CoDry II - Concrete drying, critical moisture levels

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RISE Rapport 2023:78
Förord

Rapporten presenterar resultat från ett forskningsprojekt som samfinansierades av RISE och Konsortiet för grundforskning inom cement och betong vid RISE som består av: Heidelberg Materials Cement Sverige AB, Heidelberg Materials Betong Sverige AB; Heidelberg Precast Abetong AB, Strängbetong AB, Swerock AB, Thomas Concrete Group AB.
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REPORT

Summary

Two concrete classes with water to cement ratios of 0.38 & 0.50 were studied for moisture related indicators. These were relative humidity levels (internal, i.e. RBK method, and external) and salt deliquescence on concrete with unidirectional drying.

A secondary cementitious material (SCM), ground granulated blast furnace slag, was used to replace 25 % of the reference cement (CEM I 52.5 R) at both w/c levels. Its effect on the above-mentioned measurement techniques were also studied.

The concretes were exposed to different initial curing conditions, firstly covered in plastic sheeting for one week followed by exposure to 50 % RH (20°C) or completely sealed post casting and placed in the same climate chamber. Some of the w/c concrete cubes =(0.38) were sealed.

One of the aims was to document the self-desiccant effects of the low w/c concrete.

At w/c = 0.50, there was no long-term benefit of adding GGBFS in terms of RH measurements up to 6 months, both in the sealed and exposed specimens. The difference in RH in exposed cubes was ca 6% lower than the sealed one.

On the other hand, the rate of deliquescence (moisture absorption) from CaCl₂ (MVER) indicated a progressive rate drop in water vapour emissions down to 9 lbs/ft² 24hrs with the addition of GGFBS (compared to ca 12 lbs/ft² 24 hours in the reference).

At w/c = 0.38, the initial reduction in measured RH is significant with the inclusion of GGFBS (ca 4-6 % lower). After 14 days the highest measurable RH (w/c = 0.38 + 25% slag) was 82 % and the lowest 78 %. Sealing the cubes (after 7 days) stopped the RH evolution in the concrete with slag but not the reference concrete.

MVER measurements were very similar in both cases and progressively lowered to 5.7 to 6.9 after 6 months.

In the completely sealed specimens (metal buckets with lids), the internal RH dropped at a slower rate than in the specimens that were enclosed with plastic sheeting. The internal RH in the concrete with w/b 0.38 and slag remained constant at ca 82 % from day 14 and onwards. In the case of the reference this drop stagewise and were within 1 % of each other after 6 months (83.7 %).

The w/b series = 0.50 had no significant development over 3 months and was at approx. 90 %. After 6 months a drop in the measured RH was noted in the slag concrete to 86.1 %.

This project even included provision for two RISE /CBI employees to attend the RBK theoretical course which both passed. The practical exam was deemed unnecessary to complete.
1 INTRODUCTION

This project is the second part following on from CoDry I, where mainly a literature study of various concrete “dryness” measurement techniques was presented. This study focuses mainly on two techniques, relative humidity (RH) using capacitive sensors and rates of deliquescence based on anhydrous calcium chloride salts.

Concrete is a widely used material for the substructure in flooring, it itself can be the flooring. Normal practice is to allow the concrete to develop its internal microstructure and lose some of its freely bound water molecules. At some point the concrete surface is deemed suitable to apply a variety of flooring systems. This could involve a self-leveling compound, some of these systems involve the application of a water-based adhesive with which e.g., a polyvinylchloride based (PVC) flooring. Research on the various problems associated with concrete and organic based flooring is numerous Alexanderson [1], Chino, Kato [2], Yokota, Kato [3], Persson [4], Sjöberg [5], Sjöberg [6], Sjöberg [7], Sjöberg [8], Sjöberg [9], Sjöberg and Ramnäs [10], Larsson, Hägerhed-Engman [11].

Initial thoughts on this project were to include a work package within which emissions were to be studied but was excluded.

Another focus in this study was the microstructure develop in cement paste over time and how this can be used in conjunction with the results from the RH and salt measurements (deliquescence) to explain the outcomes. Early in the project it was decided to not include fly ash as a possible cement clinker replacement due to its uncertain future and supply issues. GGBFS was though used as a cement replacement material.
2 MATERIALS

Recipes
Two concrete recipes with different water to cement ratios, 0.50 & 0.38, were developed. Comparable concretes with a reduced amounts of cement clinker were also mixed. 25% of the cement was replaced with GGBFS.

To prevent the influence of aggregates on the experiments these were limited in size to 8 mm. This alters the cement paste volume and is higher than a usual concrete.

Below in TABLE 2.1 amounts of in going materials in presented.

**TABLE 2.1. Recipe for concrete mixes w/c = 0.38 & 0.50.**

<table>
<thead>
<tr>
<th></th>
<th>density $\rho$ [kg/m$^3$]</th>
<th>REF w/c = 0.50 [kg/m$^3$]</th>
<th>REF (0.50) + 25% GGBS [kg/m$^3$]</th>
<th>REF w/c = 0.38 [kg/m$^3$]</th>
<th>REF (0.38) + 25% GGBS [kg/m$^3$]</th>
</tr>
</thead>
<tbody>
<tr>
<td>CEM I 52,5 R</td>
<td>3200</td>
<td>558,6</td>
<td>419,8</td>
<td>655,0</td>
<td>492,5</td>
</tr>
<tr>
<td>GGBFS Bremen</td>
<td>2890</td>
<td>0</td>
<td>139,9</td>
<td>0,0</td>
<td>164,2</td>
</tr>
<tr>
<td>Crushed 0/4mm</td>
<td>2690</td>
<td>1090,6</td>
<td>1092,7</td>
<td>1086,0</td>
<td>1089,1</td>
</tr>
<tr>
<td>Crushed 4/8 mm</td>
<td>2690</td>
<td>327,2</td>
<td>315,2</td>
<td>326,0</td>
<td>311,9</td>
</tr>
<tr>
<td>H$_2$O</td>
<td>995</td>
<td>279,3</td>
<td>279,8</td>
<td>247</td>
<td>246</td>
</tr>
<tr>
<td>SP Superplasticizer</td>
<td>1070</td>
<td>0</td>
<td>0,0</td>
<td>2,6</td>
<td>2,6</td>
</tr>
<tr>
<td>Air (volume) ca 2%</td>
<td>20</td>
<td>0</td>
<td>0,0</td>
<td>0,0</td>
<td>0</td>
</tr>
</tbody>
</table>

Superplastizer: BASF MasterGlenium 51 (35% dry content)

SLAG: Slag known as "SLAGG BREMEN" was supplied by Thomas Cement via Holcim AG that fulfils SS-EN 15167-1 & -2. According to the documentation the k value is 0.60 with the cement used in the experiments. In Table 2.2 an XRF analysis of the material is presented.

**TABLE 2.2. Analysis of slag material carried out by RISE**

<table>
<thead>
<tr>
<th>Normalized [wt%]</th>
<th>SLAGG BREMEN</th>
</tr>
</thead>
<tbody>
<tr>
<td>Na$_2$O</td>
<td>0.0</td>
</tr>
<tr>
<td>K$_2$O</td>
<td>0.7</td>
</tr>
<tr>
<td>MgO</td>
<td>9.0</td>
</tr>
<tr>
<td>CaO</td>
<td>41.6</td>
</tr>
<tr>
<td>Al$_2$O$_3$</td>
<td>11.6</td>
</tr>
<tr>
<td>SiO$_2$</td>
<td>33.0</td>
</tr>
<tr>
<td>SO$_3$</td>
<td>2.5</td>
</tr>
<tr>
<td>Fe$_2$O$_3$</td>
<td>0.5</td>
</tr>
<tr>
<td>MnO</td>
<td>0.3</td>
</tr>
<tr>
<td>TiO$_2$</td>
<td>1.0</td>
</tr>
</tbody>
</table>


In Table 2.3, the chemical analysis of the cement is presented.
Table 2.3 Skövde SH cement chemical analysis.

<table>
<thead>
<tr>
<th>Normalized wt%</th>
<th>CEMENT SH CEM I 52,5 R (2019-05-28)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Na₂O</td>
<td>0.4</td>
</tr>
<tr>
<td>K₂O</td>
<td>1.4</td>
</tr>
<tr>
<td>MgO</td>
<td>1.0</td>
</tr>
<tr>
<td>CaO</td>
<td>65.3</td>
</tr>
<tr>
<td>Al₂O₃</td>
<td>4.7</td>
</tr>
<tr>
<td>SiO₂</td>
<td>18.2</td>
</tr>
<tr>
<td>SO₃</td>
<td>4.3</td>
</tr>
<tr>
<td>Fe₂O₃</td>
<td>3.8</td>
</tr>
<tr>
<td>MnO</td>
<td>0.6</td>
</tr>
<tr>
<td>TiO₂</td>
<td>0.3</td>
</tr>
</tbody>
</table>

SALTS: CaCl₂ MERCK BATCH NR K409 33578 013, 98 % purity.

CaCl₂ purchased from VaporScore ( ASTM 1869-11 testing kit )

See APPENDIX 2 for PSD of the CaCl₂ salts used.

EQUIPMENT: EIRICH Mixer R 09T.

Coarse weighing scales with accuracy ± 1 g

Fine weigh scales with accuracy ± 0.01 g

VAISALA HUMICAP RH sensors, accuracy ± 1.5% RH upto 90% RH thereafter ± 3.0% & ± 0.10 °C

DRILLING according to RBK method with 16 mm drill tip.

Plastic moulds 150 mm: thickness equivalent to 15 mm, see FIGURE 2.1. Prior to casting, all moulds were individually weighed and recorded. This was to ease the measurement of the weight loose during the course of the experiments.
FIGURE 2.1. 150 mm plastic moulds. Metal moulds were also used for temperature development measurements.
3 METHODS

3.1 Plastic mould casting

In November 2019 the first batches were produced. The slump flow and air content were measured in the fresh concrete prior to casting into 100 mm cube and 150 mm cubes.

The weight of each cast cube was recorded before being stacked on the pallet. The entire stack was covered in building grade plastic (even under the bottom moulds) to create an enclosed environment. The specimens were then removed from the plastic, reweighed and placed in the climate room 20 °C ± 2 & RH= 50 % ±5.

In the case of the sealed buckets, no covering was used, and these were directly placed in the climate room. These were reweighed once the temperature measurements were concluded.

Fresh concrete properties such as air content and slump were documented at each cast.

Three 100 mm sided cubes were used for compression testing at 28 days for the plastic mould casts.

FIGURE 3.1.1 Four rows of specimens (2x2) wrapped in building grade plastic, temperature measured on each row and in the concrete cast in metal mould.
FIGURE 3.1.2. Specimens cast, weighed and placed in the climate room. Temperature sensors installed on each row. Specimens then covered with building grade plastic (seen to the left) for seven days and reweighed.

3.2 Sealed Bucket method
In this method, the concrete was sealed immediately post casting, thereby enclosing all moisture within a defined volume (the 3l bucket) The concrete was filled to the top and all buckets were cast to the same weight whilst on the weighing scales, see FIGURE 3.2.1.

FIGURE 3.2.2. Concrete specimens cast in a completely sealed metallic bucket. All buckets were filled with to the same weight as to reduce the air volume from concrete surface to lid. The bucket closet to the observer was used to measure the internal temperature.
development in the initial two days post casting. The air volume created in that bucket, ca. 30 mm was enough to cause an accumulation of condensation water on the top surface of the concrete.

FIGURE 3.2.3. 25mm hole produced to attach a sealed RH sensor to measure the surface RH. In the middle one sees the RBK method attachments.

FIGURE 3.2.4. Vaisala handheld unit with sealed bucket specimen in background. Currently showing the results from the surface RH attachment.
CASTING

30/10/2019  REF 0.50
04/11/2019  REF 0.38
17/02/2020  0.50 + 25% slag
24/02/2020  0.38 + 25% slag

3.3 STORAGE

All specimens were placed in a climate room, see FIGURE 3.3.1 for details. This was either directly carried out as in the sealed bucket method or after one week as in the first batches.

FIGURE 3.3.1. Example of storage conditions, climate room 20°C ± 2°C & RH 50% ± 5%, moulds remain around concrete at all time. Size of concrete 150 mm x 150 mm x 150 mm. Weight of mould written on the outside. The blue arrows show the specimens that were sealed 7 post casting.
3.4 Relative humidity (RH) measurements in concrete

RH sensor

Sensor how it works

The sensors used in the experiments are from the manufacturer Vaisala. The type of sensor used are known as Humicap®. The principle behind the functioning and results from the sensor are explained in [12] & [13]. The capacitance of the sensor changes as the amount of water vapour deposited changes, this then corresponds to a particular relative humidity in the air.

Split Cube

The splitting cube method employs a splitting action on a concrete cube that has been stored in the same manner as the cubes using the RBK methods. The aim is to gather cementitious material at the same depth in the concrete as carried out in the RBK method. The material is gathered into a glass test tube that is sealed and a Humicap® sensor is placed in the same glass tube. The measurement is carried out 24 hours after the collection of the material. The test tube is placed in a climate chamber which is kept at 20°C ± 2.

![Figure 3.4.1. Preparation stages for splitting cube method](image)

<table>
<thead>
<tr>
<th>Specimen prior (150 mm sided)</th>
<th>Splitting prep 1</th>
<th>Splitting prep 2</th>
</tr>
</thead>
<tbody>
<tr>
<td>Splitting prep 3</td>
<td>Place in plastic bag and hammer to obtain specimen in the middle</td>
<td>Remove middle piece and further break down to ca 10 mm pieces. These are placed in sealed test tube with RH sensor.</td>
</tr>
</tbody>
</table>

RBK method

The RBK method is a methodology to predict the concrete surface RH once it has been covered. A moisture sensitive sensor is placed at a specific depth related to its thickness and
drying method. The moisture readings are translated to equilibrium RH over a number of days. Flooring suppliers specify the max RH\textsubscript{critical} for their products which is usually 85% or 90%. This is the main (only) official method used in Sweden to determine the condition of the concrete flooring.

<table>
<thead>
<tr>
<th>Concrete cube that has been drilled to a depth 0.40 x 150mm. The drill bit is Ø16 mm. All dust is removed with a vacuum cleaner.</th>
<th>The sides are cleared with a metal wire brush. The picture shows that the aggregates do not cover the entire area (d\textsubscript{max} = 8mm)</th>
<th>Here one sees an installed RH sensor, with a seal between the plastic tubing and concrete surface.</th>
</tr>
</thead>
</table>

FIGURE 3.4.2. Preparation of RBK RH sensor into concrete.

Bucket Method (Sealed):

A new batch was mixed with the exact same materials as in previous batches. All concrete was then cast into 3 l metal buckets to a specification of 7800 g per bucket so that the concrete touched the lid of the bucket when sealed. The buckets were weighed post casting and then at the point of transfer into the climate chamber and the whenever a measurement was to be conducted. One bucket was used per measurement. Sufficient specimens were cast to last for several years if so desired. This are currently in the 50 % climate room at DKV 26. The RBK method and a surface RH measurement were employed to determine the self-desiccant effect of the 4 different concrete types over time. All recipes were recast, but on different occasions.

Moisture Absorption method

This method involves the absorption of water vapour that is in the air volume that is enclosed over the concrete surface. CaCl\textsubscript{2} is in a petri dish which is raised by a 30 mm spacer above the 150 x 150 mm concrete surface.
Deliquescence

The ability of a material or salt to absorb water vapour from its surroundings and not react (which can also be regenerated with heat). This can be further studied in Cantrell, McCrory [14]. Different salts have different equilibrium water vapour pressures, CaCl₂ has an equilibrium partial water vapour pressure equivalent to ca 33 % at 20 ° C.

Surface RH measurements

A British method employed to determine a concrete’s surface suitability for floor finishes is determined with a surface RH measurement. Below in FIGURE 3.4.5, an initial model can be seen in the bottom right corner of the concrete surface. In an improved model, a Vaisala Humicap® sensor has two additional seals applied in the middle of the impervious white cylinder. Mastic is applied to the bottom (interface concrete and plastic cylinder) and between the opening of sensor and data cable.
3.5 Microstructure analysis

MIP

Cement paste with the same raw material batches were mixed and sealed until time of testing. Two intervals 14 and 61 days were chosen to reflect on the RBK results in concrete. An external lab was contracted to facilitate this test. The MIP (mercury intrusion porosity tests expose a small sample to increasing amounts of pressurized mercury gas. The volumes and pressures required to reach equilibrium stages equate to pore volumes and diameters.

Exchanging cement (CEM I 52.5 R) with slag will change the microstructure and the extent of which is of great interest.

TGA + vacuum oven dried 40°C

The bound and less chemically bound water can be determined with a combination of an analytical instrument and low temperature drying. As relative humidity is a measure of the available water vapour at a specific temperature, the availability of “free water” could be an indicating factor as to the RH results in concrete. Separate samples at w/b = 0.50 or 0.38
(with and without slag) were exposed to two different paths, one was vacuum oven dried to 30°C and the other was exposed to a 10°C/min nitrogen saturated environment from 30-1000°C. The weight losses in both analyses are combined to obtain the bound and unbound water (including portlandite among others).

**DVS**

Dynamic vapour sorption is a method whereby an exact environment is established (temperature and RH). A sample is placed in this chamber and a very accurate weighing scale records the weight drop during desorption and weight increase in the absorption phase. Once the weight loss or gain is stable the next step is initiated. In total the testing takes ca 72 hrs.

The starting RH in these experiments was 20°C & 95 % concluding at 11% whereby the humidity is increased in stages back up to 95%. The age of the samples were 6 months and 1.5 years respectively. The matrix of the samples included w/b = 0.38 % 0.50 (with and without slag). The data obtained during the testing provided coordinates of weight loss and equilibrium RH established during the ab- and desorption phases.
4 RESULTS

4.1 Fresh Properties of cast concrete:

**Slump flow of concretes:**

**TABLE 4.1 Slump flow results of concretes**

<table>
<thead>
<tr>
<th></th>
<th>Slump [mm] 2019</th>
<th>Air content [%] 2019</th>
<th>Slump [mm] 2020</th>
<th>Air content [%] 2020</th>
<th>Slump [mm] Sealed Bucket</th>
<th>Air content [%] Sealed Bucket</th>
</tr>
</thead>
<tbody>
<tr>
<td>REF 0.50</td>
<td>110</td>
<td>1.8</td>
<td>120</td>
<td>1.8</td>
<td>120</td>
<td>2.1</td>
</tr>
<tr>
<td>REF 0.38</td>
<td>110</td>
<td>2.5</td>
<td>140</td>
<td>2.5</td>
<td>120</td>
<td>3.8</td>
</tr>
<tr>
<td>0.50 + 25 % slag</td>
<td>150</td>
<td>1.6</td>
<td></td>
<td></td>
<td>120</td>
<td>2.9</td>
</tr>
<tr>
<td>0.38 + 25 % slag</td>
<td>110</td>
<td>3.1</td>
<td></td>
<td></td>
<td>65</td>
<td>3.9</td>
</tr>
</tbody>
</table>

4.2 Temperature development at casting.

![Temperature profile](image)

*FIGURE 4.2.1. Temperature profile in concrete w/c = 0.38, 150 mm sided cubes, plastic and metal mould.*
FIGURE 4.2.2. Temperature profile in concrete with $c = 0.50$, 150 mm sided cubes, plastic and metal mould.

FIGURE 4.2.3. Temperature Development 0.38 + Slag in plastic cube mould.
FIGURE 4.2.4. Temperature Development 0.50 + Slag in plastic cube mould.

FIGURE 4.2.5. Temperature development in metal bucket, REF 0.50. Temperature in the core, lid and sides (lowest), see FIGURE 3.3.2.
4.3 Compression Strength

The compressive strengths of the initial batches of concrete with w/b = 0.38 & 0.50 are presented.

**TABLE 4.3.1. 28-day strengths and density of 3 x 100 mm sided cubes; w/c = 0.50 without slag. Standard deviation included in the bottom row.**

<table>
<thead>
<tr>
<th>Density</th>
<th>Unit</th>
<th>Compressive Strength</th>
<th>Unit</th>
</tr>
</thead>
<tbody>
<tr>
<td>2240 kg/m³</td>
<td>53.9</td>
<td>MPa</td>
<td></td>
</tr>
<tr>
<td>21 kg/m³</td>
<td>0.8</td>
<td>MPa</td>
<td></td>
</tr>
</tbody>
</table>

**TABLE 4.3.2. 28 day strengths and density of 3 x 100 mm sided cubes; w/c = 0.38 without slag. Standard deviation included in the bottom row.**

<table>
<thead>
<tr>
<th>Density</th>
<th>Unit</th>
<th>Compressive Strength</th>
<th>Unit</th>
</tr>
</thead>
<tbody>
<tr>
<td>2330 kg/m³</td>
<td>74.5</td>
<td>MPa</td>
<td></td>
</tr>
<tr>
<td>14 kg/m³</td>
<td>0.4</td>
<td>MPa</td>
<td></td>
</tr>
</tbody>
</table>

**TABLE 4.3.3. 28 day strengths and density of 3 x 100 mm sided cubes; w/p = 0.50 with slag as cement replacement (25%). Standard deviation included in the bottom row.**

<table>
<thead>
<tr>
<th>Density</th>
<th>Unit</th>
<th>Compressive Strength</th>
<th>Unit</th>
</tr>
</thead>
<tbody>
<tr>
<td>2240 kg/m³</td>
<td>50.5</td>
<td>MPa</td>
<td></td>
</tr>
<tr>
<td>21 kg/m³</td>
<td>0.6</td>
<td>MPa</td>
<td></td>
</tr>
</tbody>
</table>

**TABLE 4.3.4. 28 day strengths and density of 3 x 100 mm sided cubes; w/p = 0.38 with slag as cement replacement (25%). Standard deviation included in the bottom row.**

<table>
<thead>
<tr>
<th>Density</th>
<th>Unit</th>
<th>Compressive Strength</th>
<th>Unit</th>
</tr>
</thead>
<tbody>
<tr>
<td>2309 kg/m³</td>
<td>72.6</td>
<td>MPa</td>
<td></td>
</tr>
<tr>
<td>8 kg/m³</td>
<td>1.3</td>
<td>MPa</td>
<td></td>
</tr>
</tbody>
</table>

Graphical comparison of compressive strengths at 28 days.
FIGURE 4.3.1. Comparison of compressive strengths after 28 days, cube sides = 100 mm.
4.4 RH Evolution

4.4.1 Weight Loss initial and over time
All cubes were weighed directly after casting, after 7 days and then at the relevant intervals. Some concretes were sealed after the initial 7 days but are not part of the measurements.

The concretes cast into metal buckets were also weighed in this manner, with the primary purpose to verify a sealed enclosure was created, i.e. no moisture loss.

An average value of the cubes that were not on the top stack (as condensation had formed at the top of the plastic due to the temperatures reached during the hydration phase).

Below in TABLE 4.4.1.1 a summary of the weight loss is presented [non sealed concretes].

* 0.38 = 926 gH₂O/cube 150mm; 0.50 = 942 gH₂O/cube 150mm

<table>
<thead>
<tr>
<th>Concrete</th>
<th>Weight loss after 7 days under plastic covering [g]</th>
<th>Weight loss after 3 months (mostly in climate chamber) [g]</th>
<th>Percentage of weight loss during first 7 days compared to total [%]</th>
</tr>
</thead>
<tbody>
<tr>
<td>REF 0.38</td>
<td>22.5</td>
<td>44.7</td>
<td>50</td>
</tr>
<tr>
<td>Slag 0.38</td>
<td>22.4</td>
<td>35.7</td>
<td>63</td>
</tr>
<tr>
<td>REF 0.50</td>
<td>25.5</td>
<td>65.9</td>
<td>39</td>
</tr>
<tr>
<td>Slag 0.50</td>
<td>35.3</td>
<td>61.1</td>
<td>58</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>Concrete</th>
<th>Weight loss after 7 days as percentage of total concrete [%]</th>
<th>Percentage loss of available water based on recipe [%]</th>
<th>Weight loss after 3 months as percentage of total [%]</th>
<th>Percentage loss of available water based on recipe [%]</th>
</tr>
</thead>
<tbody>
<tr>
<td>REF 0.38</td>
<td>0.28</td>
<td>2.42</td>
<td>0.56</td>
<td>4.82</td>
</tr>
<tr>
<td>Slag 0.38</td>
<td>0.28</td>
<td>2.41</td>
<td>0.45</td>
<td>3.85</td>
</tr>
<tr>
<td>REF 0.50</td>
<td>0.32</td>
<td>2.70</td>
<td>0.84</td>
<td>6.99</td>
</tr>
<tr>
<td>Slag 0.50</td>
<td>0.45</td>
<td>3.74</td>
<td>0.78</td>
<td>6.48</td>
</tr>
</tbody>
</table>
### 4.4.2 RH Measurements

All measurements that were conducted in regards internal and surface related moisture measurements are presented in TABLE 4.4.2.1 below. The concrete in “Bucket\(_{\text{Sealed}}\)“ is a separate batch.

**TABLE 4.4.2.1 All RH and water vapour absorption measurements from 150 mm cubes and sealed buckets. Internal = RBK; MVER = CaCl₂ water vapour absorption.**

<table>
<thead>
<tr>
<th>Recipe</th>
<th>Specimen</th>
<th>Method</th>
<th>14d [%]</th>
<th>45d [%]</th>
<th>60d [%]</th>
<th>90d [%]</th>
<th>180d [%]</th>
<th>365 d [%]</th>
</tr>
</thead>
<tbody>
<tr>
<td>REF (0.50)</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Bucket(_{\text{Sealed}})</td>
<td>RBK</td>
<td>89,1</td>
<td>90,1</td>
<td>91,1</td>
<td>90,7</td>
<td>92,8</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Cube(_{\text{Open}})</td>
<td>RBK</td>
<td>89,3</td>
<td>&quot;-&quot;</td>
<td>84,2</td>
<td>83,7</td>
<td>n/a</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Cube(_{\text{Split}})</td>
<td>Test Tube</td>
<td>87,2</td>
<td>&quot;-&quot;</td>
<td>84,5</td>
<td>83,5</td>
<td>n/a</td>
<td></td>
<td></td>
</tr>
<tr>
<td>MVER (_{\text{Open}})</td>
<td>CaCl(_2) [lbs/ft(^2) 24hrs]</td>
<td>12,9</td>
<td>&quot;-&quot;</td>
<td>9,9</td>
<td>12,6</td>
<td>n/a</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Surf. RH (_{\text{Open}})</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>REF (0.38)</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Bucket(_{\text{Sealed}})</td>
<td>RBK</td>
<td>86,0</td>
<td>84,6</td>
<td>84,7</td>
<td>84,7</td>
<td>83,7</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Cube(_{\text{Open}})</td>
<td>RBK</td>
<td>85,1</td>
<td>&quot;-&quot;</td>
<td>79,4</td>
<td>76,4</td>
<td>n/a</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Cube(_{\text{Sealed}})</td>
<td>RBK</td>
<td>85,0</td>
<td>&quot;-&quot;</td>
<td>82,6</td>
<td>81,0</td>
<td>n/a</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Cube(_{\text{Split}})</td>
<td>Test Tube</td>
<td>83,1</td>
<td>&quot;-&quot;</td>
<td>79,4</td>
<td>77,8</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>MVER (_{\text{Open}})</td>
<td>CaCl(_2) [lbs/ft(^2) 24hrs]</td>
<td>9,7</td>
<td>&quot;-&quot;</td>
<td>6,9</td>
<td>5,7</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Surf. RH (_{\text{Open}})</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Slag (0.50)</td>
<td>Bucket\textsubscript{(Sealed)}</td>
<td>RBK</td>
<td>Tech. prob</td>
<td>90,0</td>
<td>89,4</td>
<td>90,6</td>
<td>86,1</td>
<td></td>
</tr>
<tr>
<td>-----------------------</td>
<td>---------------------------------</td>
<td>-----</td>
<td>------------</td>
<td>------</td>
<td>------</td>
<td>------</td>
<td>------</td>
<td></td>
</tr>
<tr>
<td></td>
<td>Cube\textsubscript{(Open)}</td>
<td>RBK</td>
<td>87,9</td>
<td>&quot;-&quot;</td>
<td>84,2</td>
<td>84,2</td>
<td>n/a</td>
<td></td>
</tr>
<tr>
<td></td>
<td>Cube\textsubscript{(Split)}</td>
<td>Test Tube</td>
<td>87,6</td>
<td>&quot;-&quot;</td>
<td>84,9</td>
<td>89,5</td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td>MVER \textsubscript{(Open)}</td>
<td>CaCl\textsubscript{2} \textnormal{[lbs/ft\textsuperscript{2} 24hrs]}</td>
<td>12,7</td>
<td>&quot;-&quot;</td>
<td>10,1</td>
<td>8,9</td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td>Surf. RH \textsubscript{(Open)}</td>
<td>61,2</td>
<td>&quot;-&quot;</td>
<td>59,6</td>
<td>57,4</td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>Slag (0.38)</th>
<th>Bucket\textsubscript{(Sealed)}</th>
<th>RBK</th>
<th>82,0</th>
<th>82,2</th>
<th>81,2</th>
<th>82,0</th>
<th>82,7</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Cube\textsubscript{(Open)}</td>
<td>RBK</td>
<td>79,2</td>
<td>&quot;-&quot;</td>
<td>74,5</td>
<td>75,3</td>
<td>n/a</td>
</tr>
<tr>
<td></td>
<td>Cube\textsubscript{(Sealed)}</td>
<td>RBK</td>
<td>78,4</td>
<td>&quot;-&quot;</td>
<td>76,9</td>
<td>78,4</td>
<td>n/a</td>
</tr>
<tr>
<td></td>
<td>Cube\textsubscript{(Split)}</td>
<td>Test Tube</td>
<td>76,8</td>
<td>&quot;-&quot;</td>
<td>77,3</td>
<td>77,1</td>
<td>n/a</td>
</tr>
<tr>
<td></td>
<td>MVER \textsubscript{(Open)}</td>
<td>CaCl\textsubscript{2} \textnormal{[lbs/ft\textsuperscript{2} 24hrs]}</td>
<td>9,8</td>
<td>&quot;-&quot;</td>
<td>7,3</td>
<td>6,9</td>
<td>n/a</td>
</tr>
<tr>
<td></td>
<td>Surf. RH \textsubscript{(Open)}</td>
<td>55,4</td>
<td>&quot;-&quot;</td>
<td>56,3</td>
<td>56,3</td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

*All RH data are the raw data from the VAISALA handheld device that have not been corrected for the entire RBK method. The drilling and sensor placement and time durations have been followed.

The data shows that the w/c is an important aspect in obtaining various RH measurements. The lower the w/c, the lower the obtained result. This applies for the RBK method.

A general trend on the outcome from the RBK measurements is that:

1) Allowing mass transfer of internal moisture in the pore structure to the surrounding environment will decrease the measured RH.
2) Sealing the concrete surface post curing will prevent mass transfer of moisture into the air and the RH measurements are higher.

3) Completely sealing the concrete post casting results in the least drop in RH. Differences in the w/c will determine the drop and development of RH over time.

In regards the MVER (water vapour absorption) and the surface RH measurements

The results show that the more mature the cement paste (and thus the development of the microstructure and the loss of freely bound water) the lower the amount of water vapour that can be transferred into the salt structure via diffusion. This though was not the case with REF 0.50, where after 90 days, the same rate of water vapour mass transfer is approximately the same.

The surface RH measurements are the least defined measurement and the closer to the surrounding RH (50% ±5) the least amount of data interpretation that can be applied.

4.5 Microstructure analysis

The microstructure of the cement pastes was analysed by the RISE lab in Borås. The same batches of cement and slag were used in the concrete samples in the RISE lab in Stockholm. The mixing and cement pastes were completely sealed until the point of testing.

4.5.1 MIP Analysis

Cement pastes with w/b = 0.38 & 0.50 were mixed and immediately sealed until these were analyzed externally (BAM; Berlin). The data is analyzed to reflect different size intervals, see top legend in FIGURE 4.5.1.1 and its associated pore volume, see x-axis in FIGURE 4.5.1.1. The results are presented in its entirety, see FIGURES 4.5.1.1 & 4.5.1.2 and then individually, see FIGURES 4.5.1.3-4.5.1.6. A summary table on cumulative volumes at 14 and 61 days is presented in TABLE 4.5.1.1

![Pore entry diameter intervals](image)

**FIGURE 4.5.1.1. Pore entry diameter internal sizes for 0.38 & 0.50 cement pastes after 14d & 61d.**

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FIGURE 4.5.1.2. Pore entry diameter internal sizes changed to represent 100% of individual results for \( w/c = 0.38 \) & 0.50 cement pastes after 14d & 61d.

Here, the results show that the pore diameters in different size intervals at different cement paste maturity levels. Comparing \( w/c = 0.50 \) series, the amount of pores below 0.02 \( \mu \)m increase as maturity level increases from 14 – 61 days, but the amount in the slag cement pastes is higher. Further analyses can be seen in FIGURES 4.5.3.

A similar pattern is observed in the \( w/c = 0.38 \) series.

FIGURE 4.5.1.3. MIP analysis of cement paste; 0.38 series REF & Slag (25% cement replacement) 14 and 61 days.
FIGURE 4.5.1.4. MIP analysis of cement pastes; 0.38 series (REF & Slag (25% cement replacement) 14 and 61 days. Converted data to represent 100% of specimens’ total.

FIGURE 4.5.1.5. MIP analysis of cement paste; w/c; 0.50 series (REF & Slag (25% cement replacement) 14 and 61 days.
FIGURE 4.5.1.6. MIP analysis of cement paste 0.50 series (REF & Slag (25% cement replacement) 14 and 61 days. Converted data to represent 100% of specimens’ total.

The amount of larger pore diameters in REF 0.50-14d is larger than in the equivalent Slag 0.50-14d sample. At 61 days the sample with slag has an increased percentage of smaller pores below 0.02 µm.

TABLE 4.5.1.1 Cumulative pore volume of cement pastes (MIP) after 14 and 61 days.

<table>
<thead>
<tr>
<th>Cement paste</th>
<th>Cumulative volume [mm$^3$/g] at 14 days</th>
<th>Cumulative volume [mm$^3$/g] at 61 days</th>
</tr>
</thead>
<tbody>
<tr>
<td>REF 0.38</td>
<td>127</td>
<td>119</td>
</tr>
<tr>
<td>REF 0.38 + slag</td>
<td>141</td>
<td>121</td>
</tr>
<tr>
<td>REF 0.50</td>
<td>189</td>
<td>174</td>
</tr>
<tr>
<td>REF 0.50 + slag</td>
<td>195</td>
<td>173</td>
</tr>
</tbody>
</table>

The inclusion of slag and the reduction of cement clinker minerals increases the cumulative pore volume in both w/b matrices. At 61 days, these differences have effectively reduced to no statistical difference within the same w/b matrix. Within the distribution of volume, the samples with slag had a higher percentage of pores with smaller diameters (below 0.02µm).
4.5.2 TGA + vacuum oven drying analysis

TGA + vacuum oven drying measurements were carried out to determine if there were any differences in the bound water in the cement pastes, see below in FIGURE 4.5.1.1. A comparison between the results obtained from the RBK tests on concrete, the weight loss from the vacuum oven and the TGA are presented. Two different time intervals were analyzed, 14 days and 61 days post mixing.

FIGURE 4.5.2.1. Method: TGA and vacuum oven drying (40°C). Results of bound water content after 14 days, cement paste with different water/binder content. Including measured RH % (RBK) on open 150 mm sided cubes.

The diagram, see FIGURE 4.5.2.1, shows that the amount of bound water and the oven dried cement paste do not correspond to the RBK measurements in the case of “25% Slag 0.38”. The relative humidity measurements in the concrete with the low w/c and slag do not correspond to the water moisture measurements in the cement paste which are very similar to the cement pastes without slag. A pattern can be seen in the samples, whereby a reduction in bound water resulted in a lower internal RH being measured in the concrete.
FIGURE 4.5.2.2. Method: TGA and vacuum oven drying (40°C). Results of bound water content after 61 days, cement paste with different water/binder content. Including measured RH % (RBK) on open 150 mm sided cubes.
4.5.3 DVS Analysis

Cement pastes from the same batch as used in MIP and TGA (w/b = 0.38 & 0.50) were sent to an external lab (BAM; Berlin) to analyze the cement paste structures after 6 months and 1.5 years.

The specimens are exposed to different RH % environments and the mass change at each RH is recorded. The program starts at relative humidity (RH) 95 % and 20°C down to 11% (desorption). Each step change comes to equilibrium before proceeding to the next RH step. The specimen is then exposed to ever more moist conditions (absorption). The weight loss or gain is recorded over the desorption and absorption. Below in FIGURES 4.5.3.1 & 4.5.3.2 the results can be observed.

**FIGURE 4.5.3.1.** Results from DVS of cement pastes (0.38 & 0.50) after 6 months. The cement pastes with higher w/b have a higher weight loss (see red and yellow lines).
FIGURE 4.5.3.2. Results from DVS of cement pastes (0.38 & 0.50) after 1.5 years. The cement pastes with higher w/b have a higher weight loss (see red and yellow lines). The hysteresis loop has moved upwards, i.e. less weight loss is required to obtain a larger range of potential microstructure RH.

FIGURE 4.5.3.3. Close up of the area of interest within the concrete drying area (85%). A dotted vertical line has been added to cross the weight loss of the different specimens. Cement paste = 6 months. The water loss for example to obtain 85% in REF 0.50 is ca 100% more than the REF + slag 0.50 series.
FIGURE 4.5.3.4. Close up of the area of interest within the concrete drying area (85%) A dotted vertical line has been added to cross the weight loss of the different specimens. Cement paste = 18 months.

TABLE 4.5.3.1 Cement paste weight loss to reach 85% equilibrium in DVS at 6 months and 18 months.

<table>
<thead>
<tr>
<th>Cement paste</th>
<th>[%] Weight loss for 85 % RH equivalent at 6 months</th>
<th>[%] Weight loss for 85 % RH equivalent at 1.5 yrs</th>
</tr>
</thead>
<tbody>
<tr>
<td>REF 0.38</td>
<td>-0.21</td>
<td>-0.29</td>
</tr>
<tr>
<td>REF 0.50</td>
<td>-0.61</td>
<td>-0.55</td>
</tr>
<tr>
<td>REF 0.38 + slag</td>
<td>-0.12</td>
<td>-0.07</td>
</tr>
<tr>
<td>REF 0.50 + slag</td>
<td>-0.27</td>
<td>-0.16</td>
</tr>
</tbody>
</table>

The DVS measurements show that the hysteresis between desorption and absorption is very apparent. In the case for both cement pastes and time intervals, the inclusion of slag in the cement paste volume decreases the weight loss needed to change the equilibrium RH. Increasing the w/c, in this case to 0.50 increases the amount of water loss required to alter the equilibrium RH. Here though the effect of slag is more apparent.

The cement paste maturation from 6 months to 1.5 years changes the slope of the hysteresis loop, compare FIGURES 4.5.3.1 & 4.5.3.2.

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5 DISCUSSION

The inclusion of GGBFS (slag) as a cement replacement in concrete, as used in this study, had a beneficial effect on the final measurement of RH using the RBK method, see TABLE 4.4.2.1. This was only the case with a low w/b (water to binder ratio). In this study this was 0.38. The replacement of slag with cement in a higher w/b concrete did not have a similar effect. The RH sensor though, that provides the measurement to determine the concrete’s internal RH, is dependent on the water vapour amount entering the instrument and changing the capacity of the sensor.

It is known [15] that prevalent salts in the cement paste, primarily hydroxides of K⁺ (potassium) and Na⁺ (sodium) have a low RH equilibrium, ranging from 9-15%. The amount of cement (Skövde SH) used in these experiments (> 420 kg/m³) would increase the total amount of alkalis per m³ and hence reduce the overall RH in the small volume of air that is in contact with the cement paste.

The range of alkalis (or sodium oxide equivalent) from the lowest to highest cement content in the tested concretes was ca 5.5 kgNaOeqv – 6.5 kgNaOeqv. The high presence of these alkalis did not reduce the measured RH in the 0.50 series drastically though, see TABLE 4.4.2.1. The alkali content did not affect the measured RHs in these cases.

The use of CaCl₂ as a method to determine a concrete’s surface suitability for surface finishes (e.g. PVC flooring) as described in ASTM 1869-11, compared to capacitor sensors (as in the RBK method) is debatable. The salt’s deliquesce at ca RH 33% creates a thermodynamic difference between the salt and the concrete’s surface. The results, based on the standard and requirements of flooring suppliers (glue manufacturers and floorings) would not have fulfilled their requirements of MVER < 3 lbs/ft² 24 hr or < 5 lbs/ft² 24 hr, where MVER stands for medium vapor evaporation rate. The results from the RBK method would have given a positive indication to lay a suitable flooring material in the case of the concretes in the 0.38 series, see TABLE 4.4.2.1. More details on the chosen MVER ranges set by the glue and flooring suppliers are required to determine its suitability. It is though the most simple method employed in this study.

The RBK sensor and associated method does not cause a sustained thermodynamic difference between the cement paste and the air that it is in contact with. The method would though cause an increased thermal load during the drilling operation (one of the steps within the method) to install the sealed plastic tubing, see Appendix 1. The amount of water vapour lost (if any) at the interface is extremely difficult to determine, the effect of drying and RH measurements (DVS) is discussed.

The introduction of heat into the system was negated with the “splitting cube” method. Concrete at a depth equivalent to the RBK drilling depth was removed by splitting a cube in a systematic set of operations. The RH results obtained were in very close correlation to the RBK method. The precision of depth tough is not as accurate as the RBK method (0-1 milimeter) of determined depth. The time the split cube is exposed to a dry environment needs to be reduced as this will also have an effect on the measured RH.
Close correlation between the RHs measured at RBK depth and on the surface of the totally enclosed specimens (sealed buckets) show that the developed method of sensor sealing works.

The interpretation though of the surface dry results was difficult (in the case when the specimen’s surface was exposed to a constant climate). As the concretes were maturing (and also drying out), the measured surface RH approached the RH of the surrounding environment and so providing little detail of the internal RH or the potential change of RH once any surface layer was applied. This would give this British method limited usage for sensitive flooring.

DVS measurements show that the sensitivity of cement pastes with low w/c has a bearing on the equilibrium RH, especially with added slag, see FIGURE 4.5.3.4. This was noticeable in both concrete types. This could have implications in “normal” RBK measurement techniques whereby drilling temperatures could be high and comparisons between CEM I and CEM I + SCM concrete could be more difficult to compare. The absorption path within the different series though is more similar than on the desorption path.
6 CONCLUSIONS

The drying conditions of the concrete’s surface post casting (initial 7 days) had a significant impact on the RH measurements in the concrete. The longer the drying was conducted, the lower the RH measurement in the concrete, regardless of w/c ratio.

Use of slag was effective in reducing the measured RH in the initial ca 90 days of the concrete’s hydration phase but only in the concrete with lower w/c. At w/c = 0.50 no difference could be measured.

The MVER measurements (evaporation of water vapour from concrete surface due to thermodynamic change in RH caused by deliquescence of CaCl$_2$) showed that the inclusion of slag did not reduce the MVER to a point where glue or flooring manufacturers would recommend their products be used if the American standard ASTM 1869-11 were used. There were differences noticed in the water vapour uptake rate depending on CaCl$_2$ particle size distribution, more in APPENDIX 2. This would indicate that the MVER would have been lower if the commercial CaCl$_2$ kit was used.

The MIP analysis of the different cement pastes showed initially (after 14 days) that the cumulative pore volumes of cement pastes with slag as cement replacement (w/c = 0.38 & 0.50) were higher than the reference cement pastes at 8.6% & 1.0% respectively. After 2 months though this changes in the cement paste w/c = 0.38, whereby the cumulative pore volume is 1.7% lower with the slag replacement samples compared to the reference. The pore volumes of the w/c = 0.50 series are equal after the same hydration duration (61 days).

Furthermore, the pore sizes of the cement pastes differ with the use of slag as a cement replacement and is measurable in both w/c series. The percentage of pores under the size of 0.02 µm is lower after both intervals (14 & 61 days). After 61 days, the 0.38 series cement paste with slag had a ca 10% higher pore volume in the size range lower than 0.02 µm compared to the reference. Similar findings were measured in the 0.50 series.

The DVS (dynamic vapour sorption) measurements showed clearly that the inclusion of slag increased the sensitivity of the equilibrium RH when the cement paste loses moisture. This was measured in both w/c concretes. This can be problematic during the preparation phase whereby thermal energy (i.e. heat) from the drilling operation can have a larger effect on the equilibrium RH when this particular type of slag is added.
7 FURTHER RESEARCH

The saltwater vapour method employed in the American flooring method ASTM 1869-11 has its limits i.e. the rate of weight increase in situ is unknown. Only two measurements are carried out, at the beginning and at the end. More data on the rate of moisture uptake would give more details on the mass transfer process in the concrete. Is it constant during the time of measurement or does it proceed with a high influx at the beginning, first 24 hours, followed by a gradual decrease in uptake?

The study showed that regardless of when the moisture was absorbed by the salt, that the vapour emission rate was higher than all glue and floor manufacturers recommended. How were these levels established and are these applicable to today’s manufacturing practices?

The inclusion of slag in this project showed some microstructural changes to the pore radius distribution including the RH equilibrium at particular water weight losses. There are different slag types available on the market with different properties (smelter conditions and ore, slag cooling and grinding rate etc.) Inclusion of a variety of slag types would be especially useful in determining how mineralogy and particle size effect RH measurements and MVER.

Investigation work into the performance of two different CaCl₂ media show that there are differences. The standard ASTM 1869-11 only stipulates a quantity of CaCl₂ and not a PSD which the author shows having a significant impact on the results, see Appendix 2.

REFERENCES

Bfra-19950266: Göteborg.

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APPENDIX

1. Drilling scheme and observations for RBK measurements preparation:
2. Physical properties of CaCl₂ as used in the RH experiments.
1 Drilling scheme and observations for RBK measurements during drill hole preparation.

Below in Appendix 1, some experimental work is presented which were measured during the drilling step for the RBK measurement method.

**FIGURE 1.** IR temperature measurements from drill hole in concrete w/c = 0.38, continuous drilling to depth (blue data points) vs. short bursts getting shorter the closer the desired bore depth is approaching (grey and orange data points). Desired depth 60 mm

**FIGURE 2.** Drilling scheme adopted for RBK measuring technique. The data points (orange and blue circles) indicate the time interval for drilling “bursts”. From ca 50 mm, the bursts are...
reduced to 1 second. The black squares some the measured temperature after each drill burst. At the desired drilling depth (and ca 5 mm before this) a temperature of 25°C was measured.

2 Physical properties of CaCl₂ as used in the RH experiments

In the water vapour absorption tests, finely ground pure CaCl₂ was used to measure the MVER (mean vapour emission rate). Towards the very end of the study, a vapor kit was purchased from USA. This appendix presents the physical data of measurements carried out to compare absorption performance of the two CaCl₂ media.

SEM of powdered CaCl₂ 98% purity

**FIGURE 2.1. SEM micrograph of CaCl₂ powder x 100.**

**FIGURE 2.2 SEM micrograph of CaCl₂ powder x 500.**
FIGURE 2.3. SEM micrograph of CaCl$_2$ powder x 2000.

FIGURE 2.4. PSD MERCK CaCl$_2$ powder.

TABLE 2.1. MERCK CaCl$_2$ PSD Results.

<table>
<thead>
<tr>
<th></th>
<th>Dx (10) [μm]</th>
<th>Dx (50) [μm]</th>
<th>Dx (90) [μm]</th>
</tr>
</thead>
<tbody>
<tr>
<td>MERCK CaCl$_2$ 1</td>
<td>11,8</td>
<td>34,8</td>
<td>77,0</td>
</tr>
<tr>
<td>MERCK CaCl$_2$ 2</td>
<td>11,0</td>
<td>32,5</td>
<td>76,4</td>
</tr>
<tr>
<td>MERCK CaCl$_2$ 3</td>
<td>10,8</td>
<td>31,8</td>
<td>74,8</td>
</tr>
<tr>
<td>Mean</td>
<td>11,2</td>
<td>33,1</td>
<td>76,1</td>
</tr>
</tbody>
</table>

Commercial CaCl$_2$ used in ASTM F1869; Anhydrous Calcium Chloride Moisture Test Kit (purchased from USA; VaporScore)
FIGURE 2.5. CaCl$_2$ product size straight from the airtight packaging.

FIGURE 2.6. CaCl$_2$ product sizes that didn’t pass the protective sieve for PSD instrument (ca 3 mm).

FIGURE 2.7: PSD of ASTM 1869-11 approved CaCl$_2$ particles.
**TABLE 2.2. MERCK CaCl₂ PSD Results.**

<table>
<thead>
<tr>
<th>Vaporkit CaCl₂</th>
<th>Dx (10) [μm]</th>
<th>Dx (50) [μm]</th>
<th>Dx (90) [μm]</th>
</tr>
</thead>
<tbody>
<tr>
<td>Vaporkit CaCl₂</td>
<td>1260</td>
<td>2040</td>
<td>2930</td>
</tr>
<tr>
<td>Vaporkit CaCl₂</td>
<td>1450</td>
<td>2080</td>
<td>2920</td>
</tr>
<tr>
<td>Vaporkit CaCl₂</td>
<td>1210</td>
<td>2010</td>
<td>2930</td>
</tr>
<tr>
<td>Mean</td>
<td>1310</td>
<td>2040</td>
<td>2930</td>
</tr>
</tbody>
</table>

Only particles < 3 mm were measured ca 12 % by mass were > 3mm

**CaCl₂ (powder) moisture absorption observation and measurements**

![Diagram of CaCl₂ moisture absorption over time](image)

**FIGURE 2.8. Water absorption in CaCl₂ powder over time in 50% climate chamber**

Initial trial on CaCl₂ moisture absorption capability with constant air moisture content. Data lacking though from 4 hours to 72 hours (weekend).
FIGURE 2.9. Experimental setup of CaCl$_2$ powder, absorption rate difference. Spread out over petri dish (Ø 8cm) vs. heaped. Placed in climate room 20°C ± 2°C & RH 50% ± 5%.

FIGURE 2.10. Experiment 24 hrs after initial exposure to climate room (20°C ± 2°C & RH 50% ± 5%)
FIGURE 2.11. Long term water absorption of 4 g CaCl₂ in a constant climate. Spread powder in glass petri dishes.

FIGURE 2.12. Experiment on water absorption from spread CaCl₂ powder during first 48 hrs. “1” & “2” are petri dishes on the side of the short edge of the stainless steel container, see FIGURE 2.9.

FIGURE 2.13. Long term water absorption of 4 g CaCl₂ in a constant climate. Spread powder in glass petri dishes.
FIGURE 2.14. Experiment on water absorption from “heaped” CaCl$_2$ powder during first 48 hrs.

FIGURE 2.15. Comparison of average moisture uptake with one standard deviation. Black = spread powder; red = heaped powder.

Conclusions from initial tests are that the spread CaCl$_2$ absorbs more water vapour from ca 3 hrs onwards. The standard deviation is smaller in the heaped piles, but the positioning of the petri dishes has a significant effect on the rate of water vapour absorption (the flow of moisture laden air over petri dish).

Effect on absorption based on commercial CaCl$_2$ packs vs powdered CaCl$_2$.

The entire contents of 3 commercial CaCl$_2$ kits were placed in 3 petri dishes as can be seen in FIGURE 2.13 below (the petri dishes with the spheres). The same weight of powdered
CaCl₂ were placed in 3 identical petri dishes. The weight gain over time in a climate room 50% RH (20 °C) was noted and is presented in the graph below. The purpose of this experiment was to see if the surface area difference had an impact on the delinquency (weight gain). The powdered CaCl₂ had a higher initial uptake especially in the region of interest i.e. MVER (mean vapour emission rate).

**FIGURE 2.16.** Comparison of MERCK CaCl₂ vs VAPORSCORE (ASTM) CaCl₂. Initial layout; top left = MERCK, bottom left = VAPORSCORE. Time = 0 hr 0 min.

**FIGURE 2.17.** Commercial CaCl₂ kit (VAPORSCORE) (bottom left; middle top & bottom right) vs powdered CaCl₂ as used in experiments. Initial set up t = 6 hr.
FIGURE 2.18. Commercial CaCl₂ kit (bottom left; middle top & bottom right) vs powdered CaCl₂ as used in experiments. Initial set up t = 24 hr.

FIGURE 2.19. Commercial CaCl₂ kit (bottom left; middle top & bottom right) vs powdered CaCl₂ as used in experiments. Initial set up t = 124 hr.
Figure 2.20. Average moisture uptake of three petri dishes \( t = 0-30 \) hr

Figure 2.21. Average moisture uptake of 2 x three petri dishes \( t = 0-130 \) hr
Conclusions from the comparison study show that the water vapour absorption performance of the powdered MERCK CaCl₂ is higher than the ASTM CaCl₂ kit. This can be seen after the first hours of testing, see FIGURE 2.20 and FIGURE 2.21. The data points come closer.
together, but this is first after ca 124 hrs. The amount of moisture absorbed in the MVER
tests on concrete, see TABLE 4.4.2.1 are low and is equivalent to ca 2-3 g\textsubscript{vapour} on 8g of
CaCl\textsubscript{2} powder.

The results within the group see FIGURE 2.22 and FIGURE 2.23 are more stable with a
lower standard deviation due to a change in the positioning of the petri dishes, whereby they
were placed on top of the stainless-steel tray, see FIGURE 2.9 and FIGURE 2.16.

The rate of performance of CaCl\textsubscript{2} is not stipulated in the standard and on reflection with
these results one can clearly see a difference between “CaCl\textsubscript{2}”. 

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