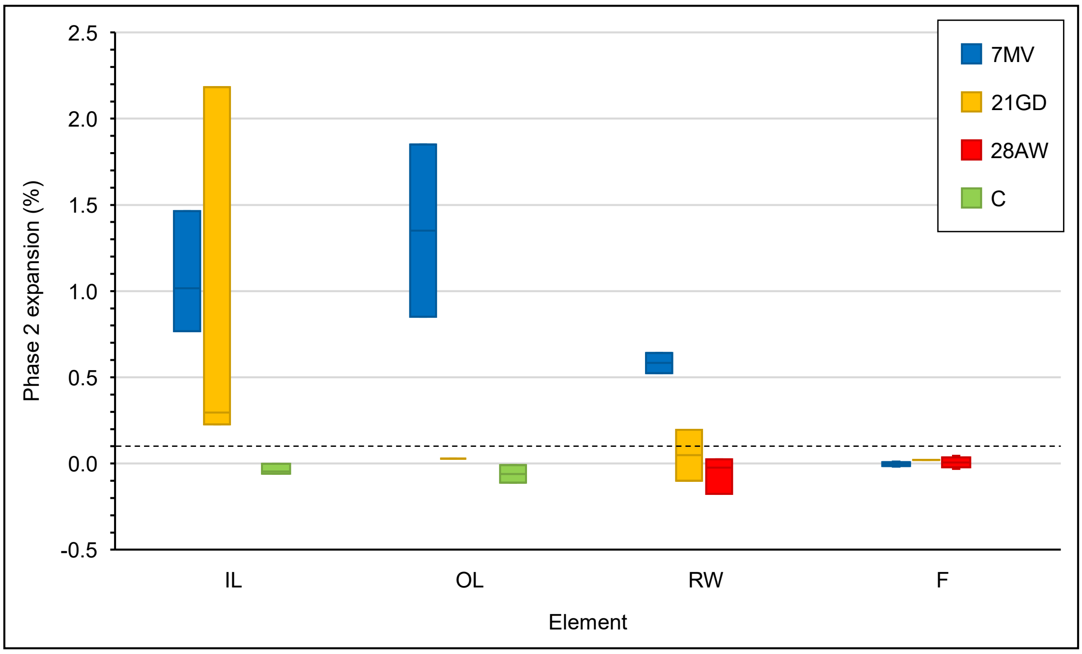
[69] R. Zhong, K. Wille, Deterioration of residential concrete foundations: (2018), see [6].

[70] CSA A23.1/CSA A23.2, Concrete test methods, P3, See [15]

[71] CSA A23.1/CSA A23.2, Concrete test methods, P3, See [15]

the cores did not reach P3 Phase 2 (particularly in 28AW) and therefore were not able to be assessed against the CSA criteria. When considering that both the Phase 2 (only) CSA P3 adapted test expansions of >0.10% and elements where any of the cores tested had suffered an integrity failure (see **Figure 7-2**) to have both failed the test, all test property concrete block elements may be considered as having failed the tests.

Figure 7-1 Phase 2 expansion



Dashed line represents the AMBT 0.10 % expansion criterion specified in CSA A23.1 P11.6[72]

However, it was deemed not meaningful to evaluate the samples against the published CSA A23.1 AMBT criteria[73] considering that the test was originally designed for mortar bars from newly prepared material, with a different surface area for both the samples and the utilised aggregate (crushed to a fine aggregate). The lower surface-to-volume ratio of the 75 mm diameter cores and depth to the centre of the sample volume may have reduced the effectiveness of the oxidising agents. The suspected high water absorption of the phyllite compared to other aggregates and greater porosity of the concrete block compared to mass concrete may offset, to some extent, some of the previously mentioned factors. However, as some concrete block samples from the test properties and the control properties completed the testing, it can be inferred that sulfide-bearing blockwork can survive the testing regime and that the results were therefore comparable at least relative to each other. Indeed, the samples of concrete blocks can be directly compared with other samples from the test properties and the control property for relative performance due to similar sample geometry and aggregate content.

In cases of severe test-generated deterioration, a comparison of time to failure (TTF), in both mean and last failure can be made per element per property. In this case, the time-to-failure metric likely indicates a combination of deterioration to date and the potential

[72] CSA A23.1/CSA A23.2, Concrete test methods, P3, See [15]

[73] CSA A23.1/CSA A23.2, Concrete test methods, P3, See [15]

for more deterioration to be caused by sulfide oxidisation, and subsequent ISA-related deterioration.

The relative comparison (see **Section 6.2**) obtained herein suggests that the concrete blockwork from the test properties was susceptible to further iron sulfide oxidisation (demonstrated by the development of increased rusting sulfides during the test), ISA and associated expansion to failure. The test properties IL, OL and rare RW sub-samples exhibited the poorest relative performance, typified by a consistent rate of expansion and mass loss during phase one of the oxidisation test with occasional sub-sample failures (typically expansions under glue) or rapid fragmentation failures of previously severely deteriorated (as-received) sub-samples observed. During phase two of the test, severe expansion and mass loss led to rapid fragmentation failure within the test property IL, OL and rare RW sub-samples indicative of possible ISA.

The test property RW samples more commonly exhibited steady late phase 2 (oxidisation test) expansion after a period of innocuous behaviour (see **Figure 6-9**). The late-stage expansion suggests some degree of potential for future deterioration but the delay in expansion can also be tentatively correlated with the relatively better as-received (Phase 1) condition of the RW samples compared to the test property IL and OL concrete block samples.

Both the control property IL and OL and the test property F samples were characterised by relatively innocuous mean expansion and mass change behaviours throughout.

Due to the severity of the observed deterioration, mean time-to-failure (TTF, see **Figure 7-2** and **Figure 7-3**) illustrates the relative performance of the concrete to resist, or susceptibility to, sulfide oxidisation and any associated ISA-related failure in the forced severe conditions. The test properties exhibited TTFs between 3-21 weeks for the IL, 1-18 weeks for the OL, 6-26 weeks for the RW and 26 weeks for the mass concrete foundations. When these are compared with the 26 week and 9-26 week TTF for the control property IL and OL respectively, a simplistic tentative order of relative pre-existing condition coupled with the relative ability to resist further ISA (in forced conditions) of OL≤IL<<RW<<F can be made.

When evaluating residual mean expansion alone, some properties appeared in better condition than others because samples had failed at lower expansions than other properties. However, when considering individual (core) and mean (core set) values of expansion to failure a more indicative performance metric can be established than residual means. This can be seen in the relationship between TTF and expansion at failure (**Figure 7-2**). Where four distinct populations can be observed.

Three expansive populations included:

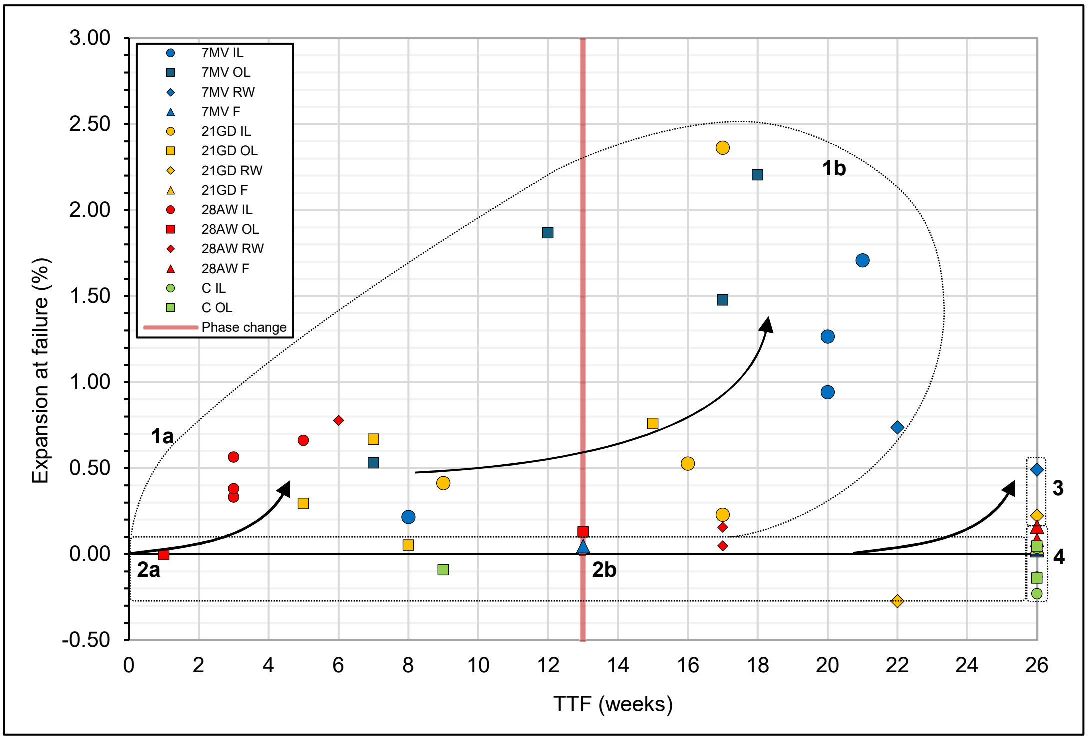
- expansive failures exhibiting greater expansion over time (1);
- non-expansive failures related to weak, possibly already severely deteriorated samples (2a)
    - surface integrity failures below measurement studs (2a & 2b);
- intact still-expanding typically late-stage expansive results (3).

The fourth population consisted of:

- ‘successful’ intact samples that did not significantly expand during the test (4).

The most severely deteriorated samples are found failing at the start of the test (1a and 2a) and should they survive, progress to 1b if they can withstand expansion and exposure cycling. Sample failures in 2a were predominantly OL samples known to exhibit the poorest Phase 1 as-received condition. As no test-induced expansions were recorded for these samples their pre-existing ISA-related deterioration had likely caused these non-expansive failures.

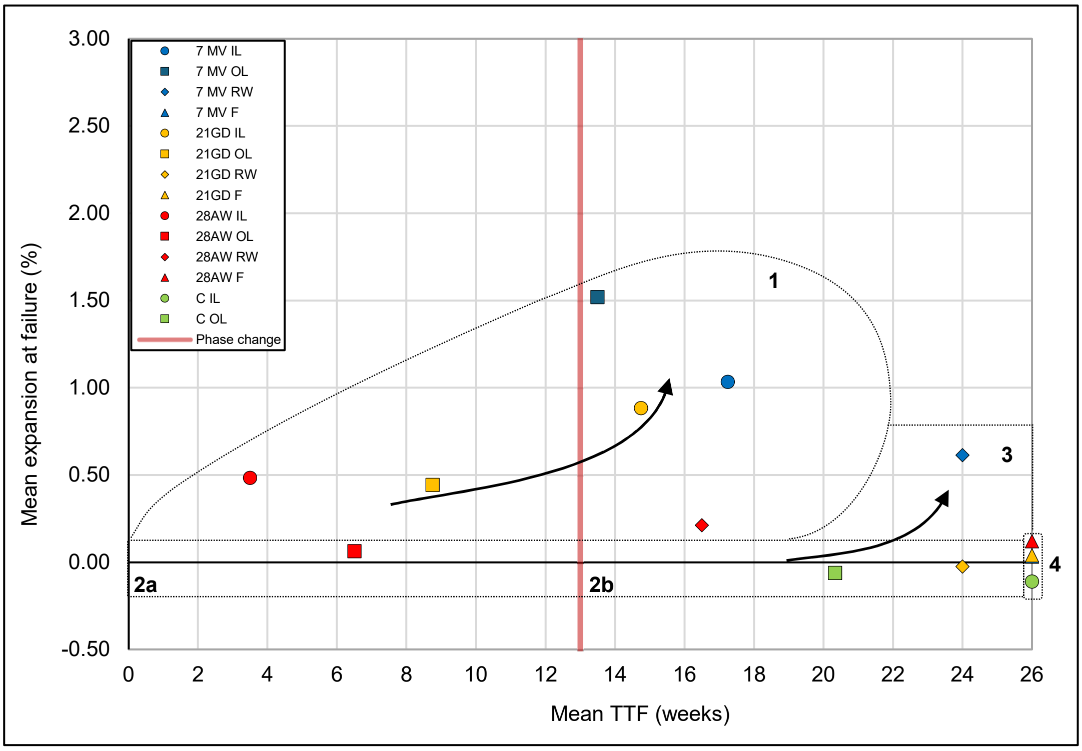
Figure 7-2 Expansion vs TTF (Core)



Mean expansions to failure and TTFs[74] populations areas 1 to 4 are approximate and relate to; 1) expansive failures with greater expansion over time; 2) non-expansive failures related to surface integrity below measurement studs; 3) intact still-expanding typically late-stage expansive results; 4) intact samples that did not significantly expand. Note that some plots are obscured behind others. Designations a and b related to phase 1 and phase 2. 7MV F 13-week TTF was taken off test purposely (non-failure). Arrows indicate the generalised progression of expansion over time.

[74] CSA A23.1/CSA A23.2, Concrete test methods, P3, See [15]

Figure 7-3 Mean Expansion vs Mean TTF (Core set)



Mean expansions to failure and TTFs[75] exhibit population areas 1 to 4 are approximate and relate to; 1) expansive failures with greater expansion over time; 2) non-expansive failures related; 3) intact still-expanding typically late-stage expansive results; 4) intact samples that did not significantly expand. Note that some plots are obscured behind others. 7MV F 13-week TTF was taken off test purposely (non-failure) and is not included in mean TTF calculations. Arrows indicate the generalised progression of expansion .

No universal criteria could be confidently assigned due to a lack of statistical representativeness and interlaboratory data for this adapted methodology. However, when considering the limitations of this study and considering mean values, a tentative limited specific performance-based criteria could be speculated. It appeared that if mean expansion to failure reached above 0.10% in phase 1 of the adapted test method, deterioration possibly coupled with a significant expansion would eventually be triggered within the concrete blockwork samples. However, this tentative expansion limit rose to approximately 0.20 % expansion in phase 2 of the adapted test method. When compared to the 0.10% expansion criteria between P3 phase 1 and P3 phase 2 from CSA A23.1/CSA A23.2[76] these limits are comparable and perhaps indicative of the similarity of the test methodology and that the differences in sample geometry and material type didn't unduly influence the generated strain other than to induce sample failures.

[75] CSA A23.1/CSA A23.2, Concrete test methods, P3, See [15]

[76] CSA A23.1/CSA A23.2, Concrete test methods, P3, See [15]

7.3 Phase 3 – Post-Durability Testing

7.3.1 OM and SEM/EDX

The test property concrete block samples all exhibited a reduction in sample integrity after the Phase 2 durability testing, particularly after CSA P3 adapted oxidisation testing. The degree of sulfide oxidisation increased specifically with iron oxides/hydroxides visually obvious (see **Figure 6-14**).

The inspections and petrographic examinations following the Phase 2 durability tests showed significant deterioration of the concrete blockwork samples in particular when compared to the relatively sound mass concrete foundation samples. Notable condition differences were present between the RICS Mundic Stage 3 and the more aggressive CSA P3 adapted oxidisation tests.

7.3.1.1 RICS The Mundic Problem Stage 3

The test property concrete block samples tested in accordance with the RICS Mundic Stage 3 test, typically exhibited major to minor increases in the degree of sulfide oxidisation, remnant highly deteriorated cement matrix with rare to common occurrences of minor ISA-related matrix conversion and associated cracking. Secondary deposits also reflected the moderate increased concrete deterioration, exhibiting possible to moderate increases in secondary sulfates including ettringite and gypsum, and rare minor changes to the abundance of secondary calcite, iron/oxide hydroxide and both increases and decreases in thaumasite observed. Notably, the rising walls appeared to suffer a notable increase in deterioration for many features observed during OM examinations when compared with as-received (Phase 1) analysis, however, this was less pronounced in SEM/EDX analysis. This notable increase could be attributed to the previous retention of sulfides prior to sampling and the exposure of the sample to an oxidising environment in the presence of water (more akin to the outer leaf, apart from temperature) rather than the chiefly saturated condition speculated in previous Phase 1 investigations. Some parameters such as cracking and frequency of iron oxide/hydroxide deposits appear to have reduced slightly, this is likely an effect of intra- and inter-block sample variation (certainly no cracks were observed to have healed for instance). Post-test sample photos (See **Figure 6-14**) show a significant increase in oxidisation sulfides compared to images of as-received materials (see **RSK Report 1283831-01**), however, the presence of mobile moisture may have helped to remove some oxidisation products and sulfates during the period of testing.

7.3.1.2 CSA A23.1 P3 Oxidisation test

All failed concrete blockwork samples including the IL and OL of the test properties and sporadic RW samples, exhibited major increases in oxidisation of reactive sulfides, severely degraded cement matrix and secondary sulfates replacing the cement matrix, including thaumasite, particularly in the OL samples. Combined with an increase in the severity of sulfide oxidisation and ISA-related cracking, frequent fragmentation of samples at the microscopic and macroscopic scale and other factors, the test had generated an effective test to destruction, indicative of both sulfide oxidisation and generation of some further degree of ISA within the test property concrete blockwork samples. The test generated comparatively innocuous response in the control and foundation samples, which exhibited relatively less, minor petrographic alteration, such as minor rusting of sulfides after the full 26 weeks of testing.

The 7MV F mass concrete sample was purposely only tested for the 13 weeks of phase 1 of the CSA A23.1 P3 Oxidisation test for comparison of the two different P3 test phases. The phase 1 only test sample represented a minor reduction in sulfide oxidisation, gypsum precipitation and cement weakening compared to the 26-week equivalent sample. It follows that the relatively aggressive test had a generally consistent slowly acting effect on mass concrete samples, due to increased density, or type 4 concrete blockwork samples, that typically contained less reactive sulfides in a generally sounder material. However, the expansion and mass data show that when a critical point of deterioration is reached within the test property pyrrhotite-bearing concrete block samples, severe rapid deterioration is generated in the test until the point of complete sample failure.

7.3.2 XRD and XRF

The comparison of Phase 1 and Phase 3 XRD data does not indicate any substantial difference between the two compositions. The most repeated trend was the relatively small increase in muscovite and chlorite in post-CSA P3 adapted samples when compared to post-RICS Stage testing. This is interpreted as the relatively increased removal of other aggregates or cement matrix preferentially to muscovite and chlorite during the CSA P3 adapted testing.

The XRF data (see **Table 6-12**) shows significant variation within building elements with only a few notable trends. Namely, that the foundations generally exhibit a greater sulfur (see **Figure 6-24**) and calcium contents (increased cement contents) than the concrete block samples (apart from one); the RICS Stage 3 samples generally included marginally greater sulfur concentrations than the corresponding CSA P3 adapted samples and finally the control property exhibited the lowest sulfur contents where measured, apart from the C IL CSA P3 adapted test samples.

7.3.3 Physical Properties

7.3.3.1 Compressive strength

The compressive strength concrete block samples following RICS Stage 3 and CSA P3 adapted tests typically decreased, between -1.4 to -8.3 N/mm^2 (-17.7 to -91.2 %) from Phase 1 values. The resulting test property concrete block compressive strengths were typically below 5 N/mm^2, particularly for the CSA P3 adapted oxidisation test (0.8-2.8 N/mm^2) rather than the RICS Stage 3 (0.7-7.4 N/mm^2) samples.

The low Phase 3 compressive strengths reflected the sample deterioration and in the case of the CSA P3 adapted lowermost strengths (~<2 N/mm^2) near or partial failure. The control property concrete blocks also decreased (-3.4 to -8.9 N/mm^2, -26.6 to -63.1 %) in compressive strength but remained stronger (5.2-9.4 N/mm^2) than the test property concrete blocks, in broad agreement with the OM, SEM/EDX observations of relatively less deterioration and no significant durability test expansions.

The mass concrete foundation samples were still significantly stronger (11.9-17.8 N/mm^2) than the concrete blocks even though they had undergone the greatest absolute reductions in compressive strength (-5.5 to -18.0 N/mm^2) and moderate to high relative changes to original compressive strength (-24.8 to -60.2 %).

Both Phase 2 durability tests had reduced the compressive strength of the samples, with the CSA P3 adapted oxidisation test resulting in a moderately greater strength reduction on average than the RICS Mundic Stage 3 test. Relative to their original compressive strengths, the greatest relative loss of strength had been generated in the test property concrete blocks compared to the test property foundations and the control property concrete block samples.

Comparison of test property concrete block elements was inconclusive as they did not present a consistent reduction in compressive strength across all test properties varying from most reduced in some properties to the least reduced in strength in other test properties. This likely illustrates the inherent variability of the individual concrete blocks in terms of their *in situ* exposure conditions, pre-phase 2 conditions, and composition but may have also been possibly affected by preservation bias towards those samples that were still intact enough to test.

7.3.3.2 Density

All samples for RICS Stage 3 testing but two increased in density during Phase 2 durability testing by up to 250 kg/m^3. This likely indicated a minor incorporation of water into hydrate reaction products or the observed conversion of the cement matrix to sulfates. In CSA P3 adapted oxidisation test samples both increases and decreases in density were recorded. Decreases in density may be accounted for by material loss or leaching, whereas increases in density may indicate incorporation of hydrates/oxides, cement conversion to calcium chlorides or the deposition of sodium chloride or sodium sulfates deposits.

7.3.4 Chemical Composition

A more diverse suite of chemistry analytical methods was conducted on the samples in Phase 3 including acid digestion, HTC, colorimetry, pH probes and ICP-OES analyses to investigate the TS, ASS, WSS, and derived values of the post-Stage 2 samples. The comparison of the results indicates that the Phase 2 durability testing has had a significant impact on sulfides, sulfates and the degree of alteration that has occurred within the samples ultimately indicating a varied deterioration of the samples. The samples available for testing were often limited by the masses required for analysis and therefore it is noted that the results are limited to some degree by the completeness of comparable data.

7.3.4.1 RICS The Mundic Problem Stage 3

The post-RICS Mundic Stage 3 cement content results typically were within ±70 kg/m^3 of the previous Stage 1 values. Considering analytical repeatability error and some variation within the sub-samples tested, values of ±30 kg/m^3 cement content from original values can be considered of no change. Negative change values of <-30 kg/m^3 (21GD OL and RW) may indicate cement depletion and removal had occurred. Conversely, positive change values of >30 kg/m^3 (7MV F) may indicate minor alteration of the aggregate possibly increasing CaO or soluble silica, or possible alteration of the hardened cement paste (indeed, analysis of the leachate produced in the Mundic test showed the release of some soluble sulfates, which may have derived from the cement or acidic byproducts of oxidisation reactions).

Test property TS values determined by EN 1744-1 acid digestion, EN 1744-1 HTC typically returned between 0.0 to 0.2 % increase and -0.1 to -0.5 % decrease of % TS by mass respectively when compared with Phase 1 results. The test property Phase 3 TS by HTC values (0.00-1.01 % S by mass of aggregate) however, were closely (typically within 0.1-0.2 % S by mass of aggregate) comparable to TS by ICP-OES TS determinations (0.33-0.75 % S by mass of aggregate). The increase in TS determined by EN 1744-1 acid digestion is likely unrepresentative as sulfates are known to have left the system by leaching, as shown by WSS leachate results (483-3850 SO_4 mg/l) and sulfides are known to have been further oxidised as seen in OM and SEM EDX investigations. It shows that the RICS Stage 3 test has released further sulfur from the pyrrhotite that can be picked up by the EN 1744-1 acid digestion methodology. As other TS methodologies have shown a decrease in TS, this increase in TS (but still to lower levels than determined by HTC) provides further evidence of the inability of the EN 1744-1 acid digestion methodology to release sulfides from the pyrrhotite present within these concrete samples. It should be noted that one isolated increase in TS determined by EN 1744-1 HTC methodology (+0.40 % TS by mass aggregate) is likely indicative of sample variability and the inclusion of a large isolated amount of pyrrhotite or other sulfide within the analysed sample. Overall the reduction in TS determined by HTC indicated that sulfates and sulfides had likely leached out of the samples during the durability testing.

The determined EN 196-2 sulfide contents change (+0.03 % to +0.30 % S^{2-}% by mass of aggregate) were broadly comparable to the unexpected EN 1744-1 TS by acid digestion contents change (+0.1 to +0.2 % S by mass of aggregate) for all test property concrete block samples. As both methods were acid digestion and barium sulfate precipitation techniques they may have been affected by the same increased digestible sulfur effect caused by RICS Stage 3 testing.

Acid-soluble sulfate (ASS) determinations by mass of sample by EN 1744-1 (0.2-0.5 % SO_4) and EN 196-2 (0.17-0.49 % SO_4) were broadly comparable however ASS determinations by ICP-OES (0.22-3.36 % SO_4) were variably significantly higher for the outer leaves, rising walls and foundations of 7MV, 21GD and 28AW. Where comparable, the Phase 3 ASS inconsistently varied (-0.40 % to +0.21 % SO_4 by mass of sample) from Phase1 results, with perhaps a preference for a slight increase in ASS observed. Negative values may represent the preferential removal of acid-soluble sulfates by leaching into the bleach during cycling (as previously discussed) or by preferential physical removal of sulfate-bearing species (e.g. crack filling deposits) during disintegration. Positive values of ASS change are consistent with some degree of further oxidisation that occurred during these accelerated durability tests as observed in OM and SEM/EDX investigations. These two processes may be competing in the deteriorated test property concrete block samples resulting in inconsistent ASS determined values. However a consistent large ASS decrease was observed in 21GD IL , possibly indicative of significant removal of acid-soluble sulfate or sulfide from the system in a similar manner to that indicated by WSS leachate values.

Assuming all the sulfur was present as a singular sulfide species for comparative purposes, the RICS Stage 3 Phase 3 calculated pyrite and pyrrhotite contents (using HTC

TS values), chiefly represented between -0.90 % to +0.13 % pyrite content by mass or --1.32 % to +0.19% pyrrhotite content change by mass of the test property concrete blocks. The typical large reductions in sulfides agrees with OM and SEM/EDX observations and chemical test results that an increase in sulfide oxidation and leaching of sulfur (WSS) out of the samples had occurred. A notable outlier was the inner leaf of 21GD which exhibited a notable increase of 1.00-1.50 % sulfide by mass due to reduced acid-soluble sulfate and increased TS content. This increase may be possibly accounted for by sample variation, with the Phase 3 sub-sample containing a substantially higher initial sulfide content.

As mentioned above, given the significant amounts of WSS present within the leachate, WSS determinations for the RICS Stage 3 samples were, as expected, notably low (0.00-0.24 % SO_3 by mass). However, in some TP IL and OL elements, despite a high determined leachate WSS (e.g. 3850 mg/l SO_4, 7MV IL), WSS had increased (+0.09 % SO_3 by mass) during RICS Stage 3 testing, which indicated relatively high WSS generation or mobilisation within the sample and likely gypsum generation indicative of ISA.

The pH within the concrete samples suggested that the IL and the OL were less alkaline (8-11) than the RW and F (11.5-12.5) post-RICS Stage 3 durability testing, indicative of more remnant cement content in the RW and F compared to the sulfide oxidisation generated acidity of the more deteriorated IL and OL samples.

7.3.4.2 CSA A23.1 P3 Oxidisation test

The Stage 3 cement content results of the samples were within ±80 kg/m^3 of the previous values. Considering analytical repeatability error and some variation within the sub-samples tested, values of ±30 kg/m^3 cement content from original values can be considered of no change. Positive change values of >30 kg/m^3 (28AW IL and OL, 7MV F) may indicate minor alteration of the hardened cement paste or release of calcium and/or silica from the aggregate. Conversely, negative values <-30 kg/m^3 (21GD OL and C OL) may indicate that cement depletion and removal by leaching have occurred.

Test property TS values determined by EN 1744-1 acid digestion, EN 1744-1 HTC returned between -0.3 % to 0.1 % change and -0.08 to -0.91 % change of % TS by mass respectively when compared with Phase 1 results. The TS by HTC values (0.00-0.47 % S by mass of aggregate) did not consistently agree with comparable TS by ICP-OES determinations (0.03-0.82 % S by mass of aggregate), particularly for 21GD OL and RW concrete block samples, which differed by approximately 0.4-0.5 % TS by mass of aggregate. Chiefly the TS determined by EN 1744-1 acid digestion, was not significantly altered from Phase 1 results but was still notably lower than HTC and ICP-OES TS techniques. As with the RICS Stage 3 samples, this provides further evidence of the unsuitability of the EN 1744-1 acid digestion test method for the analysis of these samples. The consistent and sizeable decrease in TS determined by EN 1744-1 HTC methodology was interpreted to possibly represent the severe oxidisation of sulfides observed in the corresponding OM and SEM/EDX analyses and the removal of sulfate from the system in the bleach cycling suspension or in constant humidity conditions.

The variability and lack of consistent pattern within the change in ASS (-0.50 % to +0.32 % SO_4 by mass of sample) and WSS (-0.17 % to +0.18 % SO_3 by mass of sample) data between Phases 1 and 3 for the CSA P3 adapted oxidisation test samples suggests different dominant processes may have occurred. Negative values may represent the preferential removal of acid-soluble sulfates by leaching into the bleach during cycling or by preferential physical removal during disintegration. Positive values of ASS may indicate that some degree of further oxidisation occurred during these accelerated durability tests as observed in OM and SEM/EDX investigations. These two processes may be competing in the deterioration test property concrete block samples resulting in inconsistent ASS and WSS determined values.

A similar post-durability testing decrease in EN 196-2 determined sulfide content (+0.07 % to -0.56 % S^{2-}% by mass of aggregate) is noted when compared to TS by HTC for nearly all samples. The relative decrease in the determined sulfides may represent the influence of easily oxidisable sulfides being oxidised during the durability testing, or the digestion of some sulfides, particularly pyrrhotite, in the acid digestion process used for the determination of sulfate. The EN 196-2 sulfide content method was also an acid digestion and barium sulfate precipitation technique, which seems to better agree with the relative changes in EN 1744-1 TS determinations by HTC results than by acid precipitation, contrary to the same observation in RICS Stage 3 test samples.

Assuming all the sulfur was present as a singular sulfide species for comparative purposes, the calculated pyrite and pyrrhotite contents, represented between -0.15 % to -1.50 % pyrite by mass or -0.22 % to -2.02 % pyrrhotite content by mass of the test property concrete blocks. This significant reduction in sulfides agrees with the TS by HTC, sulfide, OM and SEM/EDX observations that an increase in sulfide oxidation and sample deterioration had occurred.

The pH within the concrete samples was generally consistently high and likely indicative of the bleach cycling experienced by all of the CSA P3 adapted oxidisation test samples.

7.4 Overall Prognosis

The concrete used in the test property concrete blocks are known to be of a composition that includes risk factors for damaging internal sulfate attack. The Phase 3 testing confirms that sulfide oxidisation and internal sulfate attack processes have progressed within the concrete during the oxidising processes that occurred during the Phase 2 testing. The most severe progression of sulfide oxidisation and ISA-related processes was observed during CSA P3 adapted oxidisation testing and more moderately within the RICS Mundic Stage 3 moisture sensitivity expansion testing, likely representative of the relative aggressiveness of the specified conditions for each test.

The OL and IL of the test properties were already established as undergoing some degree of ISA in service (Phase 1) and had been rarely observed to fail *in situ*. As such, the as-received condition, increased ISA in both Phase 2 durability tests and the significant damage and failures observed in the CSA P3 adapted test indicated that the IL and OL samples of the test properties are at high risk of further ISA-related deterioration. The test property RWs were of relatively better as-received condition (Phase 1) than the IL and OL, and provided innocuous expansion results for the RICS Stage 3 durability test. However, the CSA P3 adapted oxidisation test was able to produce both late-stage expansion and sample failure (chiefly occurring later in the testing than for OL and IL samples). Furthermore, Phase 3 testing of the test property RWs indicated that both Phase 2 durability tests had generated an increase in sulfide oxidisation and significant further ISA within the cement matrix. Therefore we can infer that the test property RWs represent an intermediate case of having the potential to undergo significant further ISA that given enough time may generate failure. This relatively slow progression in service (when compared with the IL and OL) is likely a consequence of the position of the rising wall, being in a largely saturated condition but with occasional free oxygen exposure. As such, the environmental conditions to promote damaging internal sulfate attack are more restricted than the IL and OL but less restricted than the mass concrete foundations; additionally, the higher porosity and permeability of the concrete blocks, compared with the cast in situ foundation concrete, is likely less resistant to ISA processes.

The absence of carbonate-bearing aggregate in the concrete may also be significant, as this minimises the likelihood of the thaumasite form of sulfate attack (TSA) occurring. TSA is generally more significantly damaging than conventional sulfate attack, and is the favoured mechanism in wet/saturated conditions (compared with conventional sulfate attack, predominating in damp conditions), but requires the presence of a source of carbonate. While the carbonated cement could provide some of this, it is likely insufficient in volume to allow significant TSA progression.

Based on the above, assuming similar *in situ* conditions, it is considered that there is a severe risk that ongoing pyrrhotite oxidisation, and associated internal sulfate attack processes, will lead to the development of significant damage to the outer leaf and inner leaf concrete elements sufficient to adversely affect their engineering performance within the remaining intended design life of these buildings.

Additionally based on the above, assuming similar *in situ* conditions, it is considered that there is a moderate risk that ongoing pyrrhotite oxidisation, and associated internal sulfate attack processes, will lead to the development of significant damage to the rising wall concrete elements sufficient to adversely affect their engineering performance within the remaining intended design life of these buildings.

8 CONCLUSIONS

The accelerated durability tests of Phase 2 and the subsequent Phase 3 testing suite investigated the longer term durability of the concrete blocks and the mass concrete foundation of from the three test properties. The detailed results present herein allow the comment on the potential of the samples for further sulfide oxidisation and any subsequent deterioration of the samples. The main conclusions are as follows:

- A total of 86 sub-samples were taken from a total of 52 no. received samples, which were taken from IL, OL, and RW concrete block and F mass concrete elements.
- The test property concrete was known to contain reactive pyrrhotite and had all undergone some degree of deterioration ranging from rare traces in the mass concrete foundations to severe disintegration caused by sulfide oxidisation and subsequent ISA in some samples of the inner and outer leaves.
- Half of the sub-samples were tested in accordance with RICS The Mundic problem, Stage 3 Moisture sensitivity expansion test or CSA A23.1 P3 adapted oxidisation test. 43 No. sub-samples were tested for each test. These sub-samples covered all sampled property elements apart from the control property foundation where not enough samples remained after Phase 1 testing.
- Minor density increases in all but one sample indicated the incorporation of water, bleach and oxygen into the concrete during the testing, in the form of sulfates, oxides/hydroxides or chlorine compounds (the last in CSA P3 adapted only).

8.1 RICS The Mundic problem, Stage 3 testing

- All samples completed the RICS The Mundic problem, Stage 3 Moisture sensitivity expansion test for either 250 or 350 days and exhibited increased sulfide oxidisation although not all sulfides reacted under the test conditions.
- All bar one of the test property IL and OL samples exceeded the 0.025% mean expansion criterion specified within the RICS Mundic guidance for Stage 3 testing but remained intact.
- The test properties RWs and Fs along with the control property IL and OL exhibited innocuous expansion behaviour within the RICS The Mundic problem, Stage 3 Moisture sensitivity expansion test.
- Test property ILs, OLs and RWs all exhibited significant evidence of further sulfide oxidisation and associated ISA in post-durability test, Phase 3 analyses.
- Test property RWs were still potentially at risk due to the increase of factors associated with ISA-related concrete deterioration and eventual failure.
- Leaching of sulfates out of the samples, as seen in Phase 1 test property IL and OL *in situ* behaviour may have also resulted in less evidence of ISA and possibly reduced internal expansion and associated dimensional instability during the RICS Stage 3 testing prior to the point of failure.

- Test property IL samples only tested for 250 days had still exceeded the 0.025% mean expansion criteria, which precluded the need for further extension of the test conditions however the reduction in test length made the comparison of relative sample condition to other samples tested for 350 days relatively unreliable.
- Decreases in compressive strength were determined for all but one tested sample after RICS Stage 3 testing.
- The compressive strength of post-RICS Stage 3 test property concrete block samples (0.7-7.4 N/mm^2) generally decreased between -1.4 to -7.7 N/mm^2 (-17.7 to -91.2 %) from Phase 1 values.
- The compressive strength of post-RICS Stage 3 test property control property concrete block samples (8.4-9.4 N/mm^2) decreased between -3.4 to -5.7 N/mm^2 (-26.6 to -40.4 %) from Phase 1 values.
- The <2 N/mm^2 compressive strength post-RICS Stage 3 test property concrete block samples appeared close to failure before compressive strength testing.
- Variation in the as-received sample condition may have affected variation in the recorded Phase 3 compressive strengths.
- The Phase 3 post-RICS Stage 3, compressive strength testing showed that the PHY-bearing concrete blocks had the lowest remnant strengths when compared to the SST-bearing concrete block and the PHY-bearing foundation compressive strengths, when all were exposed to a warm moist environment.
- A large change (chiefly reduction) in TS by HTC (-0.50 to +0.05 % S) and estimated sulfide species content (-0.90 % to +0.13 % pyrite or -1.32 % to +0.19 % pyrrhotite) by mass for concrete block samples post-RICS Stage 3 testing was observed (in all but one sample), interpreted to represent sulfide oxidisation and sulfate mobilisation out of the samples as suggested by high WSS leachate levels.
- Post-RICS Stage 3 testing, TS by HTC (0.42-1.01 % by mass of aggregate) or ICP-OES (0.33-0.75 % TS by mass of aggregate) and sulfide by EN 196-2 acid digestion (0.45-0.75 % S^{2-}) appeared to give sulfur contents more consistent with petrographic and instrumental observation, compared to the EN 1744-1 TS acid digestion method (0.1-0.5 % TS by mass of aggregate) for the investigated pyrrhotite-bearing samples.
- EN 1744-1 TS (acid digestion) and EN 196-2 sulfide determination methods were acid digestion and barium sulfate precipitation techniques. Both methods appeared to have been affected by an increased digestible sulfur effect caused by RICS Stage 3 testing not seen in the EN 1744-1 TS by HTC results when compared to Phase 1 as-received values.
- Post-RICS Stage 3 testing, WSS by colorimetry within the concrete (37-1420 mg/l SO_4) was relatively ordered by element F<<RW~OL~IL. The WSS of the leachate was (2150-3850 mg/l SO_4) for concrete blocks and (483-1910 mg/l SO_4) for the mass concrete foundations. This indicated that between 1.5-5 times more WSS had been leached out of the samples than remained in the concrete blocks.

- When considering the RICS Stage 3 testing generated leachate WSS as a proxy for the amount of ISA that had occurred, the elements were relatively ordered F<RW<OL~IL.
- Post-RICS Stage 3 testing, ASS changes were variable (-0.50 % to +0.21 % SO_4 by mass of sample) with negative values suggesting possible removal of acid-soluble sulfates by leaching or physical removal during disintegration. Positive values of ASS may indicate that some relative degree of further oxidisation had occurred.

8.2 CSA A23.1 P3 Oxidisation test

- The original oxidisation mortar bar method (AMBT) was adapted for cored concrete blockwork and mass concrete sub-samples. The chosen sample geometries and materials were proven to be able to withstand the testing regime, and therefore be able to be compared relatively to each other in the absence of any specific criteria for the investigated materials.
- A high proportion of the tested sample population failed to complete the full 26 weeks of the CSA P3 adapted oxidisation test.
- The adapted test methodology generated maximum recorded expansions up to 2.4 % within tested samples, which indicated that significant expansions were able to be generated by the test methodology.
- To account for the physical sample failure (often complete fragmentation) due to induced deterioration, time-to-failure (TTF) and expansion at failure metrics were used to compare the relative performance of the CSA P3 adapted oxidisation test samples.
- Four main classifications of test performance for TTF vs expansion at failure were apparent for the investigated samples.
  1) Expansive failures progressing over time in both 1a) phase 1 (20°C) and 1b) phase 2 (80°C).
  2) Non-expansive failures related to weak, deteriorated as-received samples or repeated surface adhesion failures in both 2a) phase 1 (20°C) and 2b) phase 2 (80°C).
  3) Intact late-stage expansive (at end of test) samples in late phase 2 (P3)
  4) Intact non-expansive samples at the end of the test.
- The test property IL and OL samples typically exhibited the shortest TTFs and separately the greatest expansions at failure during CSA P3 adapted oxidisation testing. The test property OL samples exhibited more Type 2 non-expansive failures than IL samples, however, IL samples exhibited more type 1b high expansion failures than OL samples. Interpreted to be indicative of poorer *in situ* OL condition than *in situ* IL condition.
- The test property RW samples exhibited either Type 1a (rare), 1b, 2b or Type 3 late stage behaviour indicative of more resistance to sulfide oxidisation and ISA compared to other test property concrete block samples, possibly due the relatively better as-received (Phase 1) condition. The Type 3 late stage failure

and expansion also indicated the potential for the test property RW to deteriorate given enough exposure to favourable deterioration conditions.

- Control property IL and OL and all foundation samples exhibited Type 4 non-expansive intact behaviour, indicative of the ability of the PHY-bearing mass concrete and SST-bearing concrete blocks to resist deteriorative sulfide oxidisation and ISA in forced aggressive oxidisation environments designed to generate these processes, indicating a low risk of future deterioration.
- A tentative order of relative preexisting condition compared with the relative ability to resist further ISA and sulfide oxidisation (in forced conditions) of OL≤IL<<RW<<F can be made.
- Tentative failure criteria could be assigned for the samples tested.
- Rapid expansive failure tended to follow a period of relatively steady expansion, hypothesised to be related to sulfide oxidisation and then the development of ISA related deterioration.
- Test property ILs, OLs and sporadic RWs exhibited significant evidence of further sulfide oxidisation and associated ISA in post-durability test, Phase 3 analyses.
- The CSA P3 adapted oxidisation test appeared to have a reasonably consistent deteriorative affect on the mass concrete.
- The compressive strength of the limited number of post-CSA P3 adapted oxidisation test, test property concrete block samples (0.8-2.8 N/mm^2) changed (decreased) between --8.3 to -3.7 N/mm^2 (-62.7 to -91.2 %) from Phase 1 values.
- The compressive strength of post-CSA P3 adapted oxidisation test, control property concrete block samples (5.2-7.4 N/mm^2) decreased between -8.9 to -5.4 N/mm^2 (-42.2 to -63.1 %) from Phase 1 values.
- The <3 N/mm^2 compressive strength post-CSA P3 adapted oxidisation test property concrete block samples appeared close to failure before compressive strength testing.
- Variation in the as-received sample condition may have contributed to the variation in the recorded Phase 3 compressive strengths.
- The post-CSA P3 adapted oxidisation testing compressive strength testing indicated that the PHY-bearing concrete blocks exhibited lower strengths when compared to the SST-bearing concrete block and the PHY-bearing foundation compressive strengths.
- A large change (chiefly reduction) in TS by HTC (-0.91to -0.03 %) and estimated sulfide species content (-2.02 % to 0.02 % pyrrhotite or --1.50 to 0.01 % pyrite) by mass for concrete block samples post-CSA P3 adapted oxidisation testing was observed, interpreted to represent sulfide oxidisation and sulfate mobilisation out of the samples.
- Post-CSA P3 adapted oxidisation testing of the test properties TS by HTC (0.00-0.47 % S by mass of aggregate), ICP-OES (0.01-0.72 % S by mass of aggregate) and sulfide by EN 196-2 acid digestion (0.01-0.62% $S^{2-}$by mass of aggregate) appeared to give a more expected sulfur contents when compared to the EN 1744-1 TS acid digestion method (0.0-0.3 % TS by mass of aggregate) for these

pyrrhotite bearing samples even after oxidisation or leaching of S out of the system.

- EN 1744-1 TS acid digestion and EN 196-2 sulfide determination methods were acid digestion and barium sulfate precipitation techniques. Strangely, both methods appeared to have been affected differently by CSA P3 adapted oxidisation testing when compared to Phase 1 values.
- Post-CSA P3 adapted oxidisation testing, WSS results were rarely higher than 0.10 % SO_3 by mass of sample indicating potential WSS removal.
- Post-CSA P3 adapted oxidisation testing, ASS changes were variable (-0.50 % to +0.32 % SO_4 by mass of sample) with negative values suggesting possible removal of acid-soluble sulfates by leaching. Positive values of ΔASS may indicate that some relative degree of further oxidisation had occurred.

8.3 Overall Prognosis

- This report finds that, assuming similar *in situ* conditions, it is considered
    - There is a severe risk that ongoing pyrrhotite oxidisation, and associated internal sulfate attack processes, that will lead to the development of significant damage to the outer leaf and inner leaf concrete elements sufficient to adversely affect their engineering performance within the remaining intended design life of these buildings.
    - There is a moderate risk that ongoing pyrrhotite oxidisation, and associated internal sulfate attack processes, will lead to the development of significant damage to the rising wall concrete elements sufficient to adversely affect their engineering performance within the remaining intended design life of these buildings.

9 REMARKS

These findings refer only to the samples sampled and tested and to any materials or areas properly represented by those samples.

Any assessment of risk mentioned herein is based upon the findings of these specific investigations and any information provided to the investigation. Extension of this assessment of risk to any properties not included in this investigation should be with caution and ideally should include site-specific assessment of the existing concrete.

Statements of uncertainty of test measurements are provided on test certificates only where these are specifically declared by the documented Test Method and are the result of a formal inter-laboratory precision trial.