Abstract
The objective of this investigation is to study water uptake during cyclic freeze/thaw exposure for mortar specimens submerged in salt solution. This is done the following way: At regular intervals during a freeze/thaw cycle, test specimens (5 mm thick, sawn discs) are removed from the salt solution, put on a scale to register the weight gain, and then put back into the salt solution. The experiment has been carried out with different test conditions and different mortar compositions. The results show that the water uptake during a freeze/thaw cycle consists of a reversible part and an irreversible part.

1. Introduction
If concrete is submerged in water or salt solution and then subjected to freeze/thaw action, the concrete will show a net water uptake, see e.g. review in /1/. Investigations show that the water uptake is higher when a salt solution is present instead of pure water. In most cases the water uptake is even higher than if the concrete had been stored in the same solution at a constant temperature above 0°C. Moisture transport in the surface layer is an important key to understand the mechanisms causing frost deterioration of concrete. However, there is no concurrent explanation for this phenomenon.

1.1 Previous observations and theoretical explanations of water uptake
Both thermodynamics and physics have been applied to explain the water uptake, e.g.

- Lindmark /2/ refers to the osmotic ice body growth proposed by Powers, where unfrozen water is drawn to an ice front, e.g. an ice lens formed in the concrete surface layer, because of the lower free energy of ice.
- Geiker and Thaulow /3/ focus on the process of ice melting in the concrete. Thawing of ice is accompanied by a volume contraction, and if unfrozen liquid is available it may be sucked into the pore system to fill in the gap, so to say.
These hypotheses forecast water uptake at different stages during a freeze/thaw cycle. According to Lindmark, the water uptake will primarily take place during freezing, whereas Geiker and Thaulow forecast a water uptake during thawing. The hypothesis of Lindmark has been experimentally verified, but the investigation did not include thawing: The weight change was registered for saturated mortar discs placed in salt solution at constant sub-zero temperatures, where the pore solution of the mortar froze while the outer salt solution was still unfrozen. A recent study carried out by Rønning /4/ indicates that though the net water uptake during a full freeze/thaw cycle may be positive, the moisture transport is reversed during thawing, where the concrete actually expels water.

1.2 Objectives of the present study

Previous investigations of water uptake of concrete or mortar have been based on registration of weight changes, where weight gain is interpreted as a measure of water uptake. This method is experimentally simple and offers a high degree of accuracy. However, it has one major disadvantage: It is not possible to perform measurements, when the outer solution is frozen. This will often be the case during natural exposure situations and during testing, e.g. when using the Swedish test method SS 13 72 44 /5/, where the concentration of the outer NaCl solution is 3.0% and the minimum temperature is –20°C.

For this reason, Jacobsen and Sellevold /1/ only measures the weight of the specimens between freeze/thaw cycles, when the outer solution is unfrozen. Lindmark /2/ employs combinations of temperature and concentration, where the temperature is above the freezing point of the outer solution (e.g. at –16°C he uses a 19.7% NaCl solution), but he doesn’t consider cyclic exposure.

In the present study, the water uptake is measured during a temperature cycle, while the test specimen is exposed to a strong salt solution, so the outer solution is unfrozen all the time during the cycle. The aim of the study is twofold:

- to study water uptake during cyclic freeze/thaw exposure in general
- to state when during a freeze/thaw cycle the water uptake actually takes place, and thereby prove if it is the mechanism described in the hypothesis of Lindmark or by Geiker and Thaulow (or both) that governs the water uptake.

2. Experimental programme

The test programme covers three types of mortar. They are submerged in salt solution and exposed to freeze/thaw cycles, while weight changes are recorded. The test programme includes two levels of each of the variables: w/c ratio, air content, concentration of salt solution, and minimum temperature in the temperature cycle. The intention is to give a rough idea of which parameters are the most important.
2.1 Method
Each mortar is tested under a number of different test conditions, i.e. various combinations of temperature cycle and concentration of salt solution, see Table 1.

Table 1: Test conditions a, b, and c.

<table>
<thead>
<tr>
<th>NaCl solution</th>
<th>15%</th>
<th>20%</th>
</tr>
</thead>
<tbody>
<tr>
<td>Cycle A</td>
<td>a</td>
<td>b</td>
</tr>
<tr>
<td>Cycle B</td>
<td>c</td>
<td></td>
</tr>
<tr>
<td>Tmin=-8°C</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Tmin=-14°C</td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

![Graph showing temperature over time for Cycle A and Cycle B, with cycles marked at 2, 4, 6, 8, and 10 hours.]

Every 45 minutes during the test, 3 test specimens (Ø100 mm mortar discs) are removed from the salt solution, wiped with a paper towel and put on a scale, before they again are placed in the salt solution.

2.2 Materials
The measurements are carried out for three types of mortar. The mix design starts from the mixture used in EN 196-1 /6/, i.e. they have the same paste content, but w/c ratio and air content is changed. The mix compositions are stated in Table 2.

Table 2: Mortar composition. Amount of cement, water, sand, and air entraining agent is stated as gram pr. litre mortar without air. The air void parameters have been measured on hardened mortar samples according to EN 480-11 /7/.

<table>
<thead>
<tr>
<th></th>
<th>40A01</th>
<th>40A03</th>
<th>55A01</th>
</tr>
</thead>
<tbody>
<tr>
<td>Cement</td>
<td>584</td>
<td>584</td>
<td>483</td>
</tr>
<tr>
<td>Water</td>
<td>234</td>
<td>234</td>
<td>266</td>
</tr>
<tr>
<td>Sand</td>
<td>1539</td>
<td>1539</td>
<td>1539</td>
</tr>
<tr>
<td>Air entr. agent</td>
<td>0.58</td>
<td>1.76</td>
<td>0.48</td>
</tr>
<tr>
<td>w/c</td>
<td>0.40</td>
<td>0.40</td>
<td>0.55</td>
</tr>
<tr>
<td>Total air cont.</td>
<td>0.07</td>
<td>0.08</td>
<td>0.08</td>
</tr>
<tr>
<td>Spacing factor</td>
<td>0.29</td>
<td>0.17</td>
<td>0.20</td>
</tr>
</tbody>
</table>

The mortar is used for casting Ø100 mm cylinders. They are treated according to SS 13 72 44 /5/, i.e. they are demoulded 1 day after casting, stored in water the subsequent 6 days and then stored at 20°C and 65% RF until the testing starts 26-28 days after casting.
Mortar discs (approximately 5 mm thick) are sawn from the cylinders 7 days before testing. At the beginning of the test, the mortar discs are placed 3 days in water at 20°C before they are exposed to salt solution and thermal action.

3. Results

The measured weight changes during the cyclic freeze/thaw exposure are shown in Figure 1, Figure 2 and Figure 3 for mix composition 40A01, 40A03 and 55A01, respectively. None of the samples showed sign of damage after the frost exposure, so it has not been necessary to correct the weight measurements for scaling.

**Figure 1:** Water uptake measured for mix composition 40A01 (each curve represents the average of 3 Ø100 mm discs).

**Figure 2:** Water uptake measured for mix composition 40A03 (each curve represents the average of 3 Ø100 mm discs). The curve for cycle a is corrected due to problems with the scale (floating zero point) during the measurements.
As a control measure, the same experiments were performed with a constant temperature of 20°C instead of the temperature cycle. These tests showed only small weight changes, when the specimens were transferred from pure water to salt solution. However, in the experiments with varying temperature, the specimens were first left half an hour in the salt solution to adjust to the new environment before the temperature was lowered. In Figure 1, Figure 2 and Figure 3 the weights measured half an hour after the beginning of the experiment are used as reference weights.

Figure 3: Water uptake measured for mix composition 55A01 (each curve represents the average of 3 Ø100 mm discs). Temperature cycle c is run twice to test repeatability.

The maximum water uptake and the net water uptake after a freeze/thaw cycle, i.e. the water uptake 9.5 hours after test start, can be read in the figures above. The readings are shown in Table 3.

Table 3: Maximum water uptake and net water uptake during freeze/thaw cycles (average for 3 Ø100 mm discs). All values are g/m².

<table>
<thead>
<tr>
<th></th>
<th>40A01</th>
<th>40A03</th>
<th>55A01</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>max</td>
<td>net</td>
<td>max</td>
</tr>
<tr>
<td>Cycle a</td>
<td>8.7</td>
<td>0.2</td>
<td>18.3</td>
</tr>
<tr>
<td>Cycle b</td>
<td>8.9</td>
<td>1.8</td>
<td>14.4</td>
</tr>
<tr>
<td>Cycle c</td>
<td>15.5</td>
<td>0.1</td>
<td>25.3</td>
</tr>
</tbody>
</table>

4. Discussion

The general trend in all the measurements is a water uptake during the 3-hour cooling period and to a less extent also during the period of constant, low temperature. This is followed by a water loss during the period of heating. For most of the samples the net water uptake after one freeze/thaw cycle is close to zero.
4.1 Test conditions
The effect of the concentration of the outer solution can be investigated by comparing curves for temperature cycle a and b in Figure 1, Figure 2 and Figure 3. It does not seem like changing the concentration of the outer solution makes a significant difference, at least not when the concentration is high (15% b.w. or higher).

The effect of the minimum temperature appears when comparing curves for temperature cycle b and c. For 40A01 and 40A03 the maximum water uptake in temperature cycle b is approximately 60% of that in cycle c and for 55A01 this ratio is 75-100%, according to the values in Table 3. Thus it seems like the water uptake increases when the minimum temperature in the cycle is lowered.

4.2 Mortar properties
Measurements of air content in the fresh mortar showed the same air content in the mortars 40A01 and 55A01. However, in the hardened mortars, the air contents differ (7.9% and 8.5%, respectively) and differences between these mortars may as well be due to the different air contents. Therefore, it is not possible to say something conclusive about the influence of the w/c ratio on the water uptake.

The fact that there is an effect of the minimum temperature in the cycle and that the mortar with the largest air content (40A03) also has the largest water uptake calls attention to the temperature dependent contraction of air in the mortar. This may cause a suction, which draws the outer liquid into the mortar. For an ideal gas at constant pressure, the relation between volumes $V$ at different temperatures $T_1$ and $T_2$ (measured in K) is stated in formula (1):

$$V_{T_2} = \frac{T_2}{T_1} \cdot V_{T_1}$$

For example in 40A03 with an air content of 10.7%, the air volume in one mortar disc at 20°C is 4.20 cm$^3$. According to formula (1) the change in air volume, when the specimen is cooled down to –14°C, is 0.49 cm$^3$, i.e. same magnitude as the measured maximum water uptake (0.45 g).

It is not possible to isolate the water uptake caused by air contraction from water uptake caused by other reasons. There may be a supplementary water uptake during cooling and heating, e.g. caused by osmotic ice body growth as explained by Lindmark. And a possible water uptake caused by air contraction may be delayed so it may not be limited to periods of temperature change. However, if air contraction is the dominant cause of water uptake during cooling, formula (1) forecasts a linear relationship where the water uptake increases when the air content increases and vice versa during heating.

In Figure 4 the water uptake during cooling and the water loss during heating are plotted versus the air content in the mortars.
Figure 4: Left: Water uptake during cooling. Right: Water loss during heating. The salt concentration in the outer NaCl solution is 20% for all points.

Figure 4 shows large scatter and the general trend is not clear as the slopes of the regression lines are opposing. It is still plausible that the contraction of air can explain some of the water uptake, but no definitive conclusions can be drawn.

4.3 The meaning of the water uptake
The reversible part of the water uptake is probably not detrimental to the mortar matrix, though an increased moisture transport in the surface layer may have some negative side effects (e.g. on chloride ingress). The irreversible water uptake may be detrimental to the mortar, or it may be a consequence of damage. However, this cannot be concluded for sure in the light of the present results, because none of the samples showed signs of deterioration, and the net water uptake during the freeze/thaw cycles was at the same level as the test uncertainty.

As regards the hypotheses in section 1.1, the data set is in favour to the hypothesis of Lindmark. If the mechanism described by Geiker and Thaulow is going to take place, the water uptake is going to take place during heating, i.e. at the same time as this study shows an expulsion of water.

5. Conclusion

The water uptake during wet, cyclic freeze/thaw exposure has been investigated for three types of mortar by measuring the weight gain during a freeze/thaw cycle.

The results show that the water uptake consists of a reversible and an irreversible part. The measured water uptake is of the same magnitude as the calculated thermal contraction of air in the mortar when the specimen is cooled. It is tempting to conclude that the
reversible water uptake is due to this contraction. However, the present measurements cannot confirm this idea.

None of the samples showed signs of damage after testing. In that respect, this investigation does not put much light on the mechanisms of frost damage in concrete, but it seems to support the hypothesis put forth by Lindmark, whereas it weakens the hypothesis of Geiker and Thaulow.

References

Acknowledgement
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