Cryosuction Experiments on Concrete Containing Ground Granulated Blast-Furnace Slag: Influence of Temperature, Air Entrainment And Salt

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ABSTRACT

Frost deterioration of concrete is an important durability issue for structures exposed to high degree of saturation, low temperatures and de-icers. The material can then be severely damaged with internal cracking and/or scaling of the surface, which can lead to e.g. reduced protection of the reinforcement and loss of load bearing capacity. Experiments with liquid uptake in concrete using different temperature cycles was made to study cryosuction. The material used was concrete with different air content and different replacement levels of ground granulated blast-furnace slag (GGBS). The concrete samples were preconditioned by capillary suction. Three temperature conditions were used: constant temperatures of +20 °C and -20 °C, and temperature that cycled between -20 °C and +20 °C. As liquid medium, deionized water and a 3% NaCl salt solution were used. Air entrainment generally increased the liquid uptake. The amount of GGBS and the NaCl...
concentration in the liquid did not have any significant impact on the liquid uptake in these experiments.

**Key words:** Concrete, Frost action, Supplementary Cementitious materials, Cryosuction, Durability, Testing

1. **INTRODUCTION**

1.1 **General**

Concrete is the most used building material in the world and the cement industry releases 5-8% of the total man-made carbon dioxide emissions [1]. These emissions can be reduced by using SCM, supplementary cementitious materials, such as ground granulated blast furnace slag (GGBS), to replace a part of the cement used when casting concrete. When using different SCMs, the changes in chemical and material properties need to be taken under consideration e.g. the hydration progress is often slowed down [2] and with GGBS the pore structure of the concrete becomes denser [3], [4] unless it is carbonated [5]. The capillary suction properties are highly dependent on the pore structure and the denser pore structure of a concrete with GGBS results in an increased discontinuity and tortuosity in the material [6]. There is an observed correlation between the capillary suction properties of a concrete surface and frost scaling resistance [7], where a low liquid uptake from capillary suction results in higher frost scaling resistance.

The concrete can be protected against frost deterioration by a proper air void system since this allows the pore liquid to be sucked into the air voids and freeze without creating any pressure on the surrounding material as explained by Powers and Helmuth [8]. But if there is an increase of liquid uptake the protective effect decreases. The positive effect of the air void system when using air entrainment agent has been shown in several studies [9] [10] [11].

Fagerlund presented the theory of critical degree of saturation [12], which predicts that a concrete will get internal damage due to freezing conditions when it has an actual degree of liquid saturation that is higher than a critical degree of saturation. The effective spacing factor is increased with a progressive saturation of the air voids from the liquid uptake, starting by the smallest air voids, which increases the distance between unsaturated air voids [13]. Large air voids are almost never saturated through liquid uptake from capillary suction at non-freezing temperatures.

At non-freezing temperatures, capillary suction is the controlling transport property for unsaturated concrete [14], [15]. In 1921, Washburn presented the liquid uptake into a porous material with the assumption that the pores have an cylindrical shape with different radii [16]. The air voids with smaller radii are filled with liquid before the larger air voids start to take up liquid [13].

There is an observed increase of liquid uptake when a porous material is exposed to freezing temperatures, compared to non-freezing temperatures, if there is access to an external reservoir of liquid [17]. This liquid uptake is due to the presence of mechanisms like frost heave but also because of a pumping effect which is more pronounced if the temperatures are cycled. Taber presented the theory of frost heave in soil in 1930: if a porous material is exposed to freezing temperatures, the capillary suction properties are highly dependent on the pore structure and the denser pore structure of a concrete with GGBS results in an increased discontinuity and tortuosity in the material [6]. There is an observed correlation between the capillary suction properties of a concrete surface and frost scaling resistance [7], where a low liquid uptake from capillary suction results in higher frost scaling resistance.

The concrete can be protected against frost deterioration by a proper air void system since this allows the pore liquid to be sucked into the air voids and freeze without creating any pressure on the surrounding material as explained by Powers and Helmuth [8]. But if there is an increase of liquid uptake the protective effect decreases. The positive effect of the air void system when using air entrainment agent has been shown in several studies [9] [10] [11].

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temperatures on one side and non-freezing temperature on the other, there will be a transport of unfrozen liquid towards the colder regions, where the liquid will freeze and form an ice lens, due to the difference in chemical potential between the ice and the liquid [18]. This phenomenon is described in concrete as the macroscopic ice lens theory. It has been shown by Rosenqvist and Lindmark that a growing ice lens can create damage to concrete with high water/binder ratios [9], [19]. A thin film of unfrozen water covers the interface between ice and the inside porous concrete, this thin film allows liquid to flow and supply the ice lenses [19]. For an ice lens to grow, the concrete needs an external liquid reservoir, otherwise the growing ice lens will drain the surrounding pores of liquid and the growth of the ice lens will cease [9]. Note that there will be no scaling damage on a concrete surface in the absence of an external liquid [9].

There is also an observed pumping effect during multiple freeze thaw cycles that results in an increased liquid uptake [20]. The pumping effect is a result of the thermodynamic non-equilibrium between the ice inside the air voids and the liquid in contact with these, which requires that the liquid pressure is below atmospheric pressure [21]. As a consequence, liquid flows towards the air pores as they act as pumps in the material during freezing [21]. During freezing, ice crystallization starts from the largest air voids containing liquid, and as the temperature continues to decrease, the liquid in successively smaller air voids freezes [21].

The phenomenon of increased liquid uptake during freezing has been described by different models based on the poromechanical equations by Coussy [22]. Eriksson developed a hygro-thermo-mechanical multiphase model that can simulate air-entrained concrete in 3D, including phase change and hydraulic pressure in samples with non-saturated air voids [21], [23]. Unfortunately, there is at present a lack of experiments against which to validate poromechanical models.

\subsection{1.2 The importance of liquid uptake in frost deterioration}

The overall aim of this study was twofold: (1) To make measurements interesting for researchers who build frost models which need experimental verification. These models help us understand different aspects of the complex deterioration mechanisms behind frost damage [Gong Jacobsen] To be able to use an experiment for a validation of a model of any kind, a lot of data is needed. Both on the material characteristics and the exposure. All data needed for this study can not be found in this paper but more characteristics of similar materials can be found in [5] and theoretical values used for poromechanical simulations can be found in [23].

(2) In the research of salt frost scaling there are two rather different explanations for the scaling: too high water content in the surface or damage caused by the ice layer on the surface. With experiments of the liquid uptake in different concrete materials during different temperature cycles, without any ice layer on the surface, we will be able discuss which circumstances that seem to promote a high water content in the surface and therefore a sensitivity to frost damage [27]. The results will also be discussed together with the results from scaling test performed by Strand [5], as the concrete material in the present study is cast with recipes similar to the ones in that study to compare the liquid uptake to the salt frost scaling in a representative material.

This study was made to increase our knowledge of the role of liquid uptake in the mechanisms behind freeze-thaw action and the damage it causes to concrete structures. The measurements were made to examine the liquid uptake in concrete with different amount of air entrainment and GGBS when exposed to different temperature conditions and liquid solutions.
2 Cryosuction Experiment

The objective of the experiment was to quantify the effect of cryosuction by exposing samples to three different air-controlled temperature conditions, a temperature cycling between -20 and +20 °C, a constant temperature of -20 °C and a constant temperature of +20 °C. The bottom of each sample was in contact with an unfrozen liquid, consequently, this set up makes it possible to avoid the influence of an ice-layer on the exposed surface, which is an important part of the scaling mechanism [9], and has a better chance to capture the influence of cryosuction only.

2.1 Materials

The recipes for the concrete are presented in (Table 1). For each binder composition there were two concretes cast, with and without air-entrainment agent (AEA). An air-entrainment agent, Sika Air pro 5%, and a superplasticizer (SP), Sika Viscocrete RMC 520, were used to get a desired air content and slump, Table 1. The target value was 5% air content when using AEA and 165 mm slump [5]. The recipes with AEA were not adjusted for the extra volume. The air content and slump were measured on fresh concrete according to standards EN 12350-7 [24] and EN 12350-2 [25].

<table>
<thead>
<tr>
<th>Material</th>
<th>W/B</th>
<th>Cement 1) kg/m³</th>
<th>GGBS kg/m³</th>
<th>0-8 mm kg/m³</th>
<th>8-11 mm kg/m³</th>
<th>11-16 mm kg/m³</th>
<th>SP 2)</th>
<th>AEA 2)</th>
<th>Air Content Volume-%</th>
<th>Slump mm</th>
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<td>430</td>
<td>0</td>
<td>966</td>
<td>285</td>
<td>536</td>
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<td>2.30</td>
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<td>536</td>
<td>1.1</td>
<td>0.45</td>
<td>5.50</td>
<td>205</td>
</tr>
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</table>

1) CEM I 42,5 N SR3 MH/LA. (Slite, anläggningscement, Cementa AB)
2) The amount of admixture was dosed in % of binder weigh

The materials were cast in molds of 400 mm × 400 mm × 150 mm, moisture sealed for 24 hours, then demolded and water cured in 20 °C lime saturated water for 2.5 months. Samples were then drilled and sawed to cylinders with sawed surfaces on top and bottom, and with the dimensions of 50 mm diameter and 50 mm height. The samples were then stored in 20 °C lime saturated water for an additional 3 months.

2.2 Method

The samples were moisture sealed on top and sides using a 1 mm thick butyl tape that had been tested to be very water vapor tight (Isola-Platon AB), leaving only the sawed bottom exposed. The surface was dried in air for 15-30 min to ensure that the tape adhered well.
**Experimental set-up**
The samples were placed on a plastic grid, and in contact with deionized water or 3% NaCl solution. There were two different set-ups depending on if the samples were exposed to freezing temperatures or not. The set-up at constant +20 °C had a glass lid to prevent moisture loss by evaporation.

The set-ups for constant -20 °C and cycling between +20 °C and -20 °C used an insulated container, with a 50 mm thick XPS insulation that covered the bottom and sides, as is seen in Figure 1 and 2. A 20 mm XPS insulation in which the samples were placed, was positioned 8 mm above the plastic grid, to ensure that the insulation was not in contact with the liquid. Half of each sample was above the insulation and exposed to the temperature-controlled air. A heating coil was used to prevent the liquid from freezing. The heating coil was attached underneath the plastic grid. The plastic grid was supported and levelled by four screws standing on the bottom of the container. The liquid temperature was regulated with a PI-regulator to avoid decreasing below 3 °C. The temperature gradient was quantified by temperature measurements at three levels (top, middle and bottom) in two dummy samples, Figure 3, that were never weighted, one for constant temperature of -20 °C and one for cycling temperature between +20 °C and -20 °C. The temperature of the dummy samples was measured inside the material. Holes was drilled to the centre of the sample (25 mm depth) at the three levels, and the sensors were placed inside, and the holes were filled with cement paste to keep the sensors in place.

![Figure 1](image-url) *Figure 1. The test set-up for -20 °C and for cycling between +20 and -20 °C with one sample inserted.*
The liquid uptake of the samples was measured at regular intervals. The samples were taken out of the containers, dried with a wet cloth and weighed on a ±0.001 g balance. Samples in constant +20 °C and cycling between +20 °C and -20 °C were weighed once a week. The temperature cycle was 24 hours. Samples at constant -20 °C were weighed once a month. Samples exposed to freezing temperatures were weighted in a thawed state. Three parallel specimens were tested of each material in each temperature exposure.

Figure 3. Temperatures in a dummy sample on three levels, in the air and water for cycling +20/-20 °C experiment. The freezing curve is representative for the -20 °C experiment as well.

3 RESULTS AND DISCUSSION

The result is presented in Figures 5-8 as liquid uptake per area (kg/m²) of the bottom of the samples as a function of time. The main interest in the evaluation is the effect of the NaCl solution, the replacement level of GGBS, the three different temperature conditions and the air entrainment agent.
As the samples were preconditioned by capillary saturation, the moisture uptake that is studied in this paper is small compared to the moisture content achieved during sample preparation, as is schematically shown in Figure 4.

![Figure 4](image)

**Figure 4.** Schematic description of how the moisture content of a sample changes during sample preparation and measurement. Phase A is the water storage after casting; B is the water storage of the core drilled specimen; C is an example of the measurements presented in this paper. Insert in C shows example of uptake during phase C.

### 3.1 General observations

In Figure 5, the raw data from selected measurements are presented for material 4 from Table 1 with three replicate samples for each temperature condition. No visible damage (cracks or scaled material) was found in the samples after the tests. The measured liquid uptake is small, and as the samples are dried by hand with a wet cloth before each weighing, there are small differences in how much liquid that remain on the surface of the sample, and this is the most probable cause of the scatter in some curves. The dashed lines are linear regressions of the data between 10 days and the end of measurement. The results presented in Figure 7 and 8 are based on linear regressions at 100 days and not the actual measurement at 100 days, this is to reduce the noise in the measurement.
Figure 5. Example of experimental result. Results for material 4 with deionized water (Top) and a 3% NaCl solution (Bottom) for the three temperature conditions. For each temperature condition and NaCl concentration, three specimens were tested. The solid lines represent actual measurements on one specimen and the dotted line represent linear regressions of the data between 10 days and the end of measurement.

The results generally show larger spread and larger mean value for the cases with freezing temperatures (constant -20 °C and cycling between +20 °C and -20 °C) compared to the case at constant +20 °C.

3.2 The effect of air entrainment on liquid uptake,

When an air entrainment agent (AEA) is used in concrete, it produces air voids [26] with a diameter of 5 – 100 μm that results in a higher porosity and a more open pore system [26]. The effect this has on liquid uptake in the present experiment is shown in Figure 6, which present an overview of the results of all the measurements. The air content in the materials was determined in the fresh concrete. The same recipes were used in the work of Martin Strand, where more data on the air void characteristics can be found [5]. In Figure 6A-C it is seen that the spread in the result is significantly lower for the +20 °C exposure than for the other exposures, and that the samples with entrained air generally have a higher liquid uptake as the grey dashed lines are generally above the black lines. In the literature, most studies on liquid uptake in concrete come to the conclusion that AE increases the uptake, as AE gives an
increased porosity [28], [38]. However indications of the opposite behavior for high FA concrete is found in [39]. Even though the liquid uptake is higher is the degree of saturation still lower which gives protection of frost deterioration. It is also seen in Figure 6A-C that the samples exposed to freezing temperatures generally have a more rapid liquid uptake.

### Figure 6. Liquid uptake in all measurements for the three different temperature conditions. Grey dashed lines for samples with AEA and black lines for those without.

#### 3.3 The effect of replacement level of GGBS for liquid uptake

With an increasing replacement level of GGBS the pore structure becomes denser, but this does not seem to have any effect on the liquid uptake in the present study, see Figure 7 A-C. The results are presented as the liquid uptake after 100 days (on the linear regressions). Looking at the different materials, there are mixed results concerning which replacement level of GGBS that gives the highest liquid uptake and in some cases the results are almost the same for all three cases.

#### Figure 7. The mean liquid uptake after 100 days from linear regression of the measurements on three samples with three levels of GGBS replacement and three different temperature conditions A-C. The x-axis show if air-entrainment agent was used (AEA) or not (no AEA), and if the liquid reservoir was de-ionized water (No salt) or 3 weight-% NaCl (salt).
3.4 The effect of salt

The effect of salt in the liquid reservoir on the liquid uptake is presented in Figure 8 A-C. There is no trend for the liquid uptake in the presence or absence of salt. When we look at the different materials, there are mixed results concerning if de-ionized water or a 3% NaCl solution in the liquid reservoir gives the highest liquid uptake.

**Figure 8.** The mean liquid uptake after 100 days from measurements, on three samples with either de-ionized water (No salt) or 3 weight-% NaCl (salt) in the liquid reservoir. Measurements for the three different temperature conditions in A-C. The x-axis describes the condition of the sample: If air-entrainment agent was used (AEA) or not (no AEA), and what replacement level of GGBS in weight-% (S0 = 0 %, S35 = 35 % and S70 =70 %)

3.5 General discussion

The reason for the larger spread in the results during freezing temperatures could be that the higher liquid uptake in some samples was caused by the filling of stochastically distributed large pores (diameter of 1 mm or more) in the freezing. The samples were stored in lime saturated water 3 months before the experiment began so most of the pores that should be filled with liquid through capillary suction at room temperature, were probably already filled at the start of the experiment.

Tomography-results of a mortar that has been exposed to capillary suction at room temperature are presented in Figure 9. Note that this mortar does not have the same recipe as the concrete used in the present study. The tomography is performed by the author, but not in this study. Tomography is an imaging procedure in which a narrow beam of x-rays is aimed at a sample and quickly rotated around it, producing signals that are processed by the machine’s computer to generate cross-sectional images. Once a number of successive images are collected by the machine’s computer, they can be digitally “stacked” together to form a three-dimensional (3D) image of the sample. The material that absorbs the x-ray the best become brighter. In this way can we distinguish between different materials with in the material. The tomography results in this study have been processed in ImageJ, and the tomograph is a Zeiss Xradia XRM520.
Figure 9. Tomograph image of cement mortar that has been stored in water and has taken up liquid through capillary suction from all sides. Sample size 10 mm x 10 mm. Imaging done by the first author.

Figure 9 shows that there still is air in the larger air voids of the material even though it is capillary saturated. Observation and estimation of the large voids of 1mm and larger on the surfaces of the samples in this study have been performed, Figure 10. Several voids of 1-5 mm diameter were found in all concrete samples.

Figure 10. Example of larger air voids on surface of sample from the study, measurements were made on 5 samples from each material.

Tomography examples, Figure 9, and this surface observation, Figure 10, can strengthen our hypothesis that larger pores in the freezing zone can be the reason for the larger scatter in freezing temperatures.

Temperature condition

In the present study, the liquid uptake is larger during freezing temperatures than at +20 °C. As the samples were stored at lime saturated water for 2.5 months before the experiment began, the samples were already at high moisture levels, and the increased liquid uptake for freezing temperatures is due to the effect of cryosuction.

For the freezing temperatures, the liquid uptake is not clearly larger at temperature cycling than at constant -20 °C as some studies have shown [20] [28]. The difference could be that in the other studies, the liquid reservoir was able to freeze [20]. The increased liquid uptake during freezing temperatures is not a direct indicator of the surface scaling damage, but is more
connected to the degree of saturation in the specimen [20]. For a concrete with air entrainment, it takes more time or cycles until the critical degree of saturation is reached, even though the liquid uptake is higher than without AEA, but it still has a greater resistance to frost damage due to the larger air voids from the AEA. A concrete without AEA, has a lower liquid uptake but it takes less time or fewer cycles until the critical degree of saturation is reached and consequently this concrete has an higher risk for frost damage [28].

A hypothesis is that the larger uptake and the larger spread in the results at freezing temperatures is due to the occurrence and position of large pores, with a diameter of 1 mm or more in the freezing zone of the samples. For the condition with a cyclic temperature, this freezing zone is changing in position as the temperature changes and activates more of the larger pores, causing a larger spread in the results.

The gradual saturation of the pores through liquid uptake during freezing temperatures is necessary for causing severe damage, as many freeze-thaw cycles are often necessary until damage is observed [29].

Replacement level of GGBS
The pore structure of concrete changes with the addition of SCM’s [2] and, becomes denser with more GGBS [13]. However, the liquid uptake in this study does not seem to be affected by the replacement level of GGBS. With the samples already at a high moisture level at the start of the experiment, the additional liquid uptake will mostly happen in the larger pores and due to the cryosuction below freezing temperatures. The study by Strand shows that an increased slag fraction decreases the permeability by capillary suction measurements of an concrete that has not been aged and dried at the start of the experiment [5]. The effect of GGBS on frost durability is varied depending on the replacement level and test method, but a denser pore structure gives an lower rate of liquid uptake, which leads to increased frost durability and scaling resistance [27]. The effect of GGBS under standard freeze durability testing of concrete according to ASTM C 672 [30] with a large (50-60%) replacement level of GGBS shows greater scaling, but lower replacement level of GGBS (around 25%) shows less scaling, both compared to concrete made solely with Portland cement [31]. Studies with the CIF-test show an decrease in scaling due to increased level of GGBS (up to 50%) replacement [32]. The study by Strand shows that GGBS decreases the salt frost scaling in non-carbonated concrete (up to 70%) compared to CEM I [5].

Salt concentration in the liquid reservoir.
The phenomenon with a NaCl pessimum at low NaCl concentration [5] [9] [33], where the largest salt frost scaling damage occurs [34] [35] [36], cannot be explained in this study by an increased uptake of liquid with salt, as a NaCl concentration of 3% in the liquid reservoir did not give a higher liquid uptake than deionized water in the liquid reservoir. Other studies show similar results in that the NaCl concentration does not have a clear increase or decrease in liquid uptake for concrete containing different amount of GGBS at around 0-3% NaCl concentration [14]. The study by Strand shows that the salt frost scaling damage in concrete samples exposed to low NaCl concentrations of 1 or 3% are higher compared with the salt frost scaling damage in the same type of samples exposed to a higher NaCl concentration of 9% or pure water [5], as previous studies have already shown.
4 CONCLUSIONS

Note that this study investigated the added liquid uptake in samples that were already at high moisture levels before the liquid uptake experiment started. The surface of the samples in the experiment does not freeze, to isolate the effect of temperature variation on liquid uptake.

- The liquid uptake does not seem to be influenced by the presence of NaCl in the water.
- The liquid uptake does not seem to be influenced by an increased amount of GGBS replacing cement (CEM I).
- Freezing temperature that cycles between +20 °C and -20 °C and constant -20 °C have a tendency to give higher liquid uptake than in the case of constant temperature of +20 °C.
- Samples with AEA have a tendency to have a higher liquid uptake than the samples without AEA.
- The samples exposed to cycling between +20 °C and -20 °C, and constant t -20 °C have a significantly higher spread in the liquid uptake than the samples at constant temperature +20 °C.

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