Self-Compacting Concrete after 5-year Thaumasite Sulphate Attack
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ABSTRACT
This article outlines a 5-year study on Thaumasite Sulphate Attack, TSA, of Self-Compacting Concrete, SCC with low-alkali cement. For this purpose 42 concrete cylinders with low water-cement ratio, w/c = 0.39, were subjected to distilled or sea water or a solution with sodium sulphate (studies with distilled and sea water ended at 900 days’ age). Fundamental resonance frequency, FRF, length and weight were measured. Comparison was done with the corresponding properties of normal concrete, NC. Severe TSA was observed for all SCC with low-alkali cement, i.e. weight decreased between 4 and 5 years’ age for all SCC with limestone filler due to TSA while NC remains intact.

Key-words: Durability, Limestone filler, Self-Compacting Concrete, Thaumasite Sulphate Attack.

1. BACKGROUND AND OBJECTIVE

1.1 General durability aspects of SCC
The mechanism of TSA is examined based on international research. Other durability issues than TSA are mentioned briefly based on an international overview so that TSA will not be dealt with separately. The primary durability properties are chloride ingress, fire resistance, internal freezing and thawing resistance, salt freezing and thawing resistance and sulphate resistance. All the mentioned properties were recently studied [1]. Salt freezing and thawing scaling and internal freezing and thawing resistance did not alter much from the corresponding properties of NC [2]. In the Great Belt tunnel and also in the Channel tunnel large scale spalling of the concrete occurred during real fire owing the low w/c, in High-Performance Concrete, HPC, that was used in these projects. After the real fire at the Great Belt tunnel only about 10% of the tunnel wall remained due to the extensive and long-term explosive spalling. At the real fire at the Channel tunnel explosive spalling to a depth of 260 mm took place at a 500-m long part of the 500-mm tunnel wall. The HPC was too dense for moisture to evaporate fast enough during the fire. Filler in SCC also increased the risk of fire spalling as compared to NC due to the low water-powder ration of SCC that were corresponding to the low w/c of HPC. Introduction of polypropylene fibres was a way of securing the fire resistance and thus required in SCC in order to obtain resistance to fire spalling [3]. The chloride ingress in SCC with limestone filler was larger than that in NC [4]. The larger chloride ingress in SCC than in NC was related to lower cement content in SCC than in NC (the cement was partly replaced by limestone filler) [5,6].

1.2 Effect of mineral additives
SCC contains large amounts of fine particles such as limestone filler, fly ash, silica fume, and glass or quartzite filler in order to avoid gravity segregation of larger particles in the fresh mix. Normally the sum of cement and all filler varies between 450 and 650 kg/m³ for SCC, i.e. about
200 kg/m³ more fines than for NC. The increase of fines in SCC as compared to the amount of fines in NC prevents the larger aggregate from segregation in the fresh mix due to differences in gravity to the water and the cement in the fresh mix. Large amount of limestone filler in the concrete affects the durability substantially especially chloride ingress and fire resistance even though economical and ecological reasons talks for replacing Portland cement, as much as possible by filler [7]. As much as 7% of the industrial fabrication of carbon dioxide, CO₂, is related to the production of cement [7]. One way to reduce the CO₂-relaease is to use HPC where cement is more efficiently applied, or to replace the cement by filler. However, large amount of limestone filler increases the carbonation [8] and reduce other durability properties of SCC compared with NC [9]. A protective layer of NC for the steel reinforcement bars maybe needed outside the SCC under severe conditions as at the seaside of Japan [10]. When sulphates are present at a concrete construction with large amounts of limestone filler there is a substantial risk of TSA [9]. TSA for concrete with limestone filler differs clearly from normal sulphate attack since Ca₆[Si(OH)₆.12H₂O]₂(SO₄)₂((CO₃)₂ (Thaumasite) is formed instead of Ettringite Ca₆[Al(OH)₆.12H₂O]₂(SO₄)₂(SO₄)(H₂O)₂ [11]. TSA is only formed under low temperature conditions and is most abnormal since silicon atoms are surrounded by an octahedron of OH-ions [11]. The firm solution is therefore quite complex and may contain sulphate-rich and carbonate-rich forms Thaumasite [11]. The reason for this risk is the high specific surface of the limestone filler that causes a solution of CO₂ in the pore water especially at low temperature. Comparisons between mortars with OPC, OPC CEM I with 5% limestone filler and CEM II with 15% limestone filler shows TSA formation during sulphate attack even 5% limestone filler [12]. The TSA is somewhat prohibited when the sulphate solution also contained sodium chloride (synthetic seawater) but still there [12]. Mortar with OPC CEM I with 5% limestone filler was much more damaged by TSA than mortar with OPC. Only after 3-year TSA on concrete with limestone filler may observed in 1.4-3 g/l sodium sulphate solutions [13]. This international research referred to clearly shows that even low content of limestone filler in the concrete affects the TSA resistance even though low-alkali cement is used. TSA is also much more frequent at in-situ concrete that at precast probably due to quality differences [13]. The role of pH seems to be negligible as compared to the TSA [14]. In England large scale TSA was observed in 1998 at 30-year old high-way foundations structures made of concrete with limestone filler due to Pyritic clay [15]. TSA may thus take place in buried concrete foundations containing limestone aggregate and subjected to an external source of sulphate ions from the surrounding groundwater with about 1 g/l sodium sulphate solutions [16]. Only after 14 days there was little change in water soluble sulphur acid in contact with the Pyritic clay (filled around the foundation afterwards). After TSA on a concrete the CSH-binding in the cement paste converts into a more or less porous mass causing completely deterioration of the concrete [15]. In 1998 the issue of TSA even caused a question in the British House of Commons [11]. TSA can not be prevented by sulphate resistant low-alkali cement since the limestone filler in the concrete and sulphate acids interact. Low-alkali cement has a low C₃A content to avoid external sulphate attack. Otherwise formation of Ettringite or gypsum would destruct the concrete due to the increase of volume of the compound. For NC without limestone filler sulphate resistance is not a issue when low-alkali cement is used. For SCC with limestone filler TSA may be a problem even though low-alkali cement is used since the limestone filler provides the source internally of the concrete. Gypsum is due to sulphate attack on concrete without limestone filler with cement with normal alkali content.

1.3 Objective

The objective of this research was to compare over a 5-year period of time the performance of SCC with low-alkali cement and limestone filler under influence of a solution of sodium sulphate with the corresponding properties of NC with low-alkali cement but without limestone.
filler. The tests were performed in accelerating conditions since the main reason was to compare NC with SCC under sulphate attack. Measurements of length, weight and FRF were performed. A comparison with the influence of curing in seawater and distilled water on the corresponding properties was performed over 3 years. The effect of mix proportions of the SCC, i.e. filler content, filler type, mixing order and pouring pressure (at top or at bottom of 5.5-m high mould), was also studied but not in the full-scale matrix. The accelerated test was to be performed under reasonably severe conditions as compared to the worst conditions expected in real environment for concrete. Only a few years existed to see the TSA effect. The test conditions were also to follow international standards and recommendations for test of sulphate attack. The ongoing research eventually will continue until any damage is observed on NC without limestone filler.

1.4 List of symbols

B increased amount of filler
FRF fundamental resonance frequency (Hz)
HPC High-Performance Concrete (vibrated)
K crystalline limestone filler (age about 300 million years, 40 µm)
N new mixing order
NC (R) normal (compacted) concrete (vibrated)
O ordinary mixing order
S limestone filler (age about 50 million years, 15 µm)
T 5.5 m hydrostatic pouring pressure instead of 0.3 m
8 8 % air content

2. MATERIALS AND METHODS

2.1 Materials

Sulphate resistant, low-alkali Portland cement, Table 1, and limestone filler was used in the experiments together with crushed and natural aggregate, superplasticiser (polycarboxylic ether) and an air-entrainment agent (fatty oils). The motivation for using low-alkali cement was for the concrete to resist normal sulphate attack. This type of low-alkali cement is in common use when sulphates may exist or accumulate close to the concrete. Mineralogical granulometric properties of the material and the grading of the fresh concrete mix proportions are given in Figures 1-2. The following parameters were studied partly (not in the full-scale matrix) [1]:

1. Compaction type, self-compacting SCC (O) and vibrated, NC (R)
2. Mixing order, new (N) and ordinary (O)
3. Amount of limestone filler, increased (B) and ordinary (O)
4. Type of limestone filler, crystalline (K) and sedimentary (S)
5. Pouring pressure, 0.3 m (O), and 5.5 m (T)
6. Air content by volume, 5% (N) and 8% (8)
7. Curing type, distilled or sea water, sodium sulphate

A total of 7 different concrete were studied, six SCC and one NC, all with w/c = 0.39, which is a w/c normal for concrete in severe conditions. NC of type RO and SCC of type KN were performed in full-scale in large quantities at all the Southern Link site, Stockholm [1]. NC of type KN thus is a typical bridge deck, foundation or retaining wall concrete [1]. From the concrete, larger specimens, 0.23 m in diameter and 0.30 m in length, were prepared, two of each combination. The concrete was sealed in a steel container directly after casting and covered by steel. From the large specimen 3 cylinders 50 mm in diameter and 150 mm in length were core drilled.
The grounds for the new reversed mixing order was a better dispersion and frost resistance in a separate investigation of four types of limestone filler for SCC [17]. The motivation to use an increased amount of filler was to simulate mixing mistakes and its effect on TSA of SCC. Finally, the increase of pressure was performed in order to simulate the effect on TSA of pouring SCC into a 5.5 m high wall. SCC was not vibrated after casting. NC was vibrated 10 s each time after filling one-third of the steel container. The sample were taken both at 28 and 90 days 'age. In parallel 100-mm cubes were cast in order to obtain the strength of the concrete, three at each age and combination of time and concrete mix proportions. The cubes were wrapped in adhesive aluminium foil in order to be sealed cured. Two types of mixing order were used [1,17]:

\[
\text{Material passing} \\
\begin{array}{|c|c|c|c|c|}
\hline
0.00 & 0.004 & 0.016 & 0.032 & 0.063 & 0.125 & 0.250 & 0.500 & 1.000 & 2.000 & 4.000 & 8.000 & 16.000 \\
\hline
0.00 & 0.00 & 0.00 & 0.00 & 0.25 & 0.50 & 1.00 & 2.00 & 4.00 & 8.00 & 16.00 & 32.00 & 64.00 \\
\hline
\end{array}
\]

**Figure 1** – Material grading curves.

**Figure 2** – Particle grading in fresh mix.

<table>
<thead>
<tr>
<th>Component</th>
<th>(%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>CaO</td>
<td>65</td>
</tr>
<tr>
<td>SiO₂</td>
<td>21.6</td>
</tr>
<tr>
<td>Al₂O₃</td>
<td>3.5</td>
</tr>
<tr>
<td>Fe₂O₃</td>
<td>4.4</td>
</tr>
<tr>
<td>K₂O</td>
<td>0.58</td>
</tr>
<tr>
<td>Na₂O</td>
<td>0.05</td>
</tr>
<tr>
<td>MgO</td>
<td>0.78</td>
</tr>
<tr>
<td>SO₃</td>
<td>2.07</td>
</tr>
<tr>
<td>Ignition losses</td>
<td>0.47</td>
</tr>
<tr>
<td>CO₂</td>
<td>0.14</td>
</tr>
<tr>
<td>Clinker minerals:</td>
<td>21</td>
</tr>
<tr>
<td>C₂S</td>
<td>57</td>
</tr>
<tr>
<td>C₃S</td>
<td>1.7</td>
</tr>
<tr>
<td>C₄AF</td>
<td>13</td>
</tr>
<tr>
<td>Water demand</td>
<td>25%</td>
</tr>
<tr>
<td>Initial setting time</td>
<td>145 min.</td>
</tr>
<tr>
<td>Density</td>
<td>3214 kg/m³</td>
</tr>
<tr>
<td>Specific surface</td>
<td>305 m²/kg</td>
</tr>
</tbody>
</table>

Table 1 – Chemical compositions and properties of the cement CEM I 42.5 BV/SR/LA.
1. Ordinary mixing order with all dry material mixed with the water with air-entrainment for \(\frac{1}{2}\) min. and then mixed with the superplasticiser for another 2\(\frac{1}{2}\) min.

2. New reversed mixing order with all material (also water, air-entrainment superplasticiser) except for limestone filler mixed for \(\frac{1}{2}\) min. with additional mixing with filler for 2\(\frac{1}{2}\) min.

During the slump flow tests SCC performed stable without any separation which stability also was observed in the full-scale production at the Southern Link, Stockholm.

2.2 Methods

The experiments consists of accelerated tests based on CEN recommendations in order to compare over a long period of time the performance to TSA of NC without limestone filler with the performance of SCC with limestone filler, both the concrete based on low-alkali cement. All curing and testing took place at 5 °C, which is close to the average temperature of the ground in Nordic countries. One-third of the specimens were placed in a solution of 18 g/l of sodium sulphate in distilled water being a three time higher concentrations than what may be expected in sulphuric grounds [18-20], one-third in 1% sodium chloride seawater from Barsebäck, Sweden, and the rest (one-third) of the specimens placed in distilled water. The solutions were renewed every third month. A propeller was placed in the basin in order to secure a good circulation of the solution around the specimens. The method for investigation of the damage has been measurement of length, mass by weighing and of FRF. At the start of testing, either at 28 or 90 days’ age, and at 100, 300, 900, 1400 and 1700 days of exposure, length, weights of the specimens and FRF were established (1400 and 1700 days TSA only) [1,18]. The test specimen was dried off with a wet cloth before weighing removing all peeled off material with a brush. The ongoing comparative test did not as yet include the measurement of water suction but this test may be included in the test as soon the time of testing is ended. For the detection of macro voids, pressure technique under water then also may be applied. FRF is closely correlated to the elastic modulus of the specimen. A decrease of FRF is a sign of internal deterioration of the concrete for example caused by TSA. The precise measurement location for the length and FRF measurement was marked into the rim of the ends of the cylinders. The cubes were tested for strength at 1, 7, 28 and 90 days’ age. Increase of density between the fresh state and the hardened is a uncomplicated way of quantifying the degree of compaction of concrete. In the fresh state the density was obtained before casting of the concrete - after hardened also at 28 and 90 days’ age. The diameter, length and weight of all specimens were measured. The fresh density was measured on a 8-l sample of concrete. The same batch of concrete was then cast in larger specimens, 0.23 m in diameter and 0.30 m in length. Directly after core drilling of the small sample, cylinders 50 mm in diameter and 150 mm long, the density was taken of these, from the same source as the fresh density was established, both at 28 days’ age and at 90 days’ age. At 1700 days of exposure surface of all specimens except for NC were totally destroyed (measurement of FRF/length was not feasible at this time). The quality of the microstructure is very important like documentation by thin section but has not yet been performed since it is an ongoing research.
### Table 2 – Mix proportions and properties of tested concrete (kg/m³).

<table>
<thead>
<tr>
<th>Material/mix composition</th>
<th>KN</th>
<th>KN8</th>
<th>KO</th>
<th>KOB</th>
<th>KOT</th>
<th>RO</th>
<th>SO</th>
</tr>
</thead>
<tbody>
<tr>
<td>Crushed aggregate 8-16 mm</td>
<td>363</td>
<td>355</td>
<td>367</td>
<td>371</td>
<td>363</td>
<td>862</td>
<td>402</td>
</tr>
<tr>
<td>Natural sand 0-8 mm</td>
<td>853</td>
<td>836</td>
<td>864</td>
<td>872</td>
<td>855</td>
<td>715</td>
<td>786</td>
</tr>
<tr>
<td>Natural sand 0-2 mm</td>
<td>316</td>
<td>309</td>
<td>320</td>
<td>135</td>
<td>316</td>
<td>146</td>
<td>422</td>
</tr>
<tr>
<td>Limestone filler</td>
<td>183</td>
<td>180</td>
<td>186</td>
<td>375</td>
<td>184</td>
<td>0</td>
<td>94</td>
</tr>
<tr>
<td>Cement</td>
<td>418</td>
<td>409</td>
<td>423</td>
<td>427</td>
<td>419</td>
<td>431</td>
<td>416</td>
</tr>
<tr>
<td>Air-entrainment (g/m³)</td>
<td>585</td>
<td>1203</td>
<td>106</td>
<td>213</td>
<td>117</td>
<td>474</td>
<td>125</td>
</tr>
<tr>
<td>Superplasticiser</td>
<td>2.97</td>
<td>3.2</td>
<td>3.39</td>
<td>4.13</td>
<td>3.69</td>
<td>7.32</td>
<td>2.99</td>
</tr>
<tr>
<td>Water</td>
<td>163</td>
<td>160</td>
<td>165</td>
<td>167</td>
<td>163</td>
<td>168</td>
<td>162</td>
</tr>
<tr>
<td>w/c</td>
<td>0.39</td>
<td>0.39</td>
<td>0.39</td>
<td>0.39</td>
<td>0.39</td>
<td>0.39</td>
<td>0.39</td>
</tr>
<tr>
<td>Air content (%)</td>
<td>5.6</td>
<td>8</td>
<td>5.5</td>
<td>4.9</td>
<td>6.3</td>
<td>5.8</td>
<td>5.6</td>
</tr>
<tr>
<td>Slump (mm)</td>
<td>720</td>
<td>735</td>
<td>620</td>
<td>780</td>
<td>640</td>
<td>710</td>
<td></td>
</tr>
<tr>
<td>Flow until 500 mm (s)</td>
<td>5</td>
<td>8</td>
<td>10</td>
<td>7</td>
<td>8</td>
<td>-</td>
<td>5</td>
</tr>
<tr>
<td>Aggregate content (% vol.)</td>
<td>0.64</td>
<td>0.63</td>
<td>0.65</td>
<td>0.65</td>
<td>0.65</td>
<td>0.65</td>
<td>0.64</td>
</tr>
<tr>
<td>Compressive strength, 1-day (MPa)</td>
<td>20.8</td>
<td>26.0</td>
<td>14.0</td>
<td>23.8</td>
<td>23.8</td>
<td>16.0</td>
<td>24.5</td>
</tr>
<tr>
<td>7 days (MPa)</td>
<td>50.0</td>
<td>63.0</td>
<td>38.0</td>
<td>56.0</td>
<td>56.0</td>
<td>45.0</td>
<td>54.0</td>
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<tr>
<td>28 days (MPa)</td>
<td>63.3</td>
<td>83.5</td>
<td>49.8</td>
<td>74.5</td>
<td>74.5</td>
<td>60.5</td>
<td>67.5</td>
</tr>
<tr>
<td>90 days (MPa)</td>
<td>76.0</td>
<td>92.0</td>
<td>58.0</td>
<td>81.0</td>
<td>81.0</td>
<td>70.0</td>
<td>79.0</td>
</tr>
<tr>
<td>Density at mixing</td>
<td>2297</td>
<td>2250</td>
<td>2323</td>
<td>2348</td>
<td>2300</td>
<td>2325</td>
<td>2285</td>
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<tr>
<td>Density at 28 days’ age</td>
<td>2345</td>
<td>2299</td>
<td>2351</td>
<td>2436</td>
<td>2436</td>
<td>2440</td>
<td>2361</td>
</tr>
<tr>
<td>Density at 90 days’ age</td>
<td>2385</td>
<td>2309</td>
<td>2380</td>
<td>2416</td>
<td>2428</td>
<td>2415</td>
<td>2362</td>
</tr>
<tr>
<td>28-day increase of density (%)</td>
<td>2.1</td>
<td>2.2</td>
<td>1.2</td>
<td>3.7</td>
<td>5.9</td>
<td>4.9</td>
<td>3.3</td>
</tr>
<tr>
<td>90-day increase of density (%)</td>
<td>3.8</td>
<td>2.6</td>
<td>2.5</td>
<td>2.9</td>
<td>5.6</td>
<td>3.9</td>
<td>3.4</td>
</tr>
<tr>
<td>1400-day scaling 28 d (kg/m²)</td>
<td>0.54</td>
<td>0.90</td>
<td>3.8</td>
<td>0.14</td>
<td>0.35</td>
<td>0</td>
<td>2.25</td>
</tr>
<tr>
<td>1400-day scaling 90 d (kg/m²)</td>
<td>0.96</td>
<td>0.92</td>
<td>3.1</td>
<td>1.09</td>
<td>0.28</td>
<td>0</td>
<td>1.82</td>
</tr>
<tr>
<td>1700-day scaling 28 d (kg/m²)</td>
<td>3.54</td>
<td>5.02</td>
<td>12.5</td>
<td>0.83</td>
<td>1.79</td>
<td>0</td>
<td>6.02</td>
</tr>
<tr>
<td>1700-day scaling 90 d (kg/m²)</td>
<td>5.56</td>
<td>4.92</td>
<td>6.99</td>
<td>4.66</td>
<td>3.54</td>
<td>0</td>
<td>5.49</td>
</tr>
</tbody>
</table>

### 3. RESULTS

#### 3.1 Nine hundred days’ age

The list of symbols makes it easier to read the Figures. There is no acceptance criterion given in this comparative test series since the objective was to show if the risk of TSA enhances over a long time of period or not when using limestone filler in SCC as compared to the risk of TSA in NC without limestone filler. The strength development of the concrete is shown in Table 2 and Figure 3. The strength development was clearly influenced by the filler mineral additives in the concrete (substantially increased strength) and by the air content (lower strength). A continuous increase of the length, weight and FRF over 900 days was observed for all concrete types dependent on the way of mixing (ordinary mixing order). The increase of the length, weight and FRF was independent of type of compaction, SCC or NC, independent of the content of limestone filler, independent of the content of air and independent of the pouring pressure, Figure 4-6 [1].
Only for concrete KO28 a decrease of weight was observed when curing in sodium sulphate. This decrease was owing to a crack-through the specimen at the rim of the end of it. The general increase of length, weight and FRF over 900 days was the result of continuous hydration and water absorption, slowly compensating for the chemical shrinkage due to the hydration of the cement. The water absorption did not differ much between SCC and NC at this point. The weight increase may also be explained by deposits inside the concrete. The tests were prolonged until significant change was observed of length, weight and FRF between the types of concrete studied. The grounds for the new reversed mixing order was a better dispersion and frost resistance in a separate investigation of four types of limestone filler for SCC [17]. A significant de-
crease of weight was observed for SCC with new mixing order exposed to a solution of 18 g/l of sodium sulphate in distilled water. No corresponding decrease as related to FRF of these SCC was observed. The decrease of weight was due to a surface scaling attack of the specimen, clearly visible. The specimen performed as it did after salt freezing and thawing scaling tests [2]. Obviously limestone filler should be added simultaneously together with cement during the mixing in order to avoid this kind of deterioration of the surface of SCC. Otherwise, when filler was added afterwards, the water was primary attached to the cement and a proper mixing of cement and limestone filler was prohibited. The mechanism of the new mixing order is referred to the improved dispersion of limestone filler [17]. However, the new mixing order may not be feasible in common practise.

3.2 One-thousands-four-hundred-day TSA

After 1400 days only TSA was studied, since only large length change and mass changes were observed after accelerated treatment with sodium sulphate, cp. Figures 4-6 with Figures 7-9. For almost all concrete an increase of FRF, and then also of the elastic modulus, was observed which indicated that no internal deterioration took place of the concrete due to TSA except for SCC, SO 28 (decrease of FRF), and SCC, KO 28 (divided specimen). For vibrated NC continuous 5% increase of FRF was observed – more or less as for SCC. After about 8% increase of FRF the NC, KO 28, was divided - SCC specimen SO28 exhibited about 7% increase of FRF before a sudden decrease of FRF to about -3% FRF was measured. Continuous water absorption probably led to an increase of FRF. Accelerated treatment with sodium sulphate caused much larger expansion than with fresh or sea water probably due formation of Thaumasite, cp. Figure 5 with Figure 8. Since low-alkali cement was used little or no gypsum was formed. The largest expansion after 900 days of TSA was observed for NC, RO 90, about 7‰, and for SCC, KO 28, about 6‰. Between 900 and 1400 days’ age continuous length increase was observed, up to 8‰ for NC, RO 90, which is much. For SCC, SO 28, 10‰ increase was observed and decrease of FRF in parallel which indicated that the specimen was undergoing internal destruction. For some of the SCC specimens the length may not be measured at 1400 days’ age (KO 28 and KO 90) since the measurement point disappeared due to surface destruction of the specimens. The most significant development after 1400 days of TSA was the weight decrease of SCC after 900 days’ age (normal mixing order) and after 450 days’ age (new mixing order), cp. Figure 6 with Figure 9. For NC continuous mass increase took place. Calcium hydroxide, Ca(OH)$_2$, probably leached from the SCC at a distance of 1-3 mm from the surface under the influence of external sulphate attack and internal carbon acid attack originating from the fine dispersed limestone filler. The surface of SCC, still with a flat and smooth surface and without any sign of destruction, exhibited white residuals, probably from escaped Ca(OH)$_2$, break down when touched because an empty space was left just behind the surface. The surface deposits are not yet investigated with respect to composition since the tests are ongoing. However, all loose deposits were removed with brush before weighing. After brushing up to 12% mass loss was observed for SCC, KO 28. For SCC except for KOB 28, KOT 28 and KOT 90, the surface scaling, still after taking into account the water absorption, exceeded 0.5 kg/m$^2$ (up to 3.8 kg/m$^2$ for specimen KO 28), which was much, Table 2. With 0.5 kg/m$^2$ surface spalling the aggregate of the surface is clearly visible indicating severe TSA on the gel. However, even though large mass losses were observed for most of the SCC, almost no specimen exhibited internal percolation of Ca(OH) leading to loss of FRF and elastic modulus decrease. After 1400 days of TSA all surface of SCC was affected. However, the sulphate concentration and the time of exposure are not linear parameters from the experimental point of view [19].
Figure 7 – FRF change after 1400 days of exposure in 18 g/l of sodium sulphate in distilled water at 5 °C.

Figure 8 – Length change after 1400 days of exposure in 18 g/l of sodium sulphate in distilled water at 5 °C.

Figure 9 – Mass change after 1400 days of exposure in 18 g/l of sodium sulphate in distilled water at 5 °C.
3.3 One-thousands-seven-hundred-day TSA

After 4 years until 5 years a sudden huge scaling of the concrete surface took place caused by TSA, more or less for all concrete with low-alkali cement and with limestone filler, Table 2, Figure 10. For NC with low-alkali cement but without limestone filler no scaling at all took place due to TSA since no carbon acid was available in the concrete in this case, Figure 10. The mechanism of TSA is a interaction by carbon and sulphate acids which attack the gel forming Thaumasite. If no limestone filler was presented as in NC with low-alkali cement in this investigation, no weight decrease was observed. No normal sulphate attack with Ettringite formation was observed in the gel on NC since low-alkali sulphate resistant cement was used. NC with low-alkali cement but without limestone filler thus withstood a solution of high sulphate content at 5 °C for at least five years. The tests are ongoing to eventually find any sign of normal sulphate attack also on NC with low-alkali sulphate resistant cement but without limestone filler.

![Graph](image1.png)

*Figure 3 – Concrete strength development.*

**Figure 10 – Scaling at 1400 and 1700 days.**

4. DISCUSSION

4.1 Effect of amount of limestone filler in mix proportions

The limestone particles were much more soluble in the pore water forming carbon acid which caused deterioration of the cement gel together with the sulphate attack, forming Thaumasite, than when much larger limestone aggregate particles were mixed with cement and covered. At 90 and 450 days of contact the effect of a solution of sodium sulphate on SCC was small but accelerated from 450 until 900 days of experiment, Figures 4-9. No accelerating effect of a solution of sodium sulphate was observed in SCC with increased amount of limestone filler, Figure 9. Larger amount of limestone filler, i.e. lower water-powder, w/p = 0.21, when w/c was held constant, did not lead to any increase of interior deterioration, which was expected. Results of SCC with w/p = 0.21 (about the same amount of limestone filler as cement in the mix proportions) showed about the same FRF as for NC probably due to lower permeability of SCC with limestone filler than that of NC. Still SCC with w/p = 0.21 showed larger surface scaling than NC with w/p = 0.39 at w/c = 0.39 for both the concrete types, indication the observed deterioration of the surface (shell formation maybe owing to percolation of Ca(OH)$_2$, 1-3 mm from the face of SCC).
4.2 Effect of compaction technique

Three different compaction techniques were used in the experiments:

1. Pressure compaction, 5.5 m pouring height (SCC, KOT, Table 2, Figure 11)
2. Self-compaction, 0.3-m pressure from a pouring height (other SCC than KOT, Table 2)
3. Vibration (NC)

The largest increase of density, i.e. increase of density between fresh state and hardened one, was observed for SCC, KOT, with pressure compaction, about 6% as followed by NC, RO, about 5% increase of density from fresh state till the hardened one, Table 2 and Figure 11. The lowest increase of density, i.e. the lowest degree of compaction was observed for SCC, KO, which is the same type of concrete as SCC, KOT, but with only 0.3-m pressure compaction, less than 2% of increase of density between fresh and hardened state, Table 2 and Figure 11. SCC with large amount of filler, KOB, or with small size limestone filler, SO, exhibited degrees of increase of density in between concrete as mentioned above, about 3%, Figure 11. SCC with new mixing order also showed about 3% degree of increase of density. A tendency relationship between the mass losses, $\delta m/m$ (%) and the increase of density between the fresh and the hardened state, $\delta \rho / \rho$ (%), was obtained for all SCC (%), Figure 12 ($R^2 = 0.68$):

$$\delta m/m_{SCC} = 17.3 \ln(\delta \rho / \rho) - 35.55 \quad \{1 < \delta \rho / \rho < 6%; \ R^2 = 0.68\} \quad (1)$$

$\delta m/m$ denotes the mass losses between start of testing and 5 years’ age (%)

$\ln(\delta \rho / \rho)$ denotes the natural logarithm of density increase between fresh and hardened state.

The accuracy of Figure 12 is acceptable ($R^2 = 0.68$) taking into account that the starting age of the test differs, 28 or 90 days, and also the pouring pressures and mixing order differs. Figure 12 indicates a tendency that the TSA attack of SCC is dependent on the compaction of the SCC, i.e. differences between density at start of testing and at fresh state influenced the sulphate resistance.
5. SUMMARY AND CONCLUSIONS

Accelerated testing of Thaumasite sulphate attack, TSA, has been performed on concrete with low-alkali cement, both normal compacted, NC, without limestone filler and self-compacting, SCC, with limestone filler. The solutions of the accelerated test was according to the one proposed by CEN, 18 g/l, at 5 °C. The experimental results and international studies clearly shows a potential risk of using limestone filler in self-compacting concrete even with low-alkali cement since Thaumasite formation may destroy the concrete. NC with low-alkali cement but without limestone filler thus withstood a solution of high sodium sulphate content at 5 °C for at least five years. The following detailed conclusions may be drawn at 900 days of exposure:

1. For NC continuous increase of length, weight and fundamental resonance frequency, FRF.
2. For SCC cast under 5.5 m pressure also a continuous increase of length, weight and FRF.
3. For SCC with normal mixing cast without pressure up to 4 kg/m² TSA surface scaling.
4. For SCC with new mixing order cast without pressure up to 1 kg/m² TSA surface scaling.
5. For SCC (except with sedimentary limestone filler) a continuous increase of FRF.
6. The amount or type of limestone filler did not affect the amount of TSA surface scaling.

At 1700 days of exposure the following detailed conclusions may be drawn:

1. A rapid increase of surface scaling of SCC with limestone filler, between 2 and 12 kg/m².
2. Still no surface scaling of NC without limestone filler.
3. A tendency relationship between the TSA mass losses (%) and the increase of density (corresponds to degree of compaction) between the fresh and the hardened concrete state.
4. Less TSA of SCC with 5.5 m gravity pressure compaction than of SCC without pressure.

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