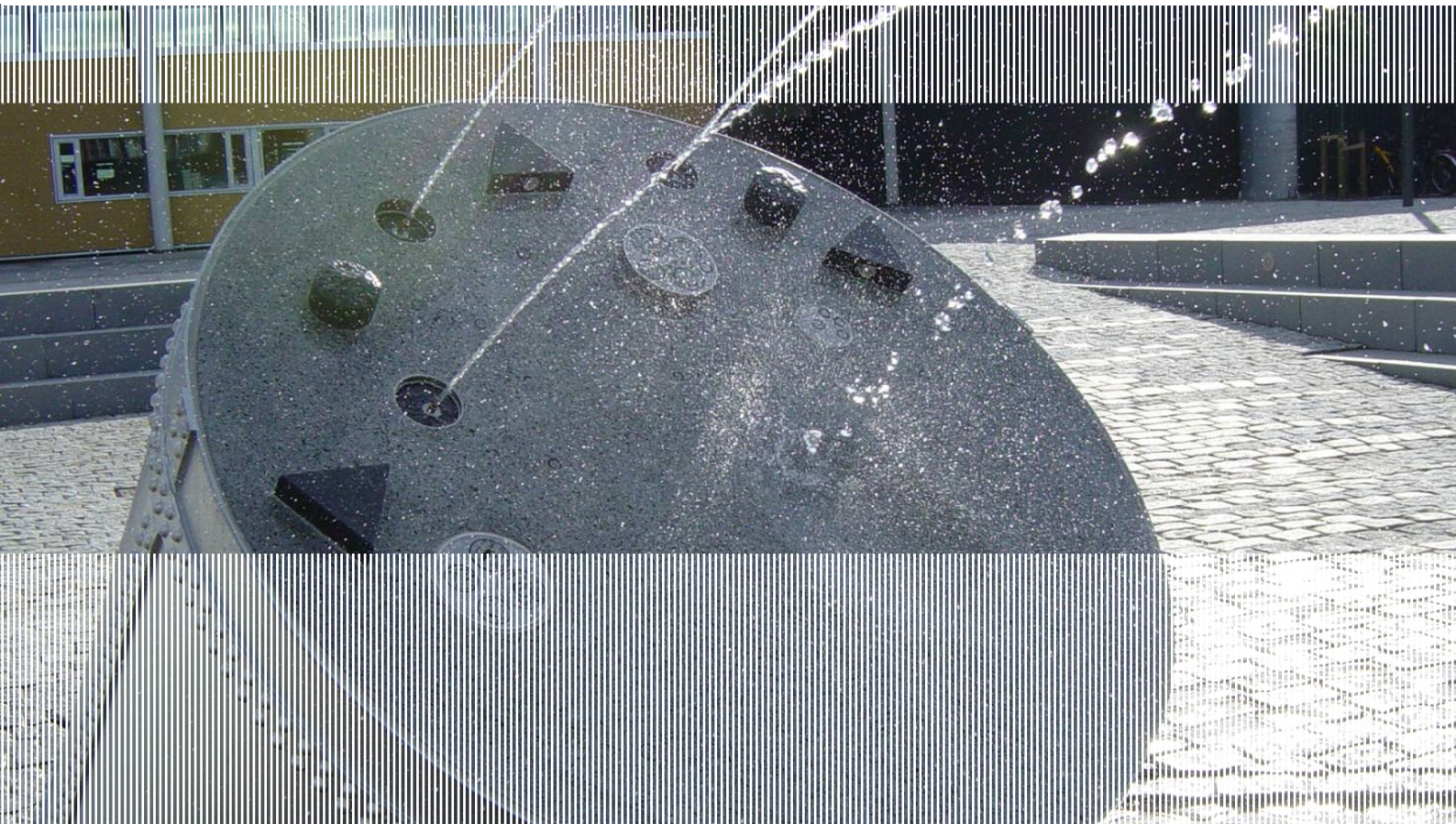


SINTEF Building and Infrastructure Tone Østnor and Harald Justnes

# Durability of mortar with calcined marl as pozzolan 2 year results

COIN Project report 50 – 2014



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FA 1 Environmentally friendly concrete structures

SP 1.1 Binders with low emission and reduced consumption

COIN Project report 50 – 2014

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Keywords:

Cement; pozzolan; clay; marl; admixture

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## Preface

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This study has been carried out within COIN – Concrete Innovation Centre – one of presently 14 Centres for Research based Innovation (CRI), which is an initiative by the Research Council of Norway. The main objective for the CRIs is to enhance the capability of the business sector to innovate by focusing on long-term research based on forging close alliances between research-intensive enterprises and prominent research groups.

The vision of COIN is creation of more attractive concrete buildings and constructions. Attractiveness implies aesthetics, functionality, sustainability, energy efficiency, indoor climate, industrialized construction, improved work environment, and cost efficiency during the whole service life. The primary goal is to fulfil this vision by bringing the development a major leap forward by more fundamental understanding of the mechanisms in order to develop advanced materials, efficient construction techniques and new design concepts combined with more environmentally friendly material production.

The corporate partners are leading multinational companies in the cement and building industry and the aim of COIN is to increase their value creation and strengthen their research activities in Norway. Our over-all ambition is to establish COIN as the display window for concrete innovation in Europe.

About 25 researchers from SINTEF (host), the Norwegian University of Science and Technology – NTNU (research partner) and industry partners, 15–20 PhD students, 5–10 MSc students every year and a number of international guest researchers, work on presently eight projects in three focus areas:

- Environmentally friendly concrete
- Economically competitive construction
- Aesthetics and technical performance

COIN has presently a budget of 200 million NOK over 8 years (from 2007), and is financed by the Research Council of Norway (approximately 40 %), industrial partners (approximately 45 %) and by SINTEF Building and Infrastructure and NTNU (in all approximately 15 %).

For more information, see [www.coinweb.no](http://www.coinweb.no)

Tor Arne Hammer  
Centre Manager

## Summary

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The primary objective of this report was to find out why calcined marl replacing cement in mortars leads to such a high strength and how it possibly can lead to longer term strength increase after the calcium hydroxide generally thought to be required for pozzolanic reactions has been depleted.

The strength of all mortars with calcined marl replacing cement from 0, 20, 35, 50 and 65 vol% increases from one year to years curing. The increase is the highest for the mortar with the highest cement replacement in spite of not containing any calcium hydroxide to sustain a pozzolanic reaction since it was 90 days old. After two years the compressive strength is about equal for mortars where 35, 50 and 65 vol% cement has been replaced by calcined marl, considering the standard deviations.

The strength gain of the mortar with the 65 vol% cement replacement can partly be explained by the higher relative pore refinement of this mortar (-3.5 % since 90 days) compared to mortars with 35 and 50% replacement that only had a relative capillary pore refinement of about 1 % from 90 days to 2 years. However, the reason for the pore refinement is not found.

Some hypotheses are put forward that could explain the strength increase or pore refinement in spite of lack of calcium hydroxide to sustain classical pozzolanic reaction;

- 1) There are two types of CSH in the system. One with high C/S from hydration of cement and one with low C/S from the direct pozzolanic reaction between calcined marl and calcium hydroxide. The one with higher C/S has higher solubility of  $\text{Ca}^{2+}$  and is considered weaker mechanically than the one with lower C/S. Slowly the two different CSHs will equilibrate, and one ends up with an overall stronger CSH with intermediate C/S.
- 2) The solubility of  $\text{Ca}^{2+}$  from CSH with high C/S and pH is so high that it might lead to a direct further pozzolanic reaction with unreacted calcined marl.
- 3) When calcined marl reacts, both silicate and aluminate is released, forming CSH and CAH, as well as possibly intermediate products like  $\text{C}_2\text{ASH}_8$ . Crystalline CAH generally demands a higher atomic Ca/Al than amorphous CSH demand Ca/Si, and the CSH is more flexible in its Ca/Si. The craving for Ca by the aluminate could lead to the de-calcification of inner CSH as observed, as well as lowering the Ca/Si of the overall CSH that will adapt to that by increased polymerization of silicates. Inclusion of aluminate in the CSH structure (i.e. bridging of dimers) will also contribute to a higher degree of polymerization of CSH. In total this may lead to a higher strength binder.

It is recommended to make paste sample for further studies by  $^{27}\text{Al}$  and  $^{29}\text{Si}$  MAS NMR (nuclear magnetic resonance), XRD (X-ray diffraction), or other analytic techniques that otherwise might be disturbed by the aggregate of mortars. The objective would be to find out whether or not extensive polymerization of the CSH gel with inclusion of aluminum in the structure can explain the strength increase for high replacements of cement by calcined marl. It would also reveal any other compounds containing aluminum or silicon. Other analytic techniques might be disturbed by the aggregate of mortars.

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# 1 Introduction

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## 1.1 Objective

The primary objective is to find out why calcined marl replacing cement in mortars leads to such a high strength and how it possibly can lead to longer term strength increase after the calcium hydroxide generally thought to be required for pozzolanic reactions has been depleted. The follow up on sulphate resistance of mortars where cement is replaced by calcined is a secondary objective of this report.

## 1.2 Background

### 1.2.1 Introduction

The cement industry is still the third largest carbon dioxide emitter after energy production for housing and fuel for transport, not because it is a particular polluting material but because it is so popular and abundant in use. On a short time basis, the easiest way to reduce CO<sub>2</sub> emission is to replace clinker in cement making blended cements or to replace cement in the concrete mix with supplementary cementing materials (SCMs) such as blast furnace slag or fly ash from coal fired energy plants (Lothenbach et al. 2011). However, as the trend of making blended cements to produce "greener" construction materials increases, there is a need to look for alternative pozzolans since the common SCMs may be used up and become scarce. Marl is clay containing substantial amounts of calcium carbonate. It is not suitable for use by the traditional clay industry for production of burnt clay products (e.g. bricks and light weight aggregate) since it will form CaO after burning, that can lead to "pop outs" in reaction with water during service. Marl is largely an exploited resource and in the calcined state it is shown by Justnes et al. (2011) and Danner et al. (2012) to be an effective pozzolan. A part of the efficiency of calcined marl in enhancing strength is that the calcium aluminate hydrates formed by the pozzolanic reaction may react further with remaining calcium carbonate after the calcination process to form calcium carboaluminate hydrates binding even more water. The synergic reaction between alumina containing pozzolan and limestone was first thoroughly studied by De Weerd et al. (2008, 2009, 2010, 2011 a, b) for the system fly ash-limestone and later by Antoni et al. (2012) for the metakaolin-limestone system. Calcined marl, along with other natural clays, is probably one of the few pozzolans sufficiently abundant world-wide to serve the needs of the cement and concrete industry as a whole to reduce the clinker factor. Achieving and maintaining sufficient strength is a requirement for using it in buildings, but more important for structures exposed to the natural environment is how it will affect the durability.

### 1.2.2 Previous work

Based on results and observations in test programme on durability of mortars with calcined marl as pozzolan in COIN report No. 49 "Durability of mortar with calcined marl as pozzolan", further study was recommended on longer term strength and durability.

Between 90 days and 1 year of curing at 90 % RH and 20 °C the compressive strength kept on rising despite the absence of calcium hydroxide.

The hypotheses for the strength gain without calcium hydroxide can be several;

- 1) The mortar with high replacements of cement has more unreacted cement than the reference at 90 days that can further hydrate and give more calcium hydroxide for further pozzolanic reaction.
- 2) There are two types of CSH in the system – one with high C/S from hydration of cement and one with low C/S from the direct pozzolanic reaction between calcined marl and calcium hydroxide. The one with higher C/S has higher a solubility of Ca<sup>2+</sup> and is considered weaker mechanically than the one with lower C/S. Slowly,

the two different CSHs will equilibrate, and one ends up with an overall stronger CSH with intermediate C/S.

- 3) The solubility of  $\text{Ca}^{2+}$  from CSH with high C/S and pH is so high that one might have a direct further pozzolanic reaction with unreacted calcined marl.
- 4) The silicate anions of in particular CSH with low C/S will polymerize over time creating longer chain lengths of the CSH which may lead to even higher strength.

The present report discusses the compressive and flexural strength up to two years of curing, and the microstructure in general. The long term durability tests of mortar samples stored for two years submerged in sulphate solution at 20 °C and 5 °C are reported as well, along with the capillary absorption and measurement of the electrical resistivity on samples cured at 90 % RH and 20 °C.



## 2 Materials

---

### 2.1 Materials

The marl (or rather calcareous clay) consists of about 70 % of a mixture of clay minerals and about 20 % calcium carbonate, the remainder being quartz and feldspars. The marl was provided by Saint-Gobain Weber who calcined it in a rotary kiln close to industrial conditions. About 20 % of the calcium carbonate survived the calcination process, leaving about 4 % CaCO<sub>3</sub> of calcined marl mass. The calcined marl was ground to  $d_{50} = 7 \mu\text{m}$ . Ordinary Portland cement (CEM I 42.5R according to NS-EN 197-1) produced by Norcem, Brevik, Norway, was used for all the mortar mixes. The aggregate used was 0–8 mm crushed granite rock from Årdal, Norway. The superplasticizer used was Dynamon SP 130 supplied by Mapei AS Norway, for details see Table 1.

### 2.2 Mortar mix design

The mortars were made with 0, 20, 35, 50 and 65 % replacement of cement by calcined marl by volume to secure a constant volume of binder. The consistency of fresh mortar was determined using a flow table. The aim was to obtain a w/b ratio of 0.5 in all the mortars while maintaining the flow by varying the amount of superplasticizer in the range 0.1–0.8 % (of binder weight). The flow of all mortars was within  $\pm 5 \%$  of the reference. The mortar mixes were cast in 40·40·160 mm moulds and 100·200 mm cylinders. After 24 hours the prisms and cylinders were removed from the moulds and stored in a cabinet at 90 % RH and  $23 \pm 2 \text{ }^\circ\text{C}$ .

**Table 1**  
Amount of superplasticizer used in the different mixes

Mix	Amount of Dynamon SP-130 (% binder weight)
M0 (Ref-OPC)	–
M20	0.2
M35	0.3
M50	0.5
M65	0.9

### 3 Experimental

---

#### 3.1 Strength measurements

The compressive and flexural strength were measured on the 160·40·40 mm prisms at 1, 3, 7, 28, 90 days, one and two year of curing at 90 % RH and 23 °C according to NS-EN 196-1. There were three parallel prisms for the flexural strength and six parallels for the compressive strength, since it was measured on the end-pieces of the three prisms.

#### 3.2 Capillary suction measurement

The capillary suction technique was performed on four parallel 20 mm slices sawn from cast cylinders from each mortar mixture. The specimens were dried at 105°C drying before capillary suction measurements.

The procedure consists of 6 important steps for the specimen:

1. Drying the specimen to constant weight at 105°C
2. Capillary suction for 4 days with weight monitoring
3. Water saturation by submersion 3 days in water 1 atm
4. Pressure saturation by submersion 3 days in water at 80 atm
5. The outer volume ( $V$ ) is recorded by differential weighing the specimen under water and saturated surface dry in air according to the principle of Archimedes.
6. Drying the specimen to constant weight at 105°C

From these 6 steps was the initial moisture content, total porosity ( $\epsilon_{\text{tot}}$ ), capillary porosity ( $\epsilon_{\text{cap}}$ ), entrained air volume ( $\epsilon_{\text{air}}$ ), average density of mortar solids ( $\rho_s$ ) and dry density of mortar ( $\rho_d$ ) calculated, as more thoroughly described by Justnes et al (2002).

#### 3.3 Electrical resistivity measurement

The resistivity measurements were performed at 20 °C on the 20 mm discs from the capillary suction experiment after they were water saturated. Conducting gel was applied on the end surfaces ensuring electrical contact before placing two steel plates towards them and the resistivity was measured using a multimeter in the AC mode and a frequency of 1 MHz

#### 3.4 Expansion measurements due to sulphate attack

Two sets of three prisms with embedded studs from each mortar mix were submersed in 5 % sodium sulphate solution at 20 °C and 5 °C respectively, and the prisms were weighted above and below water for calculation of the volume, followed by uniaxial length measurements each month. The prisms were submerged in water for three days before measuring their length as a starting point before sulphate exposure. The 5 °C test was carried out in case thaumasite was formed by the carbonate in the calcined marl.

#### 3.5 Scanning electron microscope (SEM) investigations

Samples from mortar mixes with 35, 50 and 65 % replacement of cement with calcined marl cured for two years were cast in epoxy resin, plane polished to achieve a cross-section of the material and sputtered with carbon. The instrument used in this study was JEOL JXA – 8500F Electron Probe Micro analyser. The samples were analysed in the BSE (back scattered electron) mode, where dense compounds and/or compounds composed of heavy elements appear bright (e.g. unreacted  $C_4AF$  mineral in cement), and compounds of low

density and/or composed of elements with low atomic number appears dark (e.g. CSH). Details of interest were first checked for elements by EDS (energy dispersive spectra) semi-quantitatively, and further quantitatively by WDS (wave length dispersive spectra) for compositional determination.

### **3.6 Thermo gravimetric analysis (TGA)**

The samples were analysed by thermo gravimetric analysis and simultaneous differential thermal analyses (TGA/SDTA) with a Mettler Toledo TGA/SDTA 851. About 150 mg of the sample was weighed into aluminium oxide crucibles. The sample was heated from 40°C to 900°C with a heating rate of 10 °C/min. The purge gas was nitrogen (N<sub>2</sub>) with a flow of 50 ml/min.

The mass loss up to 400 °C is considered to be the dehydration of various hydration products. The mass loss from 450 °C to 550°C is caused by the thermal decomposition of calcium hydroxide (CH). The total mass loss from 105 °C to 550 °C is taken as total chemical bound water. The mass loss in the higher temperature range from about 600 °C to 800 °C is attributed to the decomposition of carbonates.

The lower and upper limits of these temperature intervals were determined from the DTG (the first derivative) curve. The first interval starts at 40 °C and ends at the beginning of the decomposition peak of calcium hydroxide in the DTG curve. The third interval starts where the decomposition peak of calcium hydroxide flattens out (about 600 °C) and reaches up to 900 °C.

### **3.7 X-ray diffraction analysis (XRD)**

Samples from mortar mixes with 35, 50 and 65 % replacement of cement with calcined marl cured for two years at 90 % RH and 20 °C were analysed by AXS D8 focus X-ray diffractometer. Prior to these analyses the samples were dried in a desiccator at 30 % RH using calcium chloride as a drying agent. The dry powders were submitted to an angular scan between 5° and 75° 2 $\theta$  with a step size of 0.06° and a step time of 1.0 s.

## 4 Results

### 4.1 Strength measurements

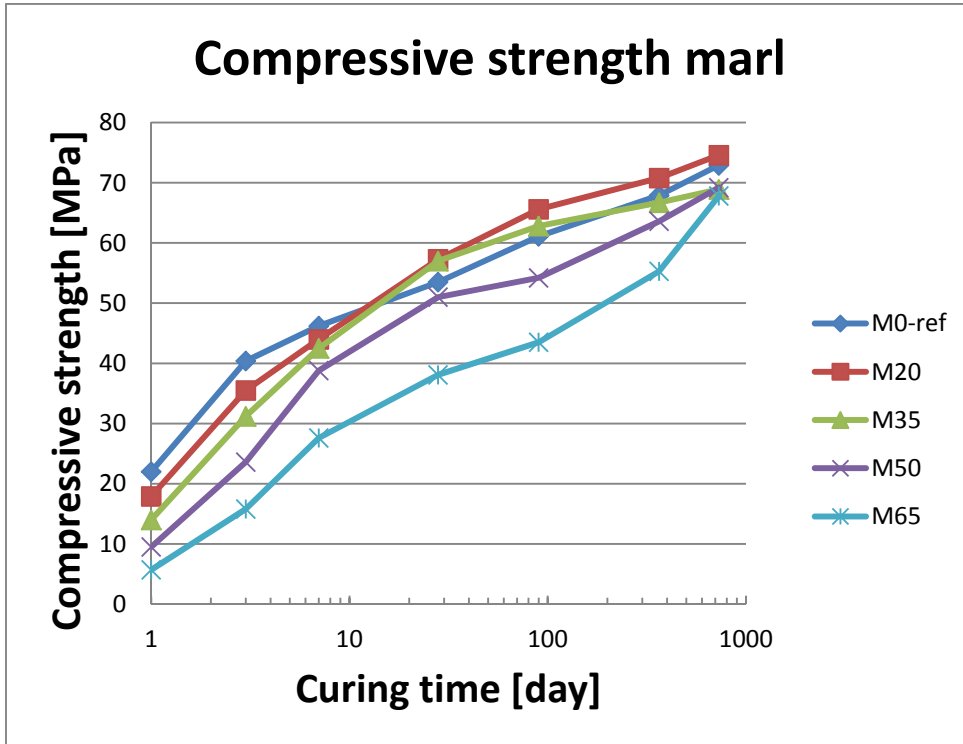
The values of the compressive strengths with their standard deviations at different ages are listed in Table 2, while the compressive strength evolution for all mortars as a function of time on a logarithmic scale is plotted in Figure 1. Corresponding values for flexural strength are listed in Table 3 and plotted in Figure 2, respectively.

**Table 2**  
**Compressive strengths as a function of curing time for mortars with calcined marl replacing cement at various volume fractions.**

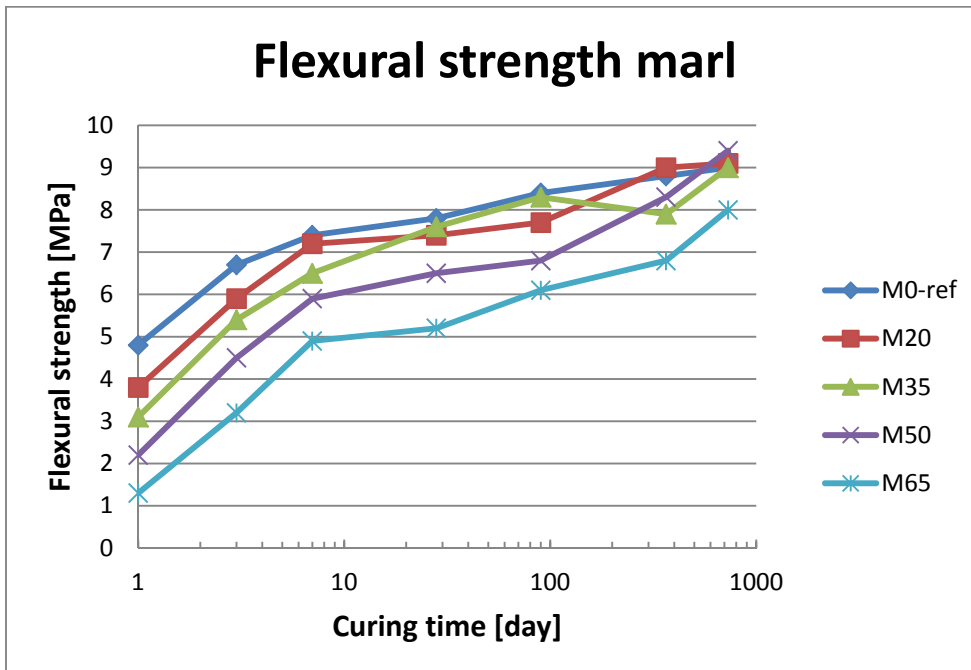
Cement replacement by marl [%]	Average compressive strength $\pm$ standard deviation [MPa] at ages [days]						
	1	3	7	28	90	365	730
0 (Ref)	22.0 $\pm$ 0.3	40.4 $\pm$ 1.0	46.2 $\pm$ 1.0	53.5 $\pm$ 0.6	61.1 $\pm$ 1.6	67.9 $\pm$ 0.9	72.9 $\pm$ 2.4
20	17.9 $\pm$ 0.4	35.5 $\pm$ 0.5	44.0 $\pm$ 1.0	57.3 $\pm$ 1.3	65.6 $\pm$ 0.9	70.8 $\pm$ 1.3	74.6 $\pm$ 0.8
35	14.0 $\pm$ 0.1	31.2 $\pm$ 0.4	42.5 $\pm$ 0.6	57.0 $\pm$ 0.8	62.8 $\pm$ 1.6	68.4 $\pm$ 1.0	68.9 $\pm$ 2.9
50	9.5 $\pm$ 0.2	23.6 $\pm$ 0.3	38.8 $\pm$ 0.4	51.0 $\pm$ 1.2	54.2 $\pm$ 1.1	63.6 $\pm$ 1.3	69.2 $\pm$ 1.5
65	5.7 $\pm$ 0.1	15.8 $\pm$ 0.3	27.6 $\pm$ 0.1	38.1 $\pm$ 1.2	43.5 $\pm$ 1.0	55.9 $\pm$ 1.3	67.8 $\pm$ 0.7

**Table 3**  
**Flexural strengths as a function of curing time for mortars with calcined marl replacing cement at various volume fractions.**

Cement replacement by marl [%]	Average flexural strength $\pm$ standard deviation [MPa] at ages [days]						
	1	3	7	28	90	365	730
0 (Ref)	4.8 $\pm$ 0.1	6.7 $\pm$ 0.4	7.4 $\pm$ 1.0	7.8 $\pm$ 0.3	8.4 $\pm$ 0.4	8.8 $\pm$ 0.4	9.0 $\pm$ 0.5
20	3.8 $\pm$ 0.3	5.9 $\pm$ 0.2	7.2 $\pm$ 0.2	7.4 $\pm$ 0.2	7.7 $\pm$ 0.6	9.0 $\pm$ 0.5	9.1 $\pm$ 0.2
35	3.1 $\pm$ 0.1	5.4 $\pm$ 0.2	6.5 $\pm$ 0.2	7.6 $\pm$ 0.5	8.3 $\pm$ 0.2	7.9 $\pm$ 0.2	9.0 $\pm$ 0.5
50	2.2 $\pm$ 0.1	4.5 $\pm$ 0.2	5.9 $\pm$ 0.3	6.5 $\pm$ 0.3	6.8 $\pm$ 0.4	8.3 $\pm$ 0.9	9.4 $\pm$ 0.3
65	1.3 $\pm$ 0.1	3.2 $\pm$ 0.1	4.9 $\pm$ 0.2	5.2 $\pm$ 0.4	6.1 $\pm$ 0.3	6.8 $\pm$ 0.4	8.2 $\pm$ 0.4



**Figure 1**  
Compressive strength as a function of curing time for mortars where cement has been replaced by 0 (M0), 20 (M20), 35 (M35), 50 (M50) and 65 (M65) calcined marl.



**Figure 2**  
Flexural strength as a function of curing time for mortars where cement has been replaced by 0 (M0), 20 (M20), 35 (M35), 50 (M50) and 65 (M65) vol % calcined marl.

## 4.2 Capillary suction measurements

The average results extracted from the capillary suction experiments on four samples per mix are listed in Table 4, including their standard deviations.

**Table 4**  
**Mortar properties extracted from the capillary suction experiments at two years**

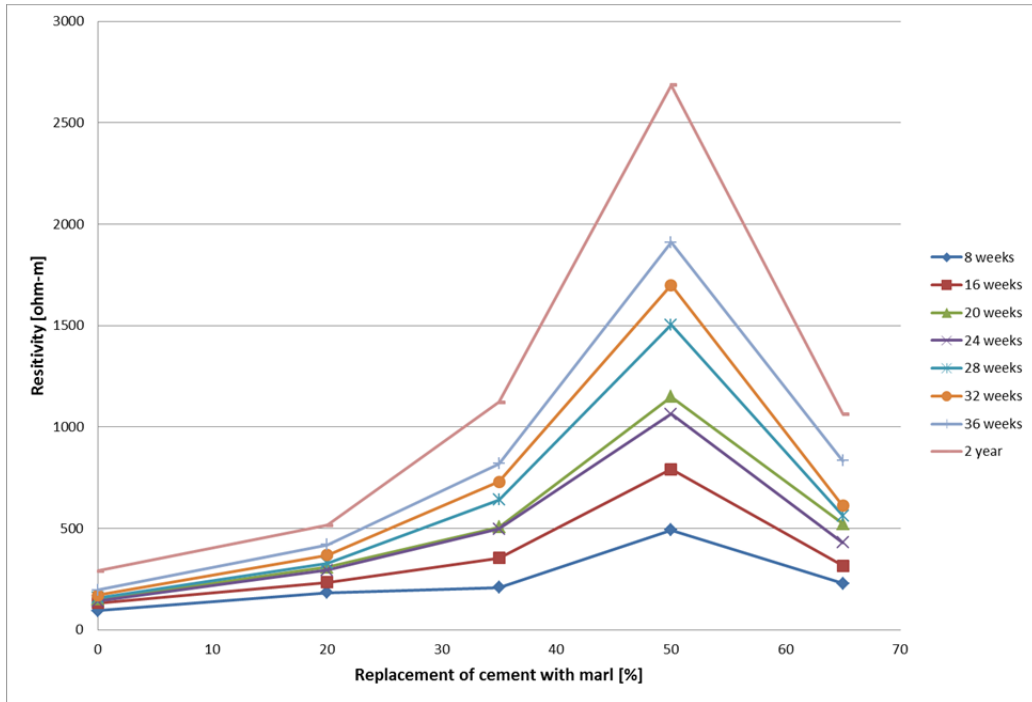
Cement replacement by marl [%]	Capillary porosity $\epsilon_{cap}$ [vol%]	Air content $\epsilon_{air}$ [vol%]	Average density of solids, $\rho_s$ [kg/m <sup>3</sup> ]	Dry density $\rho_d$ [kg/m <sup>3</sup> ]
0 (Reference)	16.8±0.3	3.2±0.2	2 660±3	2 127±10
20	19.0±0.1	2.5±0.1	2 692±3	2 114±7
35	20.4±0.1	2.2±0.0	2 706±2	2 093±4
50	20.9±0.2	2.2±0.1	2 683±13	2 063±15
65	21.8±0.1	2.1±0.1	2 680±2	2 040±2

## 4.3 Electrical resistivity measurements

The electrical resistivity of the different mortars as a function of cement replacement by calcined marl is given in Table 5 and plotted in Figure 3 for different storage times in lime water.

**Table 5**  
**Electrical resistivity of mortars with cement replaced by calcined marl as a function of curing time**

Cement replacement by marl [%]	Resistivity [ohm-m]							
	8 weeks	16 weeks	20 weeks	24 weeks	28 weeks	32 weeks	36 weeks	2 years
0 (Ref)	93±6	132±35	151±25	141±26	158±17	171±4	196±22	290±61
20	182±20	233±27	309±32	294±27	329±29	368±35	420±42	515±43
35	208±12	355±15	506±12	498±10	640±6	730±27	819±25	1 123±18
50	492±34	791±37	1 150±44	1 064±35	1 504±35	1 699±25	1 909±46	2 686±36
65	229±9	315±13	522±19	430±19	562±16	611±24	833±25	1 065±46



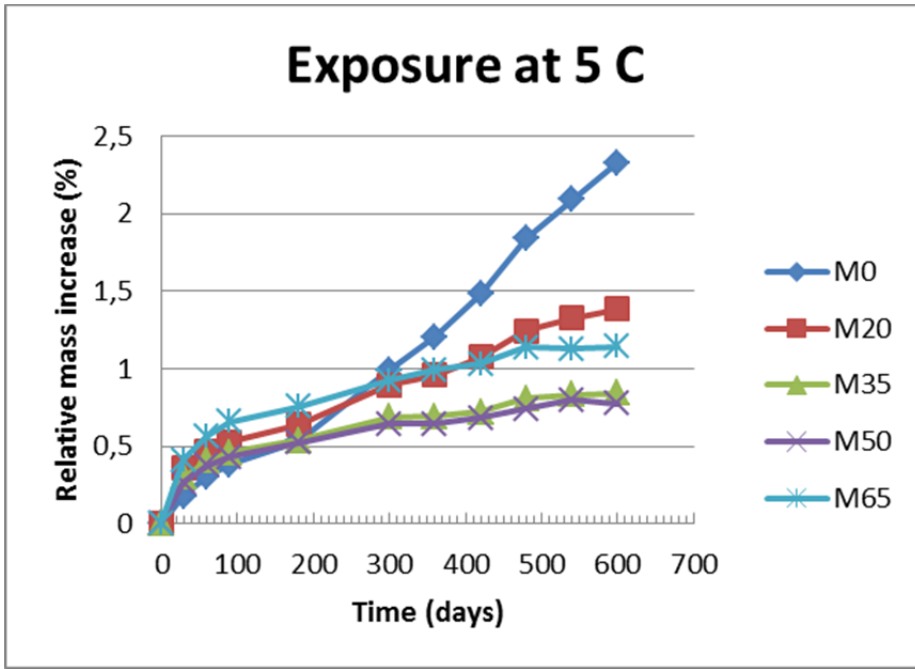
**Figure 3**  
Electrical resistivity as a function of curing time for mortars where calcined marl is replacing different volume fractions of cement.

#### 4.4 Expansion measurements due to sulphate attack

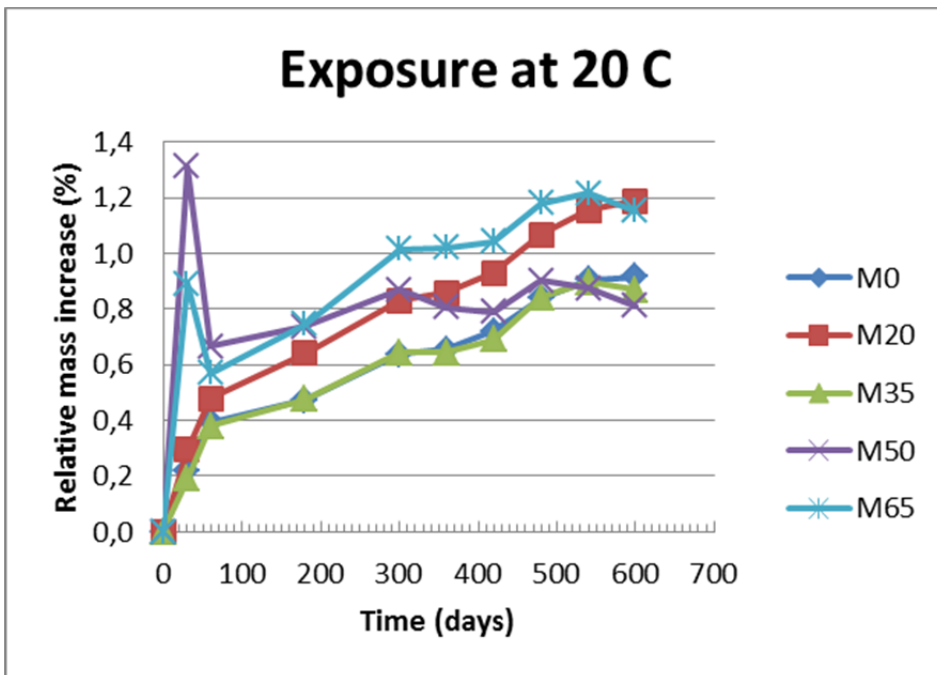
The linear expansions of the mortar prisms at 5°C and 20 °C after twenty months are listed in Table 6, while the mass increase of the mortars exposed to 5 % Na<sub>2</sub>SO<sub>4</sub> solution for twenty months at 5 °C and 20 °C are plotted in Figure 4 and 5, respectively.

**Table 6**  
Linear expansion (%) of mortar prisms where cement is replaced with various volume fractions of calcined marl after 20 months of exposure to 5 % Na<sub>2</sub>SO<sub>4</sub> at 5 °C and 20 °C

Cement replacement by marl (%)	Linear expansion (%) with standard deviations (three parallels)	
	At 5°C	At 20°C
0 (Reference)	0.0148±0.0038	0.0197±0.0019
20	0.0163±0.0023	0.0135±0.0020
35	0.0100±0.0017	0.0053±0.0019
50	-0.0152±0.0343	-0.0012±0.0056
65	0.0113±0.0010	-0.0077±0.0150



**Figure 4**  
Relative mass increase of mortars where cement is replaced with 0 (M0), 20 (M20), 35 (M35), 50 (M50) and 65 (M65) vol% calcined marl as a function of time when exposed to 5 % Na<sub>2</sub>SO<sub>4</sub> solution at 5 °C



**Figure 5**  
Relative mass increase of mortars where cement is replaced with 0 (M0), 20 (M20), 35 (M35), 50 (M50) and 65 (M65) vol% calcined marl as a function of time when exposed to 5 % Na<sub>2</sub>SO<sub>4</sub> solution at 20 °C



#### 4.5 Scanning electron microscope (SEM) investigations

Selected images are shown from the microstructure study by SEM in Figure 6–8, where an overview of samples after 2 years are compared to samples after 90 days of curing.

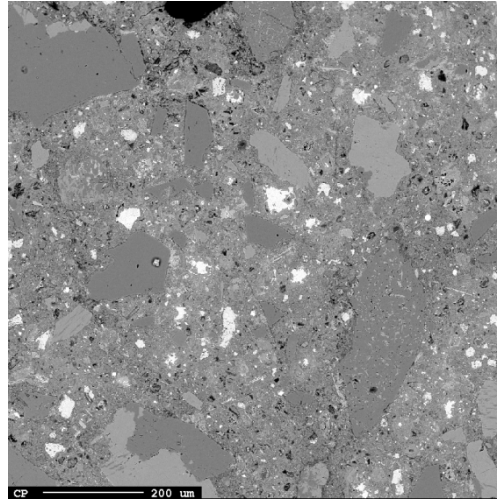
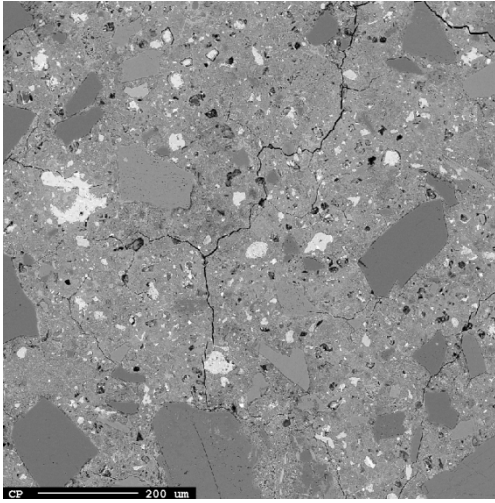


Figure 6 M35 after 90 days (left) and 2 years (right) of curing at 90 % RH and 23 °C

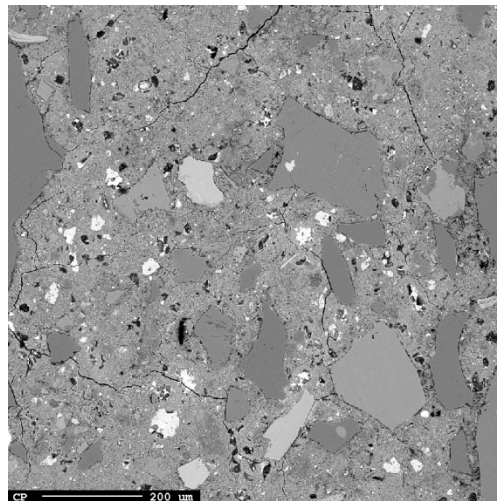
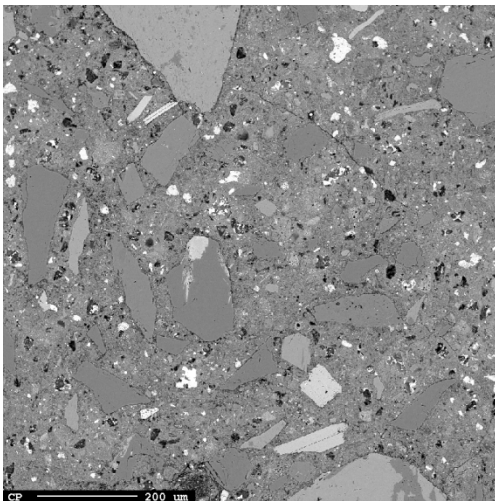


Figure 7 M50 after 90 days (left) and 2 years (right) of curing at 90 % RH and 23 °C

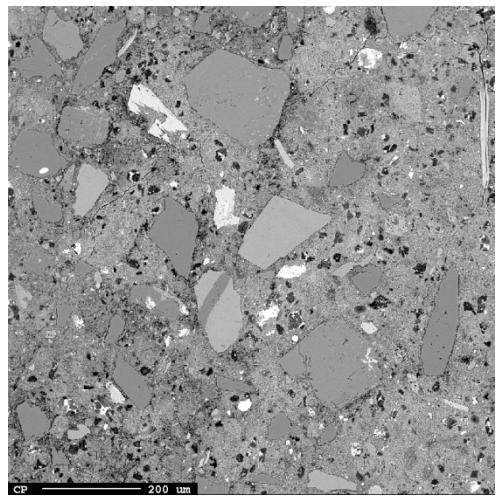
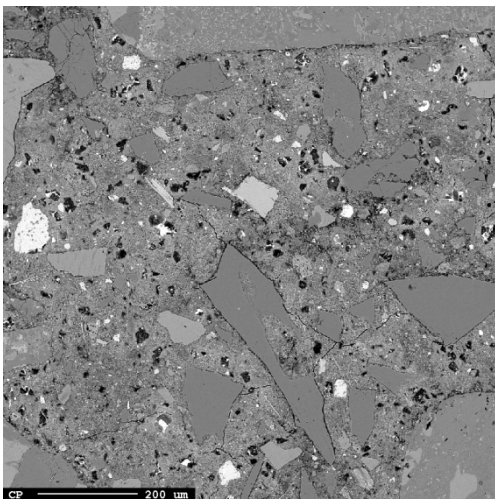
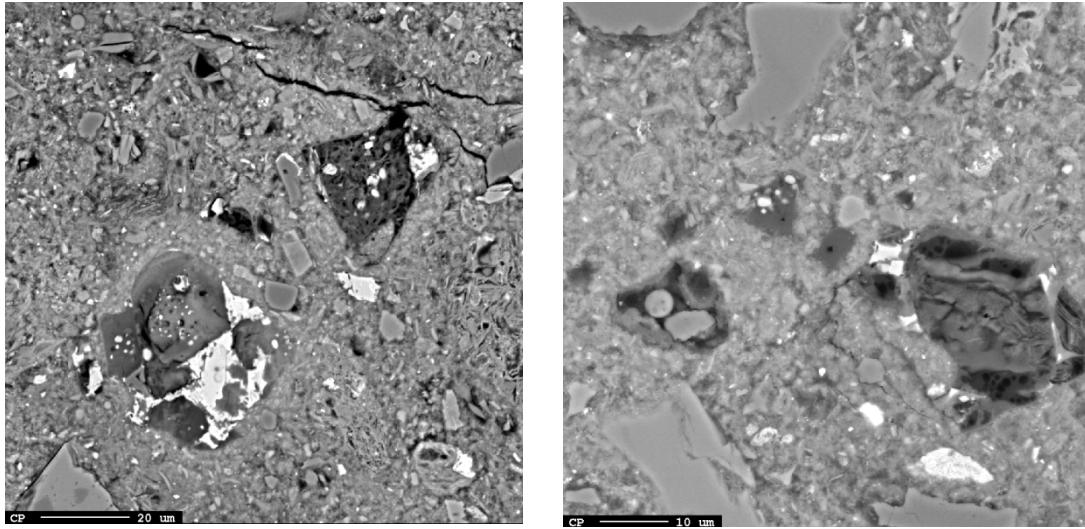


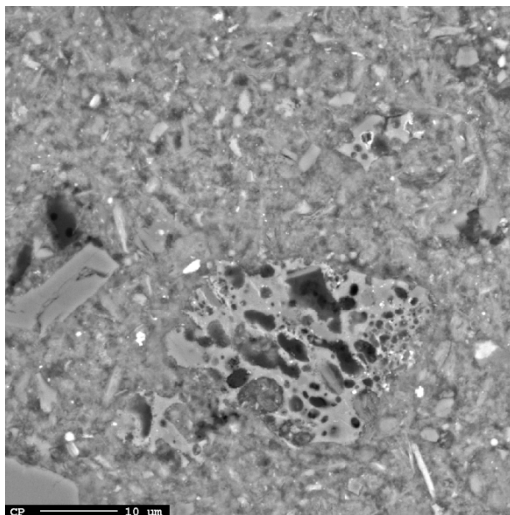
Figure 8 M65 after 90 days (left) and 2 years (right) of curing at 90 % RH and 20 °C

The chemical composition of the binder for mortars with 35, 50 and 65 % cement replacement was compared after 90 days and 2 years of curing by making WDS analyses of 12 selected areas represented by a circle of 10  $\mu\text{m}$  for each sample. The average of each element analysed and their standard deviations for each mortar samples are given in Table 7.

Many of the "dark spots" appearing in the overview images (e.g. Figure 8) is actually inner CSH products in former cement grains that has been partly decalcified. Close-ups of such "dark spots" after 90 days and 2 years curing of mortar where 65 % cement is replaced by calcined marl are shown in Figure 9. The semi quantitative results by EDS of the "dark matter" are listed in Table 8. Figure 10 shows the remains of calcined marl particle not yet reacted.



**Figure 9**  
Mortar with 65 vol% replaced by calcined marl (M65) after 90 days (left) and 2 years (right), showing a close-up of "dark spots" consisting of partly decalcified inner products. The analysis of the one to the right in the right image is given in Table 7.



**Figure 10**  
Image of M65 after 2 years of curing showing a partly reacted clay particle

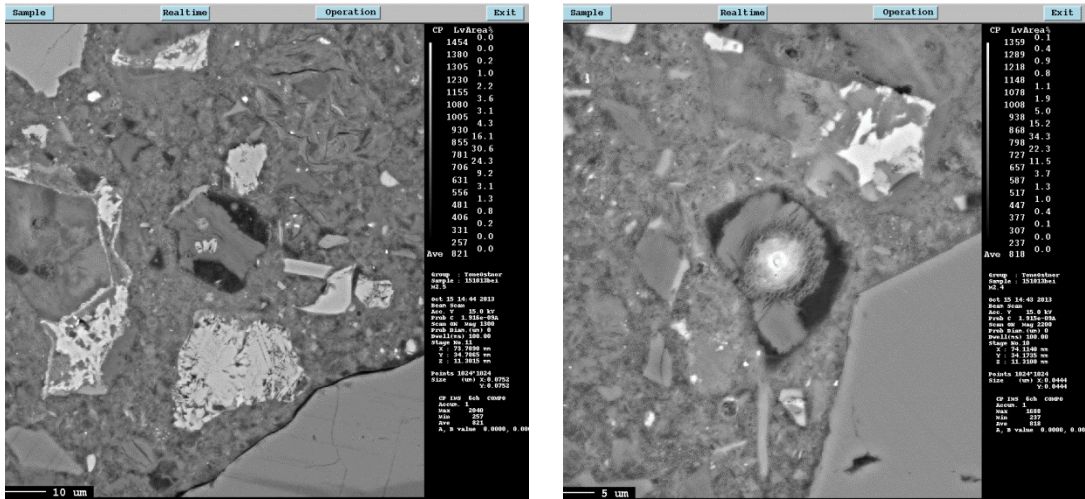
**Table 7**  
**Chemical composition of the binder for mortars with 35, 50 and 65 % cement replacement analysed with WDS after 90 days and 2 years of curing**

Cement replacement by marl (%)							
Atom mass (% with standard deviations (12 points))		M35 at 90d	M35 at 2y	M50 at 90d	M50 at 2y	M65 at 90d	M65 at 2y
	Na	0.53±0.24	0.48±0.28	0.64±0.28	0.59±0.33	0.88±0.37	0.70 ± 0.11
	O	65.04±1.46	66.37±1.69	64.00±1.23	66.64±1.09	63.52±0.89	62.91±1.77
	S	0.57±0.25	0.45±0.18	0.49±0.23	0.46±0.17	0.51±0.16	0.43±0.23
	Fe	1.53±0.42	1.39±0.54	1.83±0.40	1.40±0.34	1.68±0.41	1.94±0.44
	Mg	1.07±0.25	1.07±0.41	1.13±0.26	0.94±0.27	0.97±0.41	1.30±0.37
	K	0.95±0.48	0.85±0.49	0.97±0.37	1.53±1.26	1.03±0.27	1.06±0.33
	Al	3.95±1.07	3.82±1.47	4.47±0.91	4.30±0.82	5.60±1.24	5.88±0.87
	Ca	13.73±3.23	12.58±3.59	11.33±2.97	11.07±2.85	10.74±1.75	9.82±2.64
	Si	12.64±2.87	13.00±2.48	15.14±2.38	13.07±2.03	15.06±1.47	15.96±2.55
Ca/Si	1.19±0.52	1.04±0.41	0.79±0.29	0.88±0.30	0.73±0.19	0.65±0.26	

**Table 8**  
**Chemical composition of hydration product in a "dark spot" of mortar where 65 vol% cement has been replaced by calcined marl.**

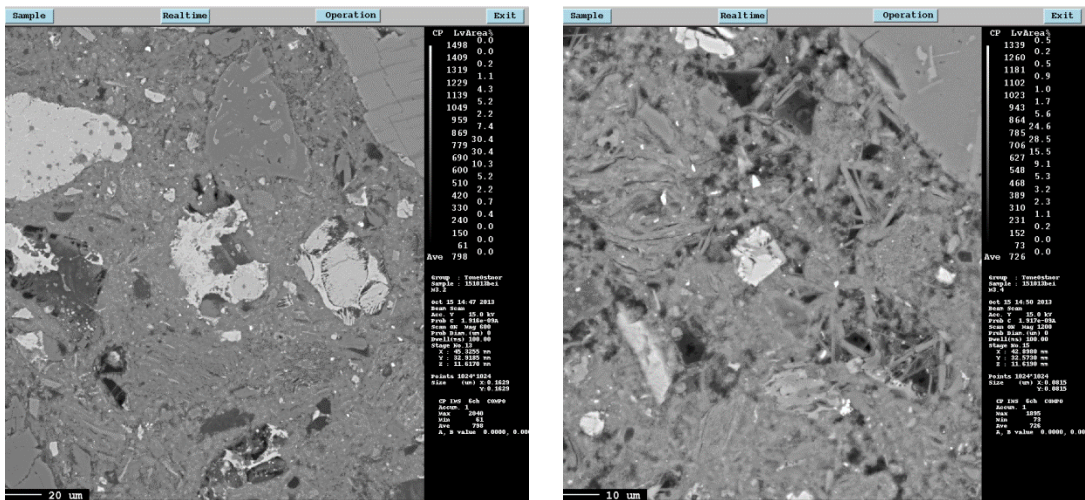
M65 at 2 years	
Element	Atom mass (%)
Mg	5.63
Al	6.57
Si	17.00
K	0.47
Ca	13.06
Fe	2.10
O	54.39

The BSE images of mortar with 35 vol% cement replacement of calcined marl after 2 years of curing in Figure 11 show to the left in the left images a cement grain that has reacted through and that is NOT decalcified with an inner CSH product with Ca/Si = 1.56. The products in the pores in the centre of images in Figure 11 are calcium aluminate hydrates (CAH), and the one to the right has composition 16.5 Ca, 8.3 Al, 1.1 Si, 0.4 S and 0.4 Fe in atom percent by WDS. A Ca/Al = 2 corresponds to CAH compounds starting with C<sub>4</sub>A in the short-hand notation of cement chemists (i.e. general AFm phases), including calcium monocarboaluminate hydrate.



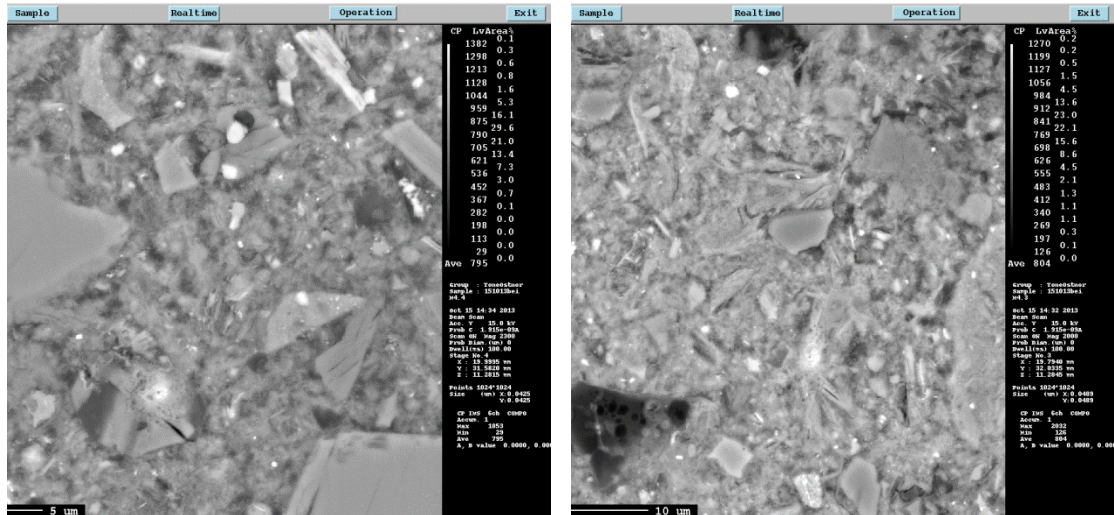
**Figure 11**  
**Images of M35 after 2 years of curing showing CAH hydration products**

The images of the mortar with 50 vol% cement replacement by calcined marl after 2 years of curing in Figure 12 show to the left three partly reacted cement grains with three different stages of decalcified inner CSH products. The image to the right shows a partly reacted clay grain in its upper left corner, an aggregate in the upper right corner and in between needle shaper calcium aluminate hydrate reaction products. The similarity of grey tones illustrates how difficult it is to distinguish the different hydration products (e.g. CSH from CAH).



**Figure 12**  
**BSE images of M50 after 2 years of curing**

The images of the mortar with 65 vol% cement replacement by calcined marl after 2 years of curing in Figure 13 show to the left a close up of the inhomogeneous binder where a CAH in the lower left corner (analysis point seen as a white spot) is analysed to be composed of 18.8 Ca, 11.5 Al, 2.8 Si, 1.3 Fe and other elements < 1 in atom percent by EDS.  $Ca/Al = 1.6$  or  $Ca/(Al+Fe) = 1.5$  corresponds well with the phase  $C_3AH_6$  for instance. The product analysed in the right image is more "blurred" (see white spot lower in middle of image) and is analysed to consist of 14.7 Ca, 13.2 Al, 2.8 Si and other elements < 1 in atom percent by EDS.  $Ca/Al = 1.1$  means a phase starting with  $C_2A$  in the short-hand notation of cement chemists. One possibility is  $C_2AH_8$ , a product generally found in the hydration of calcium aluminate cements (CAC).



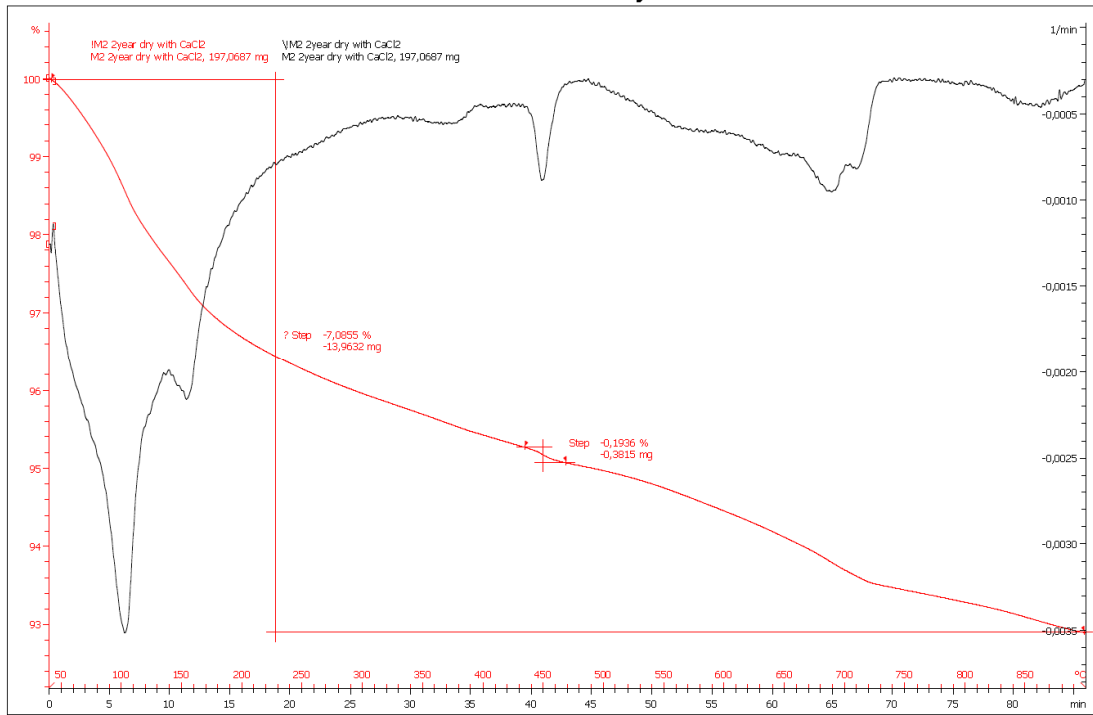
**Figure 13**  
BSE images of M50 after 2 years of curing

#### 4.6 Thermo gravimetric analysis (TGA)

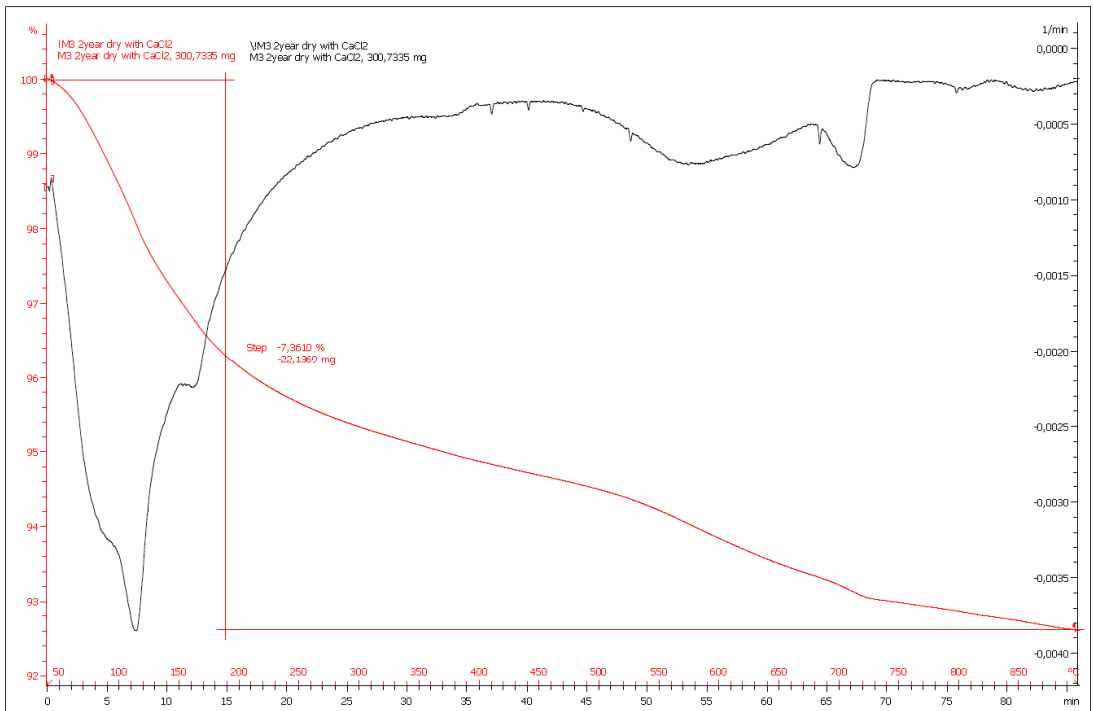
As can be seen from the DTG curves in Table 9 and Figure 14–16 for the mortars with 35, 50 and 65 % cement replacement by calcined marl, respectively, it is only the mix with 35 % replacement level of cement that still has some calcium hydroxide left in the matrix.

**Table 9**  
Calcium hydroxide (%) and total mass loss (%) in marl/cement samples

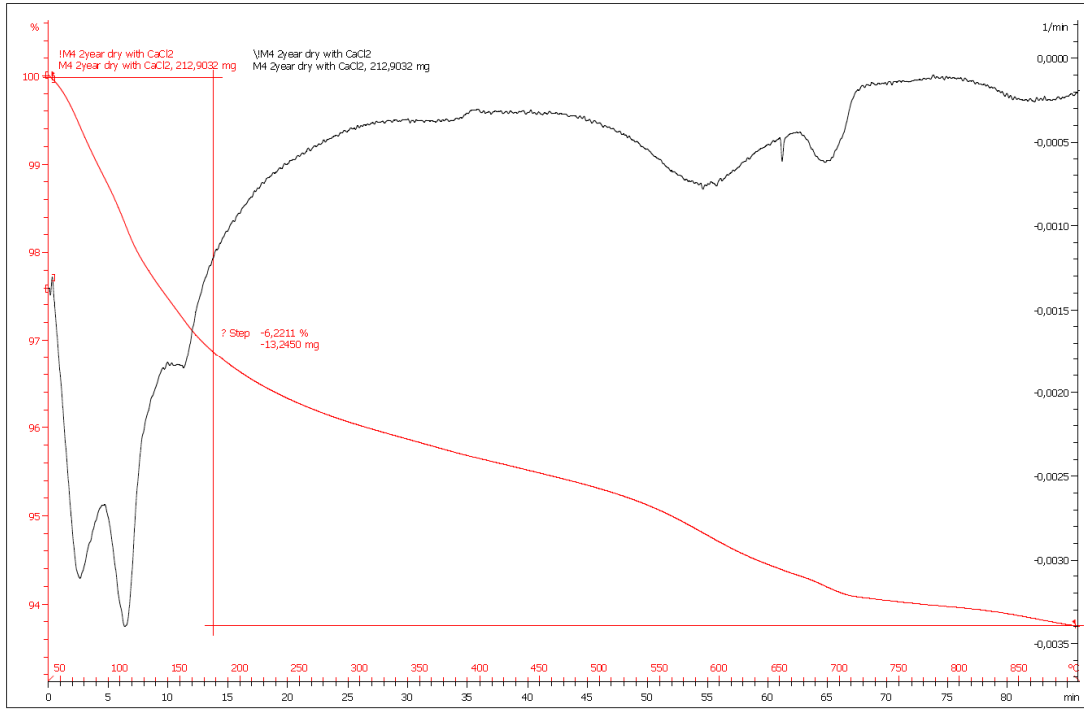
Cement replacement by marl (%)	Calcium hydroxide (%)	Mass loss until 500 °C as bound water (%)	Total mass loss (%)
35	0.8	5.0	7.1
50	–	5.5	7.4
65	–	4.8	6.2



**Figure 14**  
The thermo gravimetry curve (TG) and its derivative (DTG) of mortar where 35 vol% of the cement is replaced with calcined marl



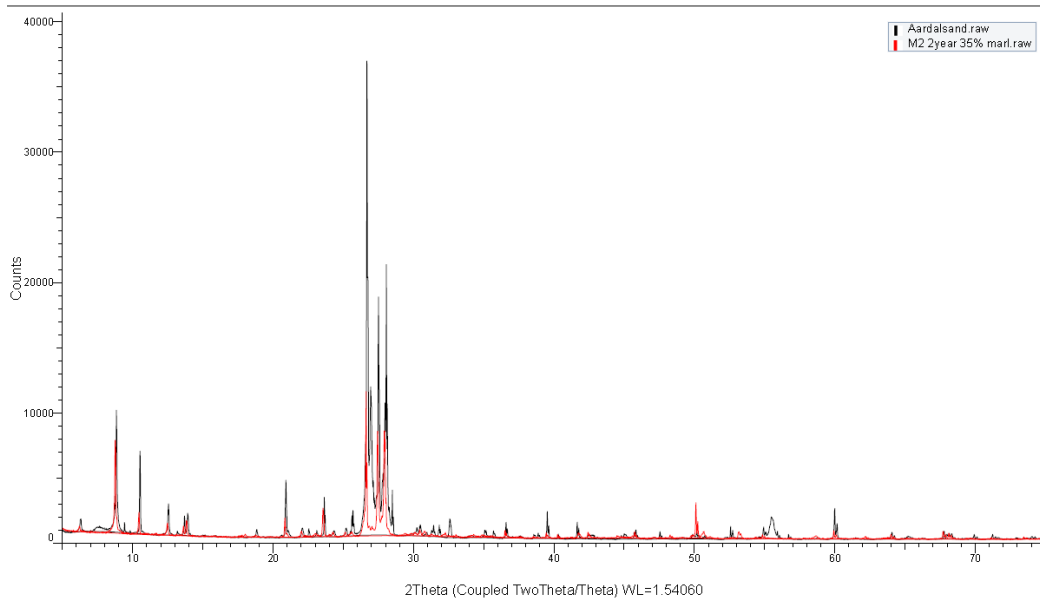
**Figure 15**  
The thermo gravimetry curve (TG) and its derivative (DTG) of mortar where 50 vol % of the cement is replaced with calcined marl



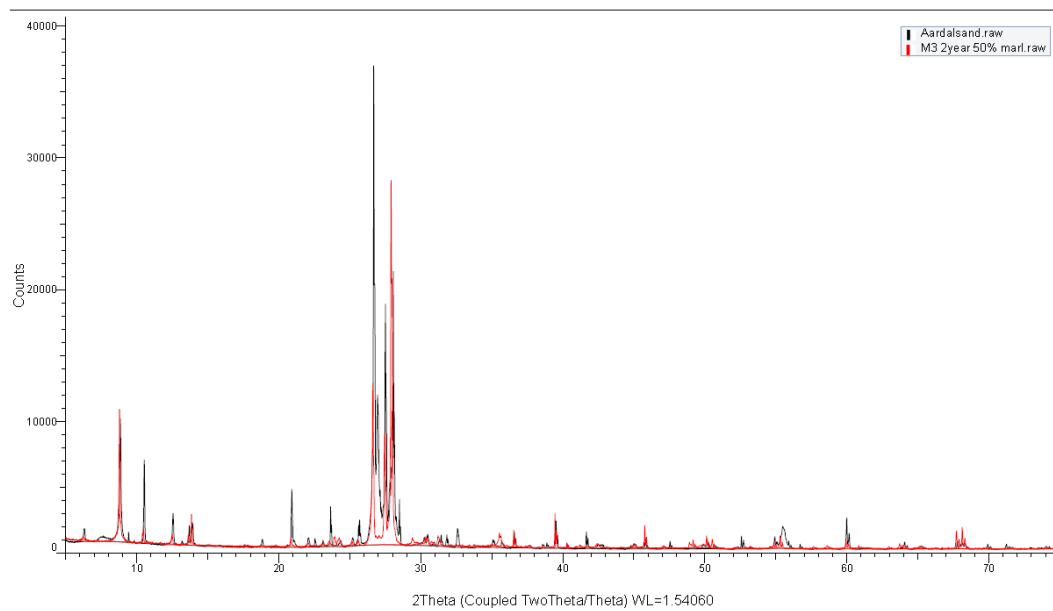
**Figure 16**  
The thermo gravimetry curve (TG) and its derivative (DTG) of mortar where 65 vol% of the cement is replaced with calcined marl

#### 4.7 X-ray diffraction analysis (XRD)

X-ray diffractograms of ground mortars with 35, 50 and 65 vol% replacement of cement after 2 years of curing are given in Figure 17, Figure 18 and Figure 19, respectively.

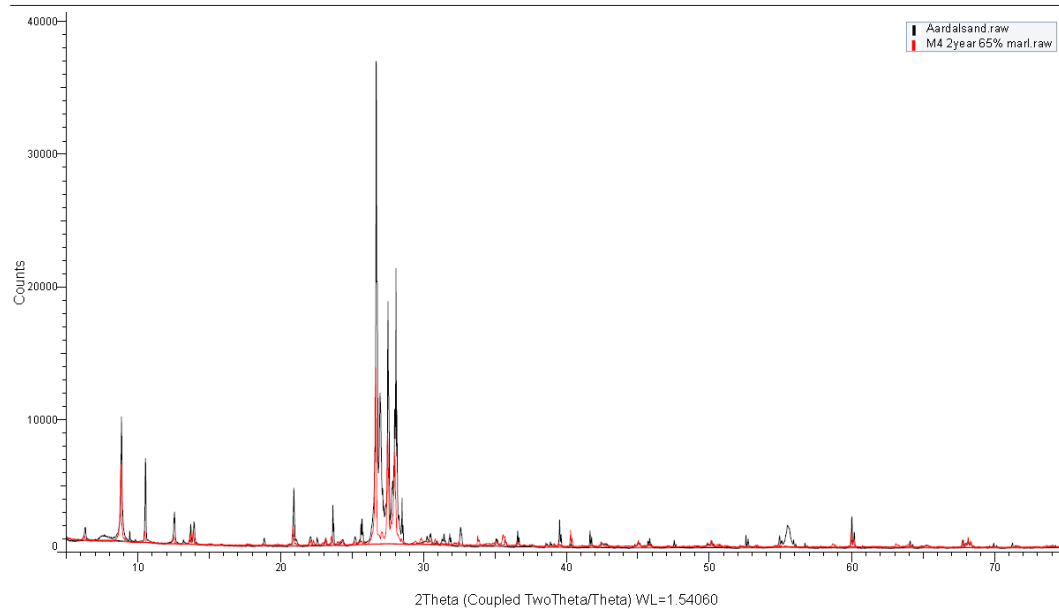


**Figure 17**  
X-ray diffractograms of mortar with 35 % cement replaced with calcined marl (red) and sand from Årdal (black)



**Figure 18**  
X-ray diffractograms of mortar with 50 % cement replaced with calcined marl (red) and sand from Årdal (black)





**Figure 19**  
**X-ray diffractograms of mortar with 65 % cement replaced with calcined marl (red) and sand from Årdal (black)**

## 5 Discussion

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As seen from Figure 1, the strength of all mortars are increasing from 1 to 2 years of curing, as expected (up to 1 year of curing was reported in COIN report no. 49 about durability of mortars with calcined marl and by Østnor and Justnes (2013)). However, the compressive strength of the mortar with 65 vol% replacement of cement by calcined marl increases more than the others, and the compressive strength was about equal for 35, 50 and 65 vol% cement replacement considering the standard deviations, but the flexural strength was somewhat lower. The continued increase in strength for in particular 65 % cement replacement is peculiar, since this mortar did not have detectable calcium hydroxide already after 90 days of curing and can thus not be explained by a classical pozzolanic reaction. Potential reasons for this latter effect were discussed by Østnor and Justnes (2013) based on results until 1 year and repeated in the beginning of this report, but the effect is even more pronounced from 1 to 2 years and hard to explain. This was the reason why more detailed investigations of microstructure was carried out on the mortars with 35, 50 and 65 vol% cement replacement by calcined marl.

The porosity measurements by capillary suction and water saturation under pressure in Table 44 show that the capillary porosity increases steadily with increasing calcined marl replacement of cement, and that the air content is about equal. The exception being the reference without calcined marl that had 1 vol% more air and the lowest capillary porosity. This increase in capillary porosity with increasing cement replacement by calcined marl is expected as more crystalline calcium hydroxide is transferred into amorphous gels in the pozzolanic reaction. This increase in capillary porosity could also be influenced by the cellular nature of unreacted calcined marl grains, as seen by SEM in Figure 10. The results in Table 4 also shows that the dry density of the mortars are decreasing with increasing volume replacement of cement by calcined marl, simply because the calcined marl has lower density (about 2.6 g/cm<sup>3</sup>) than cement (about 3.15 g/cm<sup>3</sup>). Increased porosity should lead to lower strength, not higher, unless the product formed is stronger in nature.

Comparing the porosity measured at 90 days (Østnor and Justnes, 2013) with that at 2 years, one can see that the relative reduction in capillary porosity for the mortar with 65 vol% cement replacement by calcined marl is 3.5 %, while the pore refinement of mortars with 35 and 50 vol% replacement is only 1 %. Note that the absolute porosity values in Table 4 are of mortar volume and should be multiplied by 2.37 to become per binder volume (e.g. cement + water volume). The pore refinement in the period can explain the faster increase in strength for the mortar with 65 vol% cement replacement, but the question remains what causes the pore refinement when there is no calcium hydroxide left for pozzolanic reaction.

Electrical resistivity of water saturated samples is expected to decrease with decreasing total porosity, pore sizes and decreased connectivity of pores, as well as with decreased concentration of ions in the water phase (most notably hydroxyl ions responsible for the highest charge transfer). The electrical resistivity results listed in Table 5 and plotted in Figure 3 show that the electrical resistivity of all samples is increasing with time as the porosity decreases or pores are becoming increasingly segmented. The mortar with 50 vol% cement replacement by calcined marl has the highest electrical resistivity, while mortar with 35 and 65 vol% cement replacement has about equal electrical resistivity considering the standard deviations. The thermo gravimetric curves in Figure 14–16 reveal that the mortar with 35 vol% cement replacement is the only one still having a detectable amount of calcium hydroxide after 2 years of curing, and thereby higher ionic strength in the pore water than the mortar with 65 vol% cement replacement. Hence, it is an indication that the latter sample (65 vol%) has a denser structure, or less connected pores, than the former (35 vol%), but a more open structure than the mortar with 50 vol% cement replaced by calcined marl after 2 years of curing.

The linear expansion of the mortars after 20 months storage in 5 % sodium sulphate at 5 °C and 20 °C given in Table 6 is merely a follow up on the durability testing in the previous report, but can also give valuable information about the microstructure of the mortars. However, none of the mortars has any particular large expansion, and there is little difference between them. The corresponding mass increase plotted in Figure 4 and Figure 5 for storage at 5 °C and 20 °C is not large either, with the exception of the reference at 5 °C with higher mass increase than the others. This could be due to the formation of gypsum by sodium sulphate reacting with calcium hydroxide since it is more calcium hydroxide in the reference than the others, but it is puzzling why this is not the case at 20 °C as well. The steady, but weak mass increase for the others can be explained by the filling of empty contraction pores with water as hydration and pozzolanic reactions proceed.

The overview (100 x magnification) of samples cured at 90 days and 2 years in Figure 6–8 show a general brighter binder after 2 years compared to 90 days (tip to the reader: compare relative to the darker grey aggregates) indicating a densification of the binder responsible for the strength increase. The difference seems to be greater for mortar with 50 and 65 vol% replacement of cement by marl.

Another observation from the images in Figures 6–8 is that there seems to be less unhydrated cement (appears as bright, nearly white spots) as cement is increasingly replaced with calcined marl. This is natural, since there will be less cement per area to begin with when the calcined marl content is increased. The degree of hydration of cement may also increase in general as it is dispersed by increasing content of calcined marl or as the pH is dropping when calcium hydroxide is depleted.

The comparison from 90 days to 2 years of curing in Figures 6–8 shows that there appears not to be any significant difference in the amount of "black spots" in this time period, but note that some "black spots" in this magnification can actually be holes or so called Hadley grains (i.e. former cement grains dissolved completely). The element analysis in Table 7 of the "black spot" to the right in Figure 9 reveals that this is a decalcified inner product with a current Ca/Si = 0.77 enriched in magnesium ( $\approx 6\%$ ) and aluminum ( $\approx 7\%$ ). This composition is typical for a number of other "black spots" .

The attempt to see differences in binder compositions from 90 days to 2 years of curing as reported in Table 7 failed due to high standard deviations. The binder is simply too inhomogeneous. However, it is clear that the overall Ca/Si decreased from  $\approx 1.1$  to  $\approx 0.8$  going from 35 to 50 vol% replacement and somewhat further to  $\approx 0.7$  for 65 vol% replacement.

The biggest difference between the DTG curves in Figure 14–16 of the mortars with 35–65 vol% replacement of cement by calcined marl, respectively, is that there seems to be an increasing signal of water lost already around 60°C with increasing calcined marl replacement. Only the sample with 35 vol% cement replacement by calcined had a small content (0.8 %) of calcium hydroxide left, while no calcium hydroxide could be detected by TG in the other samples with higher cement replacement. Increasing replacement with calcined marl gives less calcium hydroxide due to the dilution effect having less cement per volume unit to start with as well as increased consumption of calcium hydroxide by the pozzolan reaction. The amount of bound water seems to be marginally higher (0.5 % of mortar or 2 % of binder) for the mortar with 50 vol% replacement of cement by calcined marl and about equal for 35 and 65 %.

The XRD diffractograms in Figure 17–19 are dominated by the Årdal sand consisting basically of albite, quartz, microcline and mica. There are only very small reflections from the binder not overlapped by aggregate reflections, and paste samples have to be made sorting

out compounds formed. The mica 00l reflection at  $2\theta$   $8.9^\circ$  seems to be relatively stronger in the mortar compared to the diffractograms of ground sand alone than for the other aggregate compounds. This can be increased preferred orientation of the mica in the mortar sample relative to the ground sand sample, or it can be re-formation of clay like structures in the binder with the same position of the reflection (e.g. smectite has the 00l reflection in the same position). This possibility can only be ruled out by making paste samples or samples using sand without mica and investigate them by XRD. It is also recommended to make paste sample for further studies by  $^{27}\text{Al}$  and  $^{29}\text{Si}$  MAS NMR to find out whether or not extensive polymerization of the CSH gel with inclusion of aluminum in the structure can explain the strength increase for high replacements of cement by calcined marl. It would also reveal any other compounds containing aluminum or silicon.

Of the four hypotheses given in the introduction to explain continued strength increase for mortar with 65 vol% cement replaced by calcined marl, no. 1 can be ruled out, since there is actual less unhydrated cement in that sample. The remaining ones are then;

- 1) There are two types of CSH in the system – one with high C/S from hydration of cement and one with low C/S from the direct pozzolanic reaction between calcined marl and calcium hydroxide. The one with higher C/S has a higher solubility of  $\text{Ca}^{2+}$  and is considered weaker mechanically than the one with lower C/S. Slowly, the two different CSHs will equilibrate, and one ends up with an overall stronger CSH with intermediate C/S.
- 2) The solubility of  $\text{Ca}^{2+}$  from CSH with high C/S and pH is so high that one might have a direct further pozzolanic reaction with unreacted calcined marl.
- 3) The silicate anions of in particular CSH with low C/S will polymerize over time, creating longer chain lengths of the CSH, which may lead to even higher strength.

However, there may be one more. When calcined marl reacts, both silicate and aluminate is released, forming CSH and CAH, as well as possibly intermediate products like  $\text{C}_2\text{ASH}_8$ . Crystalline CAH generally demands a higher atomic Ca/Al than amorphous CSH demand Ca/Si, and the CSH is more flexible in its Ca/Si. The craving for Ca by the aluminate could lead to the de-calcification of inner CSH, as observed, as well as lowering the Ca/Si of the overall CSH, that will adapt to that by increased polymerization of silicates (i.e. point 3 above). Inclusion of aluminate in the CSH structure (i.e. bridging of dimers) will also contribute to a higher degree of polymerization of CSH. In total this may lead to a higher strength binder. Investigation of paste samples may reveal such structural changes better than mortar, as many techniques are disturbed by the aggregate.

## 6 Conclusions

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The strength of all mortars with calcined marl replacing cement from 0 to 65 vol% increases from 1 year to 2 years of curing. The increase is the highest for the mortar with the highest cement replacement in spite of not containing any calcium hydroxide to sustain a pozzolanic reaction since it was 90 days old. After 2 years the compressive strength is about equal for mortars where 35, 50 and 65 vol% cement has been replaced by calcined marl, considering the standard deviations.

The strength gain of the mortar with the 65 vol% cement replacement can partly be explained by the higher relative pore refinement of this mortar (-3.5 % since 90 days) compared to mortars with 35 and 50 % replacement that only had a relative capillary pore refinement of about 1 % from 90 days to 2 years.

The reason for the strength increase or pore refinement in spite of lack of calcium hydroxide to sustain classical pozzolanic reaction is not known at present and the following hypotheses is put forward;

- 4) There are two types of CSH in the system – one with high C/S from hydration of cement and one with low C/S from the direct pozzolanic reaction between calcined marl and calcium hydroxide. The one with higher C/S has a higher solubility of  $\text{Ca}^{2+}$  and is considered weaker mechanically than the one with lower C/S. Slowly, the two different CSHs will equilibrate, and one ends up with an overall stronger CSH with intermediate C/S.
- 5) The solubility of  $\text{Ca}^{2+}$  from CSH with high C/S and pH is so high that one might have a direct further pozzolanic reaction with unreacted calcined marl.
- 6) When calcined marl reacts, both silicate and aluminate is released forming CSH and CAH, as well as possibly intermediate products like  $\text{C}_2\text{ASH}_8$ . Crystalline CAH generally demands a higher atomic Ca/Al than amorphous CSH demand Ca/Si, and the CSH is more flexible in its Ca/Si. The craving for Ca by the aluminate could lead to the de-calcification of inner CSH, as observed, as well as lowering the Ca/Si of the overall CSH that will adapt to that by increased polymerization of silicates. Inclusion of aluminate in the CSH structure (i.e. bridging of dimers) will also contribute to a higher degree of polymerization of CSH. In total this may lead to a higher strength binder.

It is recommended to make paste sample for further studies by  $^{27}\text{Al}$  and  $^{29}\text{Si}$  MAS NMR, as well as XRD, to find out whether or not extensive polymerization of the CSH gel with inclusion of aluminum in the structure can explain the strength increase for high replacements of cement by calcined marl. It would also reveal any other compounds containing aluminum or silicon.

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