FROST/SALT-TESTING OF CONCRETE: EFFECT OF TEST PARAMETERS AND CONCRETE MOISTURE HISTORY

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ABSTRACT

Based on the Borås-method (SS 137244) for frost/salt-resistance a large experimental program has been carried out at five Scandinavian laboratories. An initial Round-Robin test established reasonable reproducibility. It was concluded that concrete inhomogeneity was the main cause of variation in the test results. A systematic study was carried out on the effects of concrete moisture history before testing and on the effects of variation in the following test parameters:

1) temperature cycle characteristics,

2) the salt concentration on the test surface,

3) the salt concentration in the concrete pore water.

The effects of silica fume on scaling has been investigated as well the question of whether high strength concrete needs air entrainment to be frost/salt-resistant.

Key words: concrete, frost resistance, test method

1 INTRODUCTION AND BACKGROUND

Deterioration of concrete due to frost action is not a general problem in Norway. Most of the damage occurs in connection with salts (deicing or from natural sources), but then frequently with quite severe scaling as a result. The situation in the other Nordic countries is similar in that damage is generally associated with salts, but it also appears to be more prevalent than in Norway, according to published national status reports (1). Outdoor concrete not subjected to salts is generally protected by correct air entrainment, unless the exposure results in a very high degree of water saturation at freezing.

The most important practical problem is consequently to ensure frost/salt-resistance. One important aspect is a reproducible and relevant test procedure. In Sweden a saltscaling method (SS 137244, referred to here as the Borås-method) has been developed, which has proven reproducible - at least internally.

The goal of the present project was to investigate the influence of various concrete compositions and curing/moisture-histories on the salt scaling. A prerequisite to this task is of course to have a reliable test method. It was decided to base the work on the Borås-method and to carry out the work in 3, partly overlapping, stages:

1) Round-Robin tests at 5 laboratories in 3 countries

2) Carry out experiments where important test parameters of the Borås-method were varied systematically:
   - The temperature cycle characteristics
   - The salt concentration on the surface during the test
   - The initial saltconcentration in the concrete pore water

3) Vary the moisture history of a given concrete before testing. The main point was to subject the concrete to different drying/resaturation treatments before testing. Low temperature calorimeter measurement have shown such treatment to influence the iceformation pattern drastically, Sellevold and Bager (4). Similar drastic effects on the saltscaling was expected.

22 different concrete mixes were tested in 23 different test series. The basic mixes were with w/c + s - ratios of 0,55 and 0,45, air-entrained with and without silica fume. A number of high strength concretes (> 65 MPa) without air entrainment were also tested.

The five laboratories taking part in the work was:

CBL: Cement- og Betonlaboratoriet, Aalborg Portland, Denmark
SP: Statens Provingsanstalt, Borås, Sweden
FCB: Forskningsinstituttet for Cement og Betong, SINTEF, Trondheim, Norway
NBI: Norges Byggforskningsinstitutt, Oslo, Norway
NOR: Norcem Betonglaboratorium, Brevik, Norway

The Round-Robin test is reported in full (2), as is the rest of the experimental work (3). This article presents a number of representative results from both reports and sums up the main conclusions. It should be noted that the extent of the experimental results is large and because space is limited the report focuses on presenting results, rather than interpreting these in terms of mechanisms.

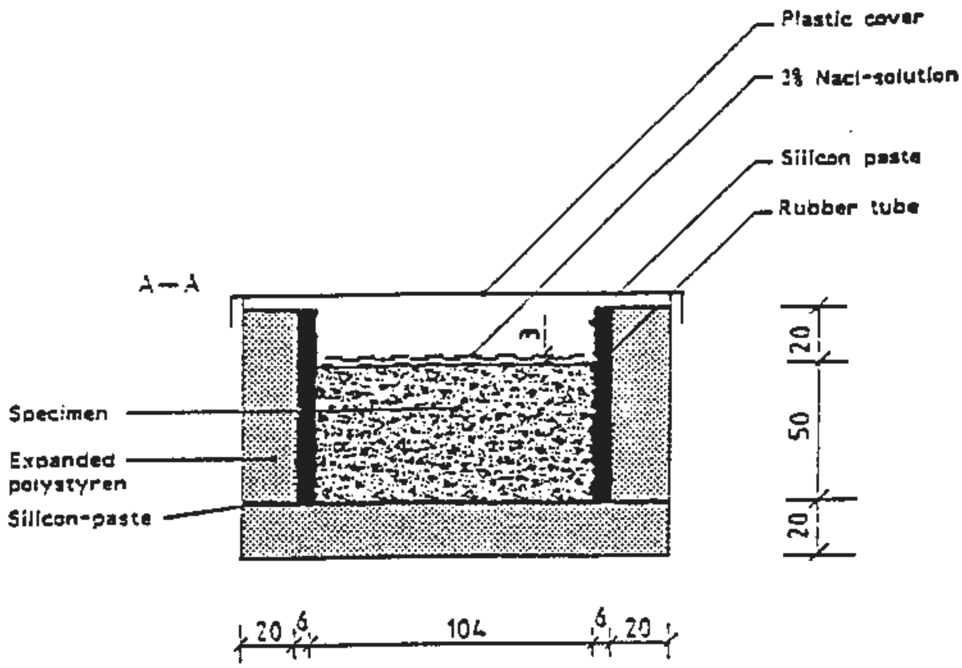
2 THE ROUND-ROBIN TEST

2.1 The Borås-method (SS 137244)

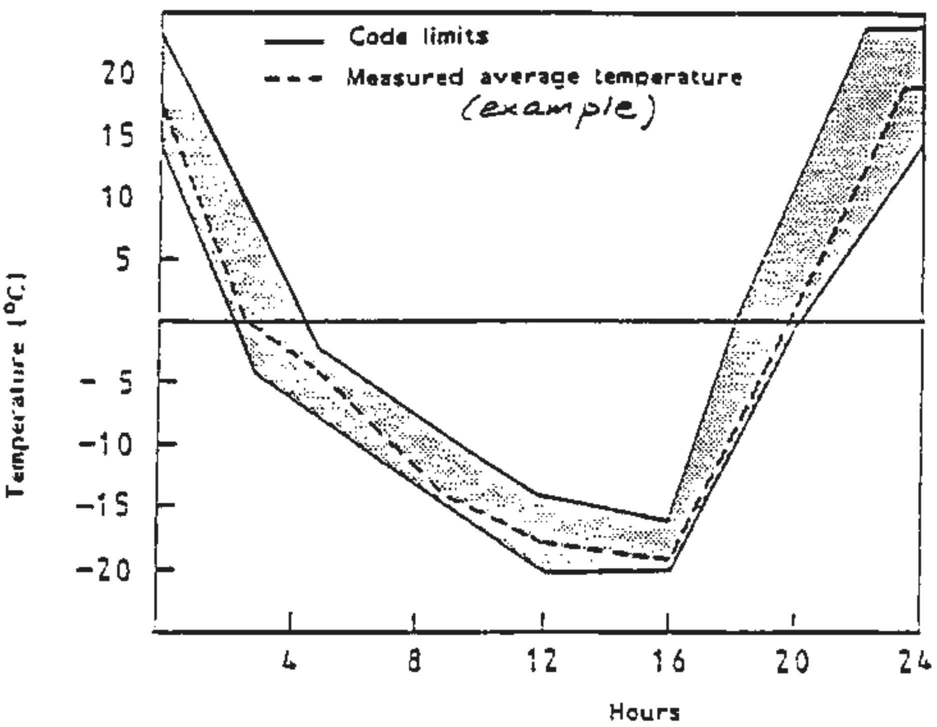
The Borås-method is a saltscaling test method similar to ASTM C 672, but with a number of details improved. The test specimen (about 50 mm thick, cut from a cylinder, cube or prism) is insulated on the side - and bottom surfaces. The test surface is covered with a 3 % NaCl solution 3 mm deep. About 20 mm above the solution a thin plastic foil is stretched to prevent evaporation of water from the solution during testing. The insulation ensures that freezing is one-dimensional and initiates at the test surface. Fig. 1 shows a prepared test specimen and the characteristics of the 24 hours temperature cycle. The temperature is measured in the salt solution.

The amount of scaled material is measured at 7, 14, 28, 42 and 56 cycles, and recorded as dry material per m^2 concrete. At 56 cycles a concrete is rated on the following scale: ACCEPTABLE < 1 kg/m^2, GOOD $< 0,5$ kg/m^2 and VERY GOOD < 0.1 kg/m^2, with the additional requirement that the scaling the last 28 cycles shall be less than the first 28 cycles.

For testing of 150 mm cubes four samples are recommended (900 cm^2),while for samples taken from structures a minimum test surface of 400 cm^2 is recommended.



Specimens prepared for SS 13 72 44 freeze/thaw test



Freeze/thaw cycle according to SS 13 72 44

Fig. 1 The Borås-method: Specimen preparation and temperature cycle

2.2 REPRODUCIBILITY

The Round-Robin test included 9 concretes, all produced at NOR in the form of prisms 150 x 150 x 1000 mm. 50 mm slices were cut from the prisms for testing. 4 laboratories tested at about the same time at a concrete age of minimum 3 months (sealed curing), while one laboratory started 14 months later. Their results were somewhat different and are reported in section 3.1.

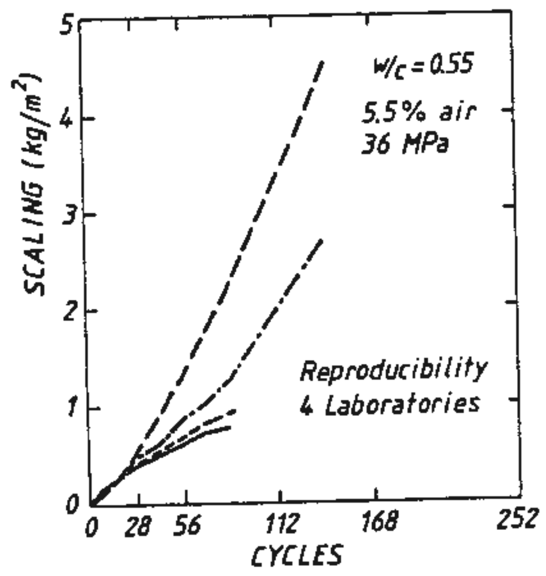


Fig. 2 a Scaling for concrete B1. Mean values including all test specimens

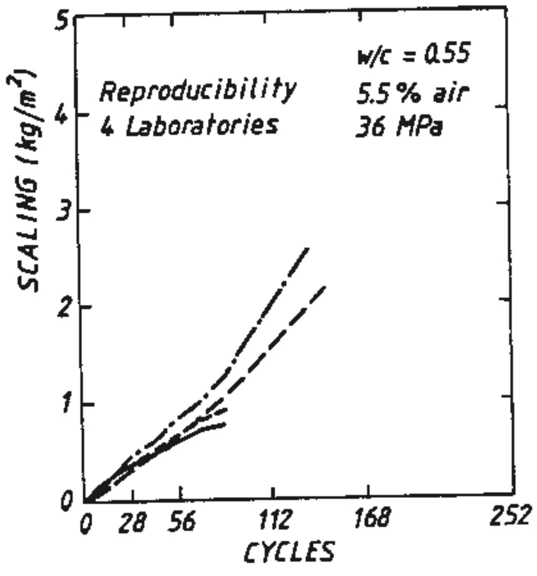


Fig. 2 b Scaling for concrete B1. Mean valves with one specimen excluded at one laboratory

Fig. 2a shows scaling for concrete B1 in the form of mean values for 4 plates at each laboratory; while in Fig. 2b one plate is left out for one laboratory. As can be seen, one deviant specimen changes the concrete rating from ACCEPTABLE to UNACCEPTABLE at one laboratory, while the other 3 rates the concrete ACCEPTABLE. For other concretes similar deviations were observed, particularly for concretes with marginal ratings. For less sensitive concretes, i.e. VERY GOOD rating or with very high scaling, the ranking by the different laboratories was more consistent.

The reasons for the deviations are uncertain. The scaling was generally not a homogeneons phenomenon but occurs in concentrated areas (as is the case in practice). Sometimes the reason for high scaling could be edge effects caused by a pocket in the glued on dam around the edge. Leakage of salt solution would result in no scaling. The specimen preparation clearly depends on the person doing it; however, such problems are noted in the test protecol - which was not the case with the deviants discussed above. High scaling sometimes are associated with weak points in the test surface; a cavity, a loose stone etc, i.e. local inhomogeneities.

The position of a specimen in the freezer influences the temperature cycle characteristics somewhat within the allowed band, Fig. 1. Several times when different scaling was observed for two specimens, their positions in the freezer was exchanged for a period. However, this did not change the scaling pattern for either. We therefore do not believe that small changes in the temperature cycle are responsible for the observed deviations. We believe local inhomogeneity in the concrete test surface is the main cause of deviations, rather than extreme sensitivity to variations in the temperature cycle.

It should be kept in mind that any scaling test only tests a very small volume of concrete in contrast to "volume deterioration" methods such as ASTM C 666. But in practice it is the scaling problem we try to solve - hence a scaling method appears most relevant. SP has shown that it is possible to obtain consistantly low variation for their own laboratory produced concretes. Such a procedure thus yields the <u>potential</u> frost resistance of a concrete composition. For field specimens the homogeneity of the concrete is also characterized in a scaling test. Results should therefore be reported for individual specimens as well as mean values.

3 EFFECTS OF CONCRETE CURING/MOISTURE-HISTORY AND TEST PARAMETERS

In addition to the 9 concretes for the Round-Robin tests (B1-B9), 13 concretes (B10-B20 pluss two, H5 and H7, with the high strength cement P30-4A) were made for additional testing. B13-B20 were produced at readymix plants, and molded in blocks from which cores were drilled and sawn for test specimens. This procedure was followed in the hope of obtaining more homogeneous specimens. However, this turned out not to be the case, particularly for B17-B20 which produced large variation in scaling. Followup investigations showed that the air content and the scaling varied systematically with test specimen location in the blocks. Thus the results presented in this section shows relatively large variations, however, the general pattern of the results are supported by the number of test series and test laboratories. All details and individual results are given in (3).

3.1 Aging effects

As already mentioned one laboratory carried out the Round-Robin tests about 14 months after the other 4 laboratories. All 4 concretes tested displayed more scaling, after the storage in plastic bags which probably involved some drying. It is likely that this increased scaling is caused by the drying/resaturation-effect. Earlier calorimeter - measurement have shown marked increases in iceformation after long term drying at 20°C and RH of 75 and 83 %, before resaturation (4). In normal Borås-procedure drying takes place at 50 % RF for one week before water is placed on the surface 3 days and then replaced by saltsolution before testing. The importance of drying/resaturation will be further discussed below.

3.2 Drying/resaturation treatment

When concrete is dried and resaturated (submerged in water) its pore structure is changed substantially. This results in increased permeability and iceformation, indicating a change in the direction of more coarse and continuous pores (1, 4, 5). This is seldom taken into account in concrete testing. The effects of such pretreatment on scaling was investigated in several test series.

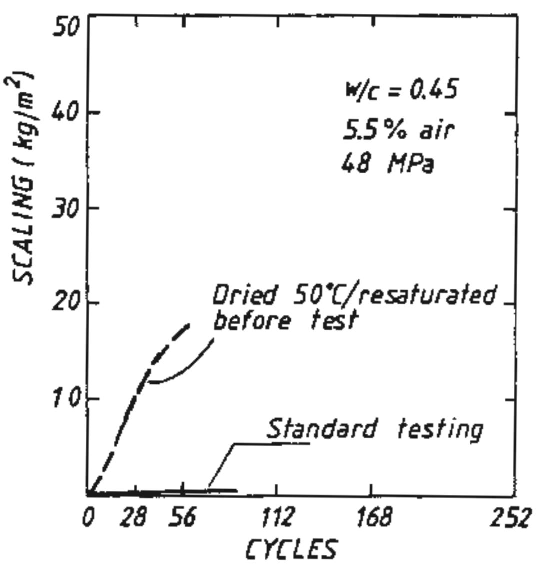


Fig. 3 Scaling with and without drying/resaturation treatment

Fig. 3 shows a typical result. Predrying at 50°C for 10 days followed by one week submersion in water led to at least an order of magnitude increase in scaling. Several other variations in treatment were tried: 2 days at 50°C, 1 day at 105°C and one simulating realistic conditions with 8 hours at 50°C followed by 16 hours at 20°C for a total of 6 days. Individual specimen variation was large and the results as a whole not systematic enough to allow establishment of any critical limit for the treatment. The only general conclusions to be drawn is that any of the pretreatment varieties led to increased scaling, and that concretes containing silica fume (7 % in a 1:1 replacement of cement) were more robust to such pretreatment. 50°C drying is not unrealistic relative to natural exposure of concrete. It is therefore an open question why such treatment increases the scaling in the laboratory so dramatically. For high strength concrete the scaling can be insignificant even after such treatment, see later.

3.3 Curing temperature

The curing temperature was not part of the experimental program. However, the tests showed that concretes B17 and B18 (Fig. 5, for example) had quite high scaling in spite of high strength (ca. 50 MPa) and a good air pore structure L ≈ 0,20 mm). These concretes were produced in blocks (1000 x 1500 x 400 mm) in a very warm period. The most likely explanation is therefore that a high temperature (over 50°C) the first curing period is responsible. Calorimetric measurements of ice formation in hardened cement paste (6) has shown that even the moderate curing temperature of 45°C increases the iceformation and thereby the damage potential substantially. A systematic followup on this point has recently been reported (7) for concrete cured at 20°C, 40°C and 60°C. Scaling was found to increase markedly with increasing curing temperature.

The clear implication of these results is that strength and air pore structure are not sufficent information to assess frost/-salt-resistance of concrete (and probably other durability properties), the curing temperature is also important.

3.4 The Temperature Cycle

There exists little systematic data on the influence of the temperature cycle on frost/salt-scaling. The question is important, at least for two reasons:

a) The temperature band in the Borås-method (Fig. 1) is quite wide. It is unavoidable that specimens are subjected to different temperature cycles depending on their position in the freezer. How much importance does this fact have for the scaling, and to what extent is it responsible for variations between parallell specimens?

b) The relationship between laboratory tests and temperature cycles in natural exposure, i.e. how "tough" is a given test method?

We believe the most important aspects of the temperature cycle to be the cooling rate, the minimum temperature and the time spent at freezing temperatures. Increased cooling rate is normally expected to increase the damage potential due to increased rate of ice formation. With salt, however, it is probably an osmotic type mechanism which dominates, and it is less clear what to expect. Time at freezing temperatures would be expected to be important since an osmotic mechanism involves transport of mass which requires time. The minimum temperature determines the amount of ice formation, and would certainly be expected to be important regardless of damage mechanism.

Several test series were carried out on these points. Different cooling rates and minimum temperatures were achieved partly by changing the specimen insulation (by placing the evaporation preventive foil directly on the saltsolution) in the normal freezer, partly by using a freezer with larger cooling capacity. In two series the specimens were remooved from the freezer on reaching about -18°C in each cycle and stored at +20°C until the next cycle. This cycle is referred to as in/out, and was included in order to minimize the time the specimens spent at low temperature. Fig. 4 shows the different temperature cycles obtained.

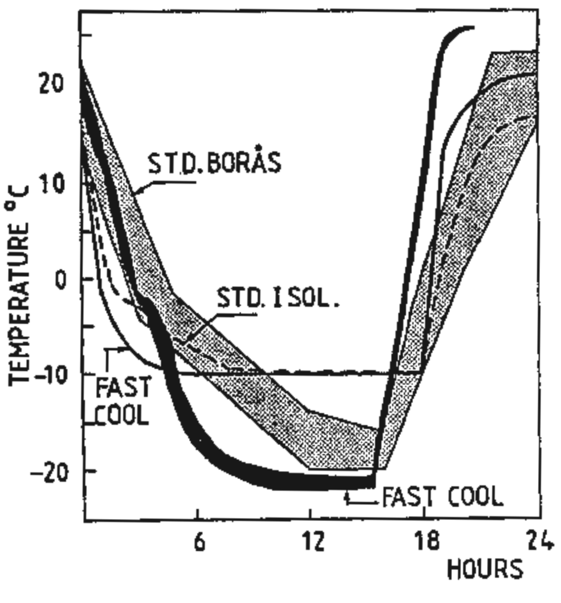


Fig. 4 Achieved temperature cycles in normal freezer (-20°C) and more powerful freezer (-10°C). Fast cool refers to specimens with less insulation (plastic foil in contact with salt solution)

The main conclusion from these test series were (3):

- Increased cooling rate to a fixed minimum temperature does not lead to significant changes in the scaling. In addition: Attempts to detect a systematic relationship between scaling and specimen position in the freezer did not succeed, in spite of a clear relationship between position and temperature cycle. We therefore conclude that temperature cycle variations within the allowed Borås limits (Fig. 1) cannot explain the large variations in scaling often found in routine testing. We believe concrete inhomogeneity is the main source.

- The minimum temperature has significant influence on the scaling. The -20°C used in the Borås-method was found to produce more than twice as much scaling as the -10°C used in a parallell test. This observation is clearly important in evaluating the "toughness" of the Borås-method relative to natural exposure.

- The In/out-variant produced signifiantly less scaling than any of the others regardless of minimum temperature. This observation indicates that pressures generated in the first strong freezing (hydraulic or direct ice-matrix) is not the main cause of scaling. It is clearly neccessary with time to build up pressure, indicating an osmotic type mechanism.

3.5 Salt concentration on the surface

It appears to be generally accepted that 3 % NaCl solution is "pessimal" with regard to scaling. This concentration is used in most test methods, but little recent documentation exists on this point. It is not unreasonable to expect that the "pessimal" concentration varies with concrete quality. Several test series were therefore carried out, on a variety of concrete qualities. Figs. 5 and 6 shows some results. Fig. 5 give the results on two concretes tested at two laboratories. The variation was quite large (caused by the already mentioned inhomogeneity of B17 and B18), but the tendency is consistant; there is a "pessimal" value near 3 % NaCl.

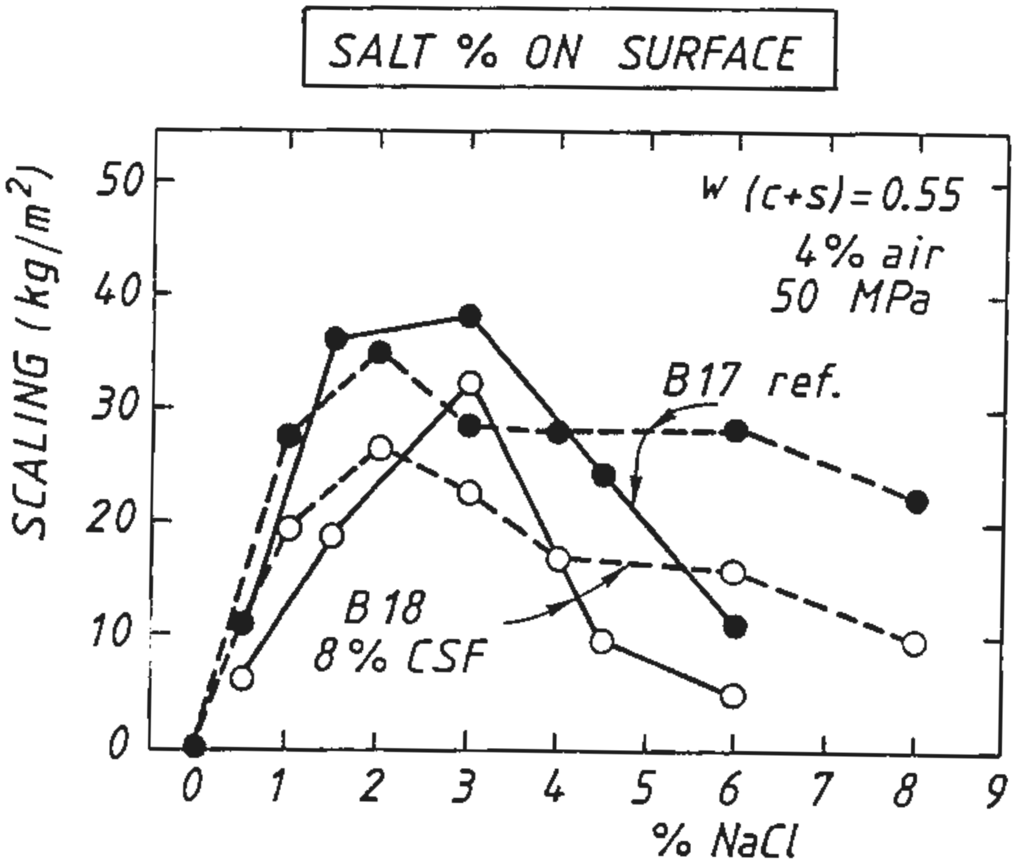


Fig. 5 Scaling with different salt concentrations on the test surface

For high strength concrete (Fig 6) no pessimal value at 3 % is indicated, rather the oposite. The effect of salt concentration is not very large, however, and does not result in altered rating of the concrete. The most striking aspect of the results is the total lack of scaling when pure water is used, and that as little as 0,5 % NaCl has a very significant effect. We lack basic understanding of the mechanism to explain this consistent observation. At least it indicates that a very high degree of saturation in the surface larger alone is not sufficient to explain scaling - since it is difficult to imagine that a small amount of salt would greatly influence this factor.

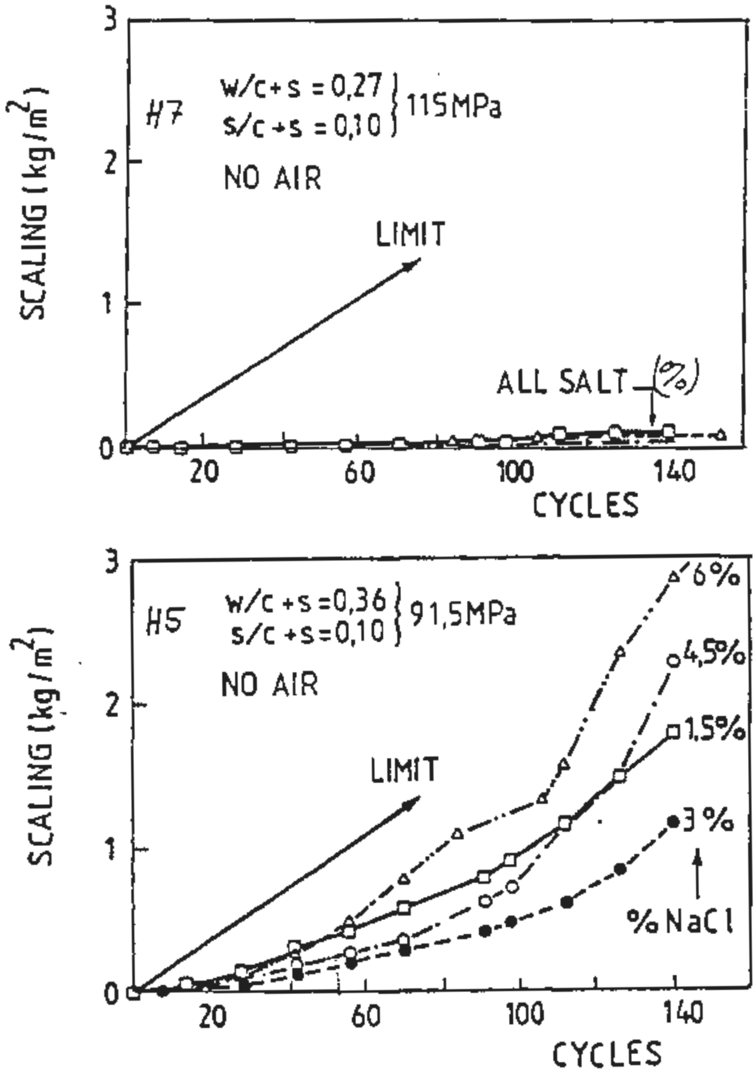


Fig. 6 Scaling of two high strength concretes made with high strength cement P30-4A

3.6 Salt concentration in the pore water

Specimens were dried at 50°C and then submerged in salt solutions with NaCl concentrations from 0 to 12 %. Fig. 7 gives the results for one series. Note that the level of scaling is much lower for B20 with silica fume than B19 without. This was found in practically all series. The concretes were quite inhomogeneous, but the results indicates that resaturation with pure water produces largest scaling. Calorimetric measurements on similarly treated pastes showed, as expected, that pure water produced faster and more iceformation at a given temperature, Fig. 8 (8). Thus our general experience that scaling and iceformation are related in this qualitative way is confirmed by these results. Many conflicting reports exists in the literature on the effects of salt solutions in the pore system on frostresistance. It is a very important point to clearify in order to obtain more basic understanding, and it should be pursued.

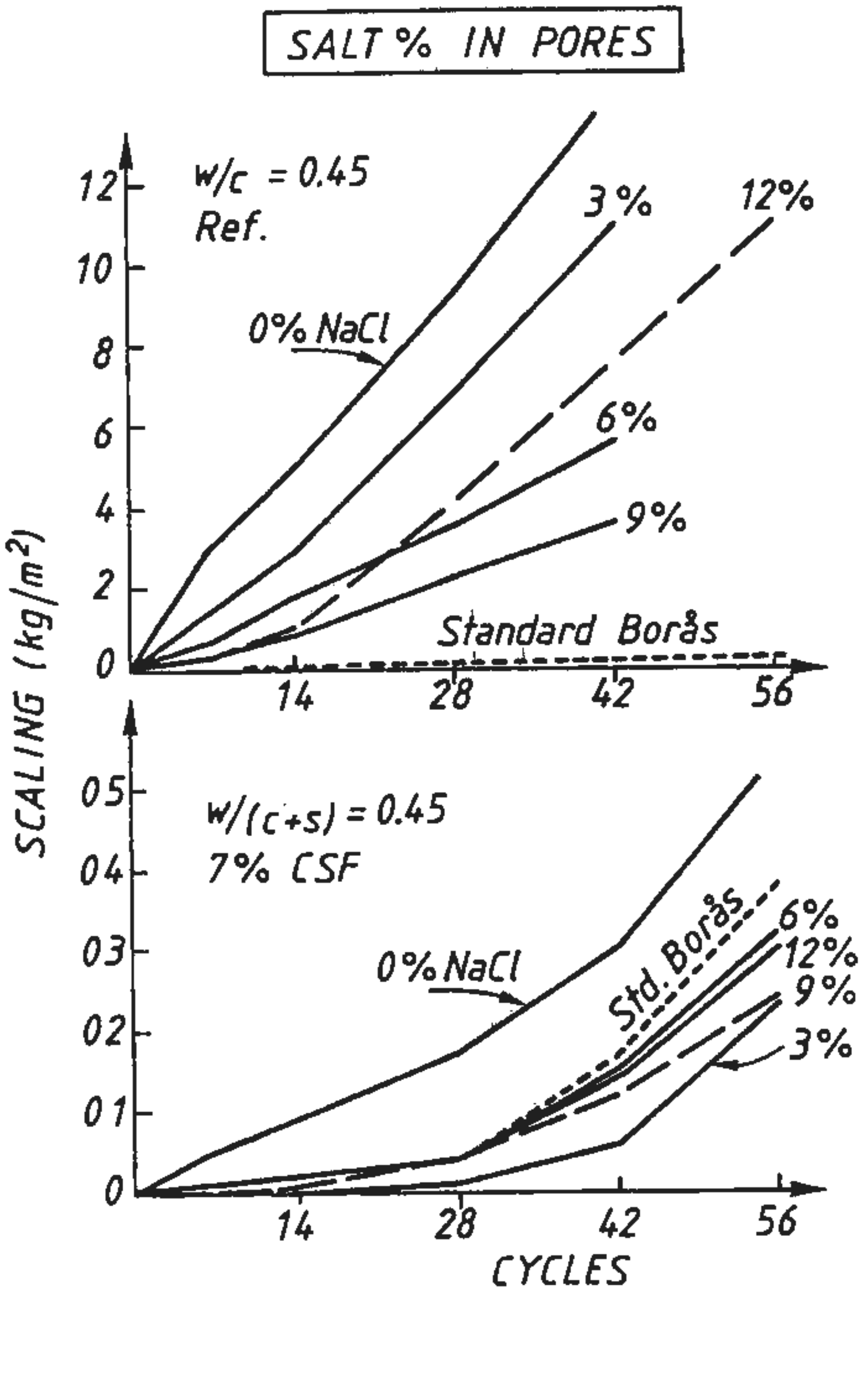


Fig. 7 Scaling for concretes with different salt solutions in the pores. The test surface salt concentration was always 3 % NaCl

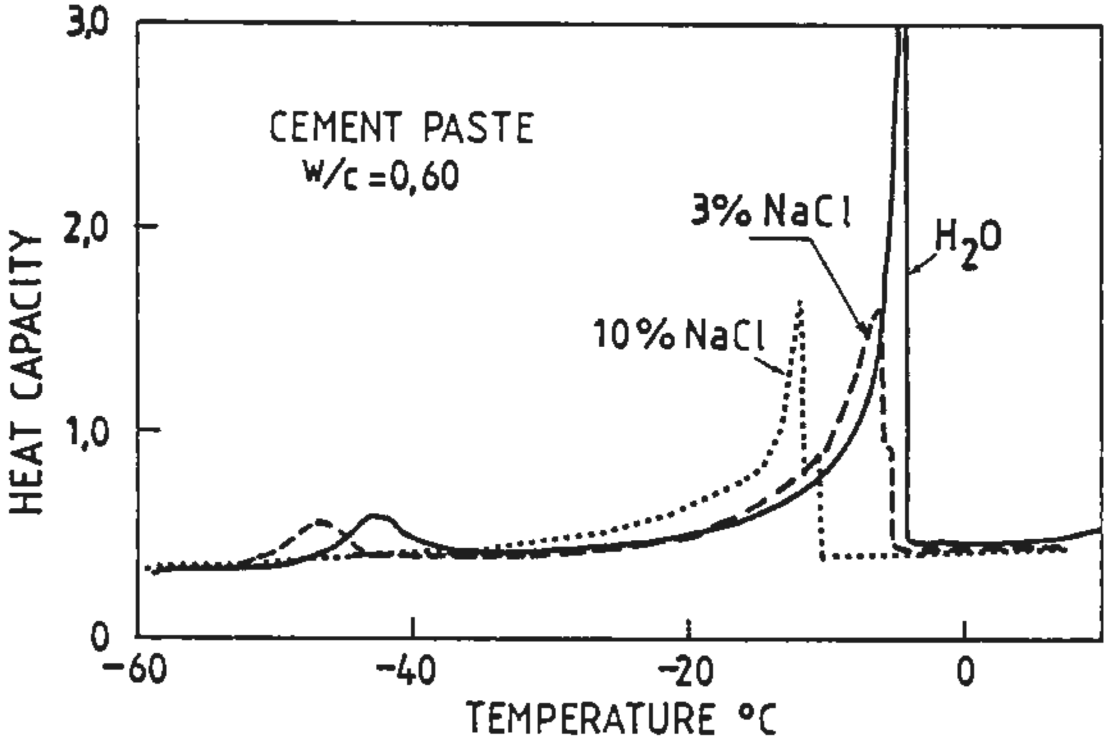


Fig. 8 Freezing calorimetry for pastes dried and resaturated with water or saltsolutions

4. EFFECTS OF CONCRETE COMPOSITION

It has already been pointed out that w/c-ratio, strength and air pore structure are not sufficient to evaluate the potential scaling resistance of concrete. Temperature and moisture histories also play important roles.

4.1 Effect of silica fume at normal strength levels

The effect of silica fume on frost resistance has been a controversial tropic in recent years (5). Several concrete "pairs" with 7 to 10 % of the cement replaced by silica fume on a 1:1 basis were tested in this project, f.ex. B17 - B18 (Fig. 5), B19-B20 (Fig. 7). Both examples shows lower scaling for concrete with silica fume. Fig. 5 shows a small reduction for concretes tested without drying/restauration treatment, while after such treatment the difference is more marked, Fig. 7. Other test series gave similar results (3). We conclude that silica fume improves scaling resistance, and, in particular, that the robustness of concrete to elevated temperature curing and drying/restauration treatment is increased when cement is replaced by silica fume a 1:1 basis.

4.2 High strength concrete with and without air

A relevant question today is whether high strength concrete needs air entrainment to be scaling resistant. 7 high strength concretes (> 65 MPa) were tested in this program, 2 of them with air entrainment. The "pair" B8-B9 w/c+s = 0.35, s/c+s = 0.1 and 0 was not air entrained. The scaling for B8-B9 measured at two laboratories is shown in Fig. 9. B8 with silica fume shows the highest scaling, but the scaling level for both concretes is fairly low, note the ACCEPTABLE limit line. B8 shows some acceleration in scaling beyond the standard 56 cycles, but not a dramatic disintegration as reported earlier for a high strength concrete containing 19 % silica fume (9). A new batch of B8 was produced and tested for 290 cycles after drying/restauration treatment. The scaling was lower than for the original B8 in spite of the pre-treatment, and the Borås-rating was VERY GOOD.

Two air entrained concretes with silica fume B6 and B7 (w/c+s = 0.35, s/c+s = 0.07, 74 MPa - 3.8 % air, 66 MPa - 5.9 % air) were both rated VERY GOOD after standard Borås-testing; B7 also after drying/restauration treatment. This in spite of rather poor values for the spacing factors of 0.40 and 0.26 mm, respectively.

Fig. 6 shows scaling for two high strength concretes with P30-4A offshore cement, both dried/resaturated before testing. The concrete with the lowest strength (H5, 91.5 MPa) shows scaling on the level of B8 with silica fume (Fig. 9) with a certain acceleration beyond 100 cycles. The concrete with highest strength (H7, 115 MPa) shows minimal scaling regardless of saltconcentration up to 140 cycles.

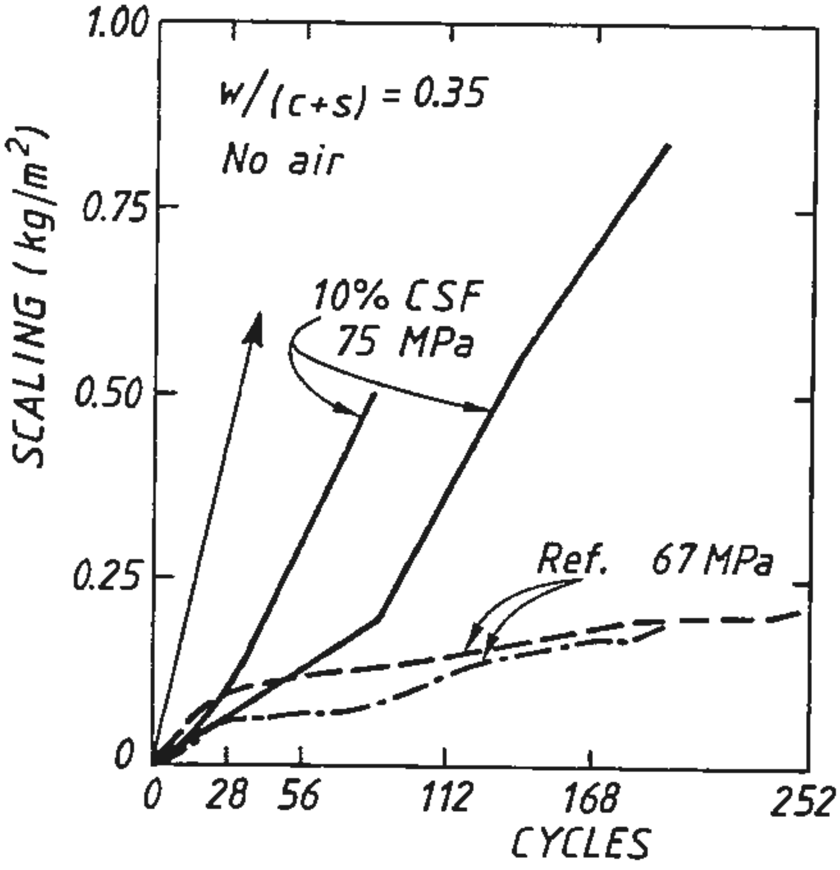


Fig. 9 Scaling of two high strength concretes, each tested at two laboratories

These results as a whole indicate that high strength concrete does not need airentrainment to be salt/frostresistant according to the Borås-method, even including a drying/restaturation treatment and extension of the number of freeze/thaw-cycles well beyond the standard 56. With sufficiently low w/c+s-ratio the rating VERY GOOD may be obtained. The limit appears to be around w/c+s = 0,30, but probably depends both on cement type and silica fume dosage. Silica fume appears to cause some acceleration in the scaling beyond the standard 56 cycles for concrete with w/c+s-ratio of 0,35. High strength concrete is much more robust to drying/resaturation treatment than normal concrete. Recent data indicated robustness of high strength concrete also to elevated temperature curing: concrete with w/c+s = 0,30 and s/c+s = 0.08 showed minimal scaling when cured at 60°C and dried/resaturated before scaling tests (10).

5 FINAL REMARKS

The Borås-method is judged well suited to rate the salt/frost-resistance of concrete. We believe the method gives the potential resistance for a given concrete composition when it is homogeneous and cured under well controlled conditions. For field concrete the homogeneity, as well as the entire temperature/moisture-history plays important roles. The present results demonstrate that scaling can increase dramatically with relatively moderate changes in these factors, particularly the drying/resaturation treatment and elevated temperature curing. Neither are unrealistic relative to natural exposure conditions. The practical use of the Borås-method in the standard version is considered tough but reasonably realistic in that concrete of reasonable w/c-ratio and air pore structures fulfill the criteria. As seen above this is not true after dry/resaturation or curing at 60°C. Similarly, cases exist where cores taken from old concrete with good field performance shows very high scaling when tested in the laboratory. Thus, we can say today that the Borås-method is very useful in rating concrete composition with well controlled temperature/moisture - histories. It appears to be too tough when the concrete pre-test treatment is other than standard laboratory procedure. The present observation that several high strength concretes without air entrainment meet the Borås criteria even after curing at 60°C and drying/resaturation treatment indicates to the author that such concretes will perform well in severe field exposure.

6. ACKNOWLEDGEMENT

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