



D1.3/2024 – Laboratory investigation plan for accelerated leaching and thaumasite attack of cementitious materials

PERCO2 / WP1 – Durability testing of existing concrete specimens

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1 Introduction

In Finnish nuclear Low and Intermediate Level Waste (LILW) repositories, the usage of reinforced concrete in the engineered barriers is significant. Reinforced concrete engineered barriers are designed for hundreds of years of effective service life. Throughout this usage period, the concrete structures must withstand a wide range of degradation drivers present in the underground cavern environment, especially regarding the long-term contact with flowing groundwater containing mineral ions. The presence of sulfate ions in the groundwater, as well as the flow of the groundwater and the pH gradient can cause sulfate and thaumasite degradation, as well as leaching of calcium from the cementitious matrix.

In this work package, sulfate attack and leaching of cementitious materials are to be investigated. The goal is to assess the performance of different mortar mixes under simulating solutions, against leaching behaviour and against sulfate attack. The conditions of storage of the magnesium sulfate solution are also relatively favourable for thaumasite formation.

1.1 Leaching of cementitious materials

The leaching phenomena of calcium phases in cementitious materials is important for long-term storage in repository conditions (post-closure). With elongated contact with water, which has significantly lower pH level compared to cement paste, leaching can occur.

When leaching of cementitious minerals occur, the calcium phases are dissolved in stages [1]. At first, the more soluble mineral, calcium hydroxide (CH) is dissolved, followed by a dissolution of the calcium in calcium-silicate-hydrates (CSH) [2], [3]. This dissolution occurs mainly due to concentration gradients between the pore solution and the surrounding solution, that result in the leaching of calcium ions and

the reduction of alkalinity in the cement phases. Leaching of calcium is associated with an increase in porosity and subsequent strength loss [4], [5], [6], [7].

Leaching is quite a slow phenomenon, and it can be difficult to simulate naturally. Leaching is affected by parameters related to the concrete itself (water/cement ratio, porosity, etc.), as well as the surrounding solution, such as the acidity of the water solution. Research in leaching typically utilize the use of de-ionized water [8], [9], or in most cases the use of an ammonium nitrate solution (6 mol typically) [10], [11], [12], or ammonium chloride solutions [13], [14]. The use of these simulating solutions utilizes the gradient between the pore solution and the ambient solution, with a higher gradient compared to natural conditions, resulting in accelerating the leaching process. The use of 6 mol ammonium nitrate solution is typically much faster, where it is estimated to accelerate the leaching phenomena by a factor of 100 [12].

1.2 Thumasite sulfate attack

Sulfate attacks are also associated with long-term storage in repository conditions post-closure. Concrete is in constant contact with groundwater, which (according to the location and depth of the repository) contains various ions, such as sulfates. The presence of significant amount of sulfate in the groundwater can increase the risk of sulfate attacks.

In sulfate attacks, the sulfate ions interact with the aluminate and CH phases in cement paste, resulting in the production of ettringite and gypsum [15]. The products can differ with the type of sulfate ion. Brucite and magnesium silicate hydrates (MSH) can form with magnesium sulfate for instance [16]. Sulfate attacks can be affected by various internal and external parameters, such as the porosity and w/b ratio of concrete [16], the chemical composition of cement [17], the solubility and type of sulfate ion [18], the pH of the concrete [19], and the possibility of thaumasite formation.

Thaumasite formation is a unique type of sulfate attacks, that can occur in cement-based structures under special conditions. The occurrence of thaumasite formation is not common, as the presence of favourable conditions for the formation can be difficult. However, these conditions can be present in repository conditions, where the structural element is in constant contact with sulfate filled groundwater (especially magnesium sulfate), and the temperatures can be relatively low in the repositories, below 15 °C, which is favourable for thaumasite formation [15], [20], [21], [22]. These conditions can increase the probability of thaumasite sulfate attacks. The product of the reactions, thaumasite, is a mushy substance that is much weaker than the original material. The thaumasite form of sulfate attack also affects the CSH in cement paste instead of aluminates [20], which can occur directly [23], or most likely after the formation of ettringite [21].

2 Experimental investigation

2.1 Materials and mortar mix plan

The mix design of different cementitious materials is to be investigated in this study, to assess the behaviour of the different cement-based products against leaching and against sulfate attacks. The mix of the specimens was decided to match the mixes used for the PERCO₂ concrete testing program [24]. Overall, five different mixes were planned with varying binder types that utilize Sulfate Resisting (SR) cement, Silica Fume, and Ground Granulated Blast Furnace Slag (GGBFS). Table 1 shows an outline of the mortar mixes used in this study and binders used in each mix.

Table 1. Mix design of the mortar specimens

Mix	Binder		w/b
	Portion [%]	Type	
SR-mix	100	CEM I	0.44
SR-SI-mix	90 10	CEM I Silica fume	0.44
KA-mix	100	CEM III/A	0.44
KB-mix	100	CEM III/B	0.44
KB-SI-mix	90 10	CEM III/B Silica fume	0.44

2.2 Preparation of specimens

The specimens are prepared in accordance with EN 196-1 [25]. For each batch, 7 moulds were used, producing 21 specimens per mix. The mixing process consists of a 30-second dry mix, followed by a gradual addition of water over 30-seconds. The materials are then mixed for 30 seconds, then the walls of the mixing drum are scraped of mortar. The mixing is then continued for 90 seconds, and the total mixing time is 3 minutes. The initial temperature of the mix is recorded, and the fresh properties are assessed.

After mixing, the mould (shown in Figure 1) is fixed into the compaction device. The mould is filled by mortar, approximately halfway, then the mortar is compacted. After the first compaction cycle (60 jolts), the mould is filled with additional mortar, and the second round of compaction is done. The mould is then de-attached from the compaction apparatus. The surface of mortar is smoothed, and the mould is then covered by glass for 24 hours, as shown in Figure 2.

After 24 hours of moulding, the specimens are demoulded, after which they are placed in the rain chamber, which has a humidity of 95%. The humidity chamber is shown in Figure 3. The specimens are placed there curing until they reach an age of 28 days. Additional initial testing of the hardened specimens and storage in the appropriate solution is conducted at further stages.



Figure 1: Mould filling and compaction of the specimens.

Chemical analysis of the specimen is done before and after storage, to investigate the phase changes in the mixes with time. Monthly visual investigation is also done to check the progression of the degradation in the different mixes. The investigation includes mass and dimensions monitoring, as well as checking the reduction in pH levels (for the leaching investigation).



Figure 2: Moulds covered with glass, to have a smoother casting surface.



Figure 3: Specimens placed in the 95% humidity chamber.

2.3 Storage of the specimens

The specimens were then stored in simulating solutions, an ammonium nitrate solution and an Epsom salt solution (close to saturation) for the leaching and sulfate attack solutions, respectively. The storage containers are placed in the research tunnel in Aalto University, which has an ambient temperature around 15°C, which is suitable for thaumasite formation [22]. Table 2 shows the used solutions and their concentrations.

Table 2: Concentration of the used simulating solutions.

Solution	Information
Leaching	Chemical NH_4NO_3
	Concentration 6 mol
Thaumasite Sulfate	Chemical $\text{MgSO}_4 \cdot 7 \text{H}_2\text{O}$
	Concentration Mg: 20 mg/L SO ₄ : 80 mg/L

The specimens are placed in separate containers. In total, 14 specimens per mix were stored in the leaching solution, and 4 specimens per mix were stored in the sulfate solution. The leaching solution requires more specimens, as some of the needed periodic tests are destructive and the specimen cannot be reused (compression and phenolphthalein tests). For the thaumasite solution, the specimen can be stored for longer durations, as the periodic tests are not destructive. The total number of casted specimens per mix is 21. The remaining 3 samples are either tested for initial parameters, 1 for strength and 1 for chemistry), and the last specimen (per mix) is kept outside the solutions in the tunnel. It is kept as a spare, but it can also be used for natural carbonation assessment. Figure 4 and Figure 5 show the storage containers used for the leaching and sulfate attack investigations, respectively.



Figure 4: Storage container for the leaching investigation.



Figure 5: Storage container for the sulfate attack investigation.

3 Testing plan of the specimens

3.1 Testing of fresh mortar

The fresh properties of mortar are measured after mixing. Directly after mixing, the initial temperature of the mix, the workability of mortar, the fresh density, and the air content of the fresh mortar are recorded in this order. The results of the 5 mixes are shown in Table 3.

Table 3: Fresh properties of the mixed mortar.

Mix	Temperature (C°)	Slump (cm)	Density (kg/L)	Air content (%)
SR-mix	26	17.9	2.22	3.6
SR-SI-mix	26.2	13.4	2.24	3.8
KA-mix	26.1	18.55	2.2	3.8
KB-mix	27.2	19.15	2.17	3.4
KB-SI-mix	25.8	15.25	2.16	3.6

3.1.1 Slump test

The workability of mortar is assessed using the flow table method in according to EN 1015-3 [26]. In this method, a flow table is used, and a truncated cone is filled with mortar in 2 layers, each compacted (10 hits). The table disc is raised at constant frequency, 15 times. The readings are then taken as the mean of 2 measurements. Figure 6 shows the slump flow table test after raising the truncated cone, and Figure 7 shows the slump after the 15-time compaction, ready for measurement.



Figure 6: Mortar slump flow test (before compaction).



Figure 7: Mortar slump flow test (after compaction + result).

3.1.2 Air content test

The air content of mortar is determined in accordance with EN 1015-7, method A (pressure method) [27]. In this method, a 1 litre container is filled with mortar in four layers, each layer compacted 10 times by tamping. Excess mortar is wiped off, and the cover is fixed onto the container. After passing water through the valves, the air content reading can be taken. Figure 8 shows the pressurized measurement apparatus.



Figure 8: Pressurized air content measurement device (1 litre).

3.1.3 Fresh density of mortar

For testing the fresh density of mortar, the same container used for air content measurement shown in Figure 8 is used. The container has a known volume of 1 litre (which was verified on site using water volume). The measured weight of compacted concrete in the container is therefore used to obtain the fresh density of mortar.

3.2 Testing of hardened mortar

The testing of hardened mortar is done through various stages. Initial testing of the hardened mortar consists of testing its compressive strength, as well as the chemical analysis of the mortar specimens. Similarly, these tests are also conducted at the end of the storage period. Additionally, periodic assessment and monitoring of the mortar is conducted to estimate the changes of the mortar mix throughout the storage period. The estimated schedule for testing is shown in Figure 9. The details of the conducted tests are explained in the following sub-chapters.

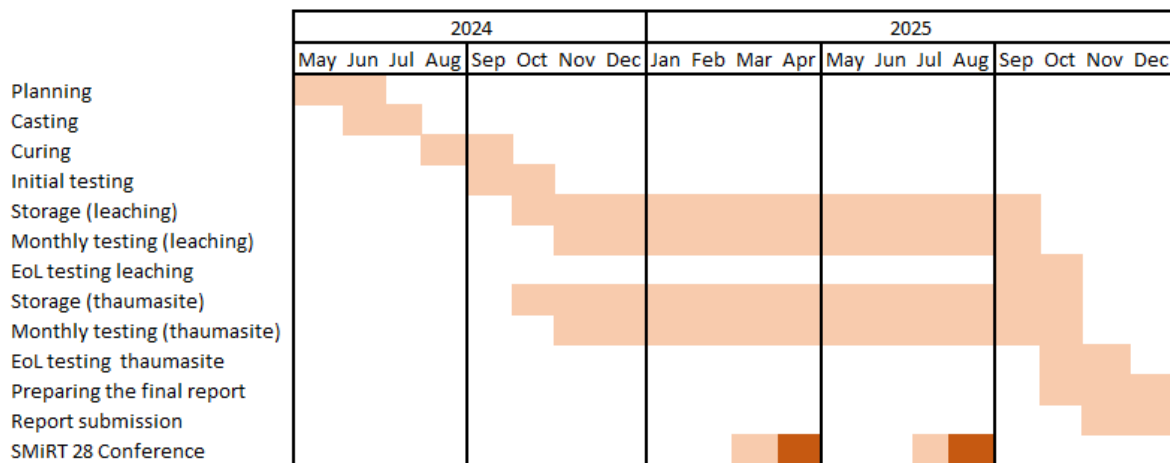


Figure 9: Estimated casting and testing schedule.

3.2.1 Compressive strength test

The compressive strength of the mortar mixes is evaluated at 91 days of storage in accordance with EN 1015-11 [28]. The testing rig is shown in Figure 10. The specimen is loaded on its side face. Each mortar specimen can be used for 3 strength tests.

This is done to obtain a baseline value that is used later for assessment. For leaching assessment, the calcium phases can decompose and leach out during storage, leading to an increase in the porosity and voids in the specimens and a reduction of its structural properties. Similarly with sulfate attack, the swelling and spalling of the

mortar specimen reduces its load-bearing capacity. Thus, assessing the strength drop at the end of the study is necessary to evaluate the extent of damage in each mix.



Figure 10: Initial compressive strength test of a mortar specimen.

The initial test results of mortar strength before storage are shown in Table 4. The target 28d compressive strength follows the required strength of the corresponding concrete mixes, which was 60 MPa. As seen in the table, the results generally fall above the requirement threshold of 60 MPa, with SR-SI mix having the highest strength. Mixes with high slag content showed lower strength values, with KB-SI having the lowest value amongst the mixes, just reaching 60 MPa.

Table 4: Initial compressive strength results of the mortar mixes.

Mix	Compressive strength (MPa)
SR-mix	68.4
SR-SI-mix	74.2
KA-mix	72.8
KB-mix	65.5
KB-SI-mix	60.1

To complement the testing at the start and end of the program, additional periodic tests are conducted for the leaching investigation, to assess the strength drop of the specimen throughout the storage duration.

3.2.2 Phenolphthalein test

Due to the leaching of cementitious phases in the mortar specimens, the pH level of the specimens drops. The leaching progresses through the depth of the specimen with time, and the pH drop increases accordingly. This behaviour, the drop of pH level with time, can be similar to the carbonation process. Thus, using a phenolphthalein solution is done here to periodically assess the leaching progress. This method was utilized in leaching research [11], [29], [30], [31]. Figure 11 shows the phenolphthalein test after the fourth month, indicating the progression the leached front deeper into the mortar mixes. This test is only conducted on the specimens under leaching simulation. The test is done in accordance with EN 12395 [32].



Figure 11: Phenolphthalein test in the mortar mixes.

3.2.3 Mass monitoring

Similar to the phenolphthalein test, the mass of the specimens is monitored monthly to assess the behavioural changes due to the simulating environment. With observing the changes in mass, the progression of the sulfate attack, and to a lesser extent leaching, can be evaluated.

Due to the sulfate attack, the chemical reaction commences in the cementitious phases, resulting in the production of expansive aluminate phases (ettringite and gypsum) [33]. The ingress of the sulfate ions, as well as the subsequent swelling of the specimen can be observed, and the mass of the specimen increases at first. With the progression of the sulfate attack, the internal stresses due to the expansion in the specimen lead to the spalling of material. This results in a reduction of the mass of the specimen. Monitoring and measuring the mass of the specimen is therefore utilized in the estimation of the sulfate attack progression.

Similarly, with the leaching of the cementitious phases in the mortar specimen, the mass of the specimen can drop slightly. The mass drops in this instance are not as significant as the changes that occur to the specimens that are under sulfate attack.

3.2.4 Chemical analysis

Chemical analysis is done to the mortar mixes before and after storage in the simulating solution, to obtain an understanding of the occurring changes to the specimen. The chosen analysis method to analyse mortar mixes is X-Ray Fluorescence (XRF), as it is suitable to obtain information of composition of a complex system throughout its depth.

4 Summary

In this investigation, five mortar mixes were prepared with fixed w/b ratio and design strength. The mixes had different binders, containing Sulfate Resisting (SR) cement, Silica Fume, and Ground Granulated Blast Furnace Slag (GGBFS). The specimens are stored in simulating solutions (6mol ammonium nitrate and a saturated Epsom salt for the leaching and sulfate attack solutions, respectively). Chemical analysis of the specimens is done before and after storage, to investigate the phase changes in the mixes with time. Monthly visual investigation is also done to check the progression of the degradation in the different mixes. The investigation includes mass and dimensions monitoring, as well as checking the reduction in pH levels (for the leaching investigation).

The findings of this research can be used for the Finnish waste repositories, by assessing the degradation rate of the used mixes and enhancing the current understanding of these degradation phenomena.

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6 Appendix

6.1 Mix design details

SR mix	
Cement	4.39
Aggregates	9.05
Water	1.94
w/b ratio	0.4
Slump	17.9
Air content	3.6
Initial temperature	26
Weight of concrete	2.22
Density	2.22

SR-SI mix	
Cement	4.07
Silica fume	0.45
Aggregates	9.35
Water	1.99
w/b ratio	0.44
Slump	13.4
Air content	3.8
Initial temperature	26.2
Weight of concrete	2.24
Density	2.24

KA-mix	
Cement	4.52
Aggregates	9.31
Water	1.99
w/b ratio	0.44
Slump	18.55
Air content	3.8
Initial temperature	26.1
Weight of concrete	2.2
Density	2.2

KB-mix	
Cement	4.52
Aggregates	9.31
Water	1.99
w/b ratio	0.44
Slump	19.15
Air content	3.4
Initial temperature	27.2
Weight of concrete	2.17
Density	2.17

KB-SI-mix	
Cement	4.07
Silica fume	0.45
Aggregates	9.31
Water	1.99
w/b ratio	0.44
Slump	15.25
Air content	3.6
Initial temperature	25.8
Weight of concrete	2.16
Density	2.16

6.2 Details of the exposure solutions

The chemicals used for the mix solutions were obtained from fi.vwr.com [34]. The provided datasheets in the following subsections were taken from their website.

6.2.1 Ammonium nitrate

SECTION 2: Hazards identification

2.1 Classification of the substance or mixture

2.1.1 Classification according to Regulation (EC) No 1272/2008 [CLP]

Hazard classes and hazard categories	Hazard statements
Oxidising solid, category 3	H272
Eye irritation, category 2	H319

2.2 Label elements

2.2.1 Labelling according to Regulation (EC) No. 1272/2008 [CLP]

Hazard pictograms



Signal word: Warning

Hazard statements	
H272	May intensify fire; oxidiser.
H319	Causes serious eye irritation.

Precautionary statements	
P210	Keep away from heat, hot surfaces, sparks, open flames and other ignition sources. No smoking.
P220	Keep/Store away from clothing/combustible materials.
P280	Wear protective gloves/protective clothing/eye protection/face protection.
P305+P351+P338	IF IN EYES: Rinse cautiously with water for several minutes. Remove contact lenses, if present and easy to do. Continue rinsing.

2.3 Other hazards

This substance does not meet the PBT/vPvB criteria of REACH, Annex XIII.

This product does not contain a substance that has endocrine disrupting properties.

SECTION 3: Composition / information on ingredients

3.1 Substances

Substance name:	Ammonium nitrate
Molecular formula:	(NH ₄)NO ₃
Molecular weight:	80.04 g/mol
CAS No.:	6484-52-2

6.2.2 Epsom salt



Material	6527.1000
Material description	Magnesium sulphate heptahydrate
Grade	Ph.Eur./USP/BP
CAS Number	10034-99-8
Molecular formula	MgSO ₄ .7H ₂ O
Molecular mass	246.48

Additional information

Characteristics	Specifications
Appearance	Conforms (see CoA/CoS)
Heavy metals (as Pb)	≤ 10 ppm
Residual solvents	Passes test
Residual metal catalysts (skip lot):	-
As (Arsenic)	≤ 2 ppm
Cd (Cadmium)	≤ 0.1 ppm
Co (Cobalt)	≤ 1 ppm
Hg (Mercury)	≤ 2 ppm
Ni (Nickel)	≤ 6 ppm
Pb (Lead)	≤ 2 ppm
V (Vanadium)	≤ 10 ppm
Conforms to Ph.Eur./USP/BP	Passes test
Ph.Eur./BP specifications:	-
Assay (dried substance) [Ph.Eur./BP]	99.0 - 100.5 %
Identification A	Passes test Ph.Eur./BP
Identification B	Passes test Ph.Eur./BP
Appearance of solution	Passes test Ph.Eur./BP
Acidity or alkalinity	Passes test Ph.Eur./BP
Cl (Chloride) [Ph.Eur./BP]	≤ 300 ppm
Fe (Iron) [Ph.Eur./BP]	≤ 20 ppm
Loss on drying (400°C) [Ph.Eur./BP]	48.0 - 52.0 %
USP specifications:	-
Assay (by ignition) [USP]	98.0 - 102.0 %
Identification A	Passes test USP
Identification B	Passes test USP
Cl (Chloride) [USP]	≤ 0.014 %
Fe (Iron) [USP]	≤ 20 µg/g
Se (Selenium) [USP]	≤ 30 µg/g