



On the effect of laboratory conditioning and freeze/thaw exposure on moisture profiles in HPC

Mette R. Geiker^{a,*}, Peter Laugesen^b

^aDepartment of Civil Engineering, Technical University of Denmark, Brovej, Building 118, DK-2800 Kgs. Lyngby, Denmark

^bDansk Beton Teknik A/S, Helleruplund Allé 21, DK-2900 Hellerup, Denmark

Received 2 February 2001; accepted 19 July 2001

Abstract

The effect of selected conditioning (drying and resaturation) and freeze/thaw exposure on the moisture profile in a two-powder concrete (equiv. $w/c = 0.39$, 8% silica fume) has been investigated. For comparison, the effect of conditioning and freeze/thaw testing according to SS 13 72 44 (“Boras method”) on moisture profiles in a three-powder concrete and two plain concretes ($w/c = 0.45$) was measured. The investigations were supplemented by determination of frost resistance and chloride profiles after freeze/thaw exposure, as well as petrographic analysis. The investigations indicate that the present methods of conditioning only have very limited effect on high-performance concretes (HPC). © 2001 Elsevier Science Ltd. All rights reserved.

Keywords: Concrete; Freezing and thawing; Absorption; Conditioning; Moisture profiles

1. Introduction

As part of an evaluation of selected methods of testing the freeze/thaw resistance of high-performance concrete (HPC, $w/c < 0.4$, two- or three-powder blends), the effect of selected conditioning (drying and resaturation) and freeze/thaw exposure on the moisture profile has been investigated [1]. The need for procedures for freeze/thaw testing of HPC became relevant during the 1990s, when lack of air void stability was observed during the execution of some HPC structures and direct performance testing thus required. The effect of selected mix compositions and pumping on the air void stability has, e.g., been dealt with in Ref. [2].

The moisture content of concrete has a significant effect on the durability. Therefore, the initial moisture content should be controlled in accelerated durability tests. It has been shown that drying and resaturation of virgin cement paste increase the freezing taking place [3]. This indicates that to provide a conservative evaluation of the freeze/thaw resistance of concrete, not virgin but

resaturated samples should be tested. This is in agreement with the work of RILEM TC 117-FDC [4].

Only very limited information appears to be available on the transport of moisture in HPC. Compared to ordinary concrete, the low permeability in combination with the small moisture capacity (low dw/dRH , where dw is the change in moisture content and dRH is the change in relative humidity) [5] of HPC is expected to limit the moisture movements taking place.

2. Materials, exposure, and testing

The specimens for measurement of moisture profiles were prepared by cutting prisms ($35 \times 35 \times 70$ mm) from concrete cylinders (diameter 150 mm and height 300 mm) and sealing the prisms on all sides, except one cut 35×35 mm face (Fig. 1). The composition and curing conditions of the concrete cylinders are given in Table 1.

The ability of the sealer to remain unchanged during exposure has been documented (no deterioration, no weight change). However, leakage (diffusion of water vapor), especially at the joint between bottom and sides, may have happened, see profiles for drying according to method D2 (Fig. 6).

* Corresponding author. Tel.: +45-45-25-18-30; fax: +45-45-88-32-82.
E-mail address: mge@byg.dtu.dk (M.R. Geiker).



Fig. 1. Subdivision of prisms for determination of moisture content.

The effect of selected combinations of the exposure conditions given in Table 2 on the properties listed in Table 3 was investigated.

3. Results and discussion

The conditioning according to SS 14 72 44 (D1/S1) had no significant influence on the moisture content of the dense concretes (HUA) (Fig. 2). The concretes with higher w/c ratio (DBT) were, as expected, more affected by the conditioning, but did only absorb less than half of the moisture lost during drying. These observations are in accordance with the measured moisture profiles (Fig. 4).

Prolonged resaturation (D1/S2) caused a slightly increased moisture content (Fig. 3). Vacuum saturation for 2 days resulted in only half of the absorption obtained after 14 days of resaturation (S2). That is, compared to saturation by submersion vacuum saturation does not provide increased saturation of concretes samples of the investigated size and composition.

The depth of the effect of conditioning and testing according to SS 14 72 44 (D1/S1/T1) is illustrated in

Table 1
Composition and curing of concrete cylinders

	HUA-3	HUA-5	DBT-1	DBT-2
Equiv. w/c (w/(c+0.5fa+2sf))	0.39	0.35	0.45	0.45
Low-alkali sulphate-resistant cement (c), kg/m ³	327	320	330	330
Silica fume (sf), % of binder	8	5	0	3.5
Fly ash (fa), % of binder	0	12	0	0
Air, %	5.5	5.5	1–2	1–2
Compressive strength at 28 days, MPa	51	53	NA	NA
Water-cured, months	12	12	1	1
Packed in plastic, months	7.5	7.5	1.5	1.5

Table 2
Exposure conditions

Exposure		Comments
<i>Drying</i>		
D0	none	
D1	20 °C, 65% RH, 21 days	according to SS 13 72 44, "Borås method" [6]
D2	50 °C, 20% RH, 14 days	
<i>Resaturation</i>		
S1	3 days of submersion	according to SS 13 72 44, "Borås method"
S2	14 days of submersion	
<i>Freeze/thaw exposure</i>		
T1	35 cycles in 3% NaCl	according to SS 13 72 44, "Borås method," except number of cycles not 56

Fig. 4. We observe that only the slice of the outer 0–10 mm of the HUA concretes is affected, whereas the depth of effect on the prisms of the DBT concretes is approximately 20 mm. This is in accordance with the chloride profiles measured after freeze/thaw exposure (Fig. 5).

The effect of various combinations of exposure on the moisture profiles in the HUA-3 is shown in Fig. 6.

Table 3
Properties investigated

Property	Method
<i>Moisture content</i>	
Variation during exposure	weight change
Moisture profiles as degree of capillary and vacuum saturation at the end of exposure	samples were obtained by splitting the prisms in approximately 10 mm thick slices (Fig. 1) the degree of saturation was determined by 24-h capillary suction followed by 24 h under vacuum (approximately 20 bars) in boiled and cooled tap water and 24-h drying at 105 °C (~ constant weight)
<i>Chloride content</i>	
Chloride profiles after freeze/thaw exposure	profile grinding and Volhard titration of powder samples
<i>Freeze/thaw durability</i>	
Scaling	according to SS 13 72 44
Expansion	length change (±2 µm) parallel to the exposed surface at two depth (5 mm, 25 mm)
<i>Microstructure</i>	
Petrographic analysis	thin sections placed perpendicular to surface and containing surface

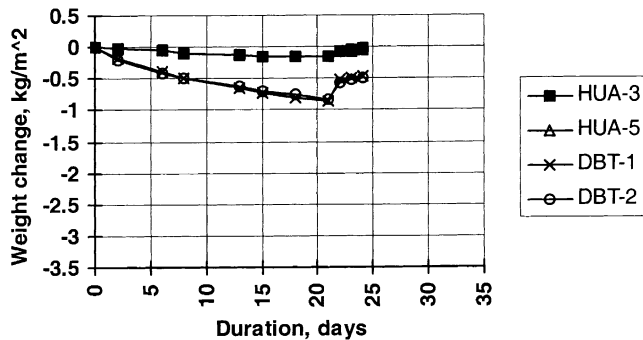


Fig. 2. Weight change of samples conditioned according to D1/S1.

It is observed that the more severe drying (D2) and subsequent resaturation and testing affects the HUA-3 concrete to a depth of approximately 10 mm. The decreased degree of vacuum saturation at larger depths is assumed caused by leakage, especially at the joint between bottom and sides of the prisms. Thus, only changes in the moisture content of the near surface part of the prisms are considered in the study.

The response to 28–35 freeze/thaw cycles according to SS 14 72 44 is given in Table 4. The effect of varying the conditioning (D0, D1/S1, or D2/S1) on the freeze/thaw scaling of the HUA-3 concrete after 28 cycles is observed to be negligible. However, the observed lack of effect of conditioning may be due to a high-frost resistance of the concrete cylinders tested, and the effect of conditioning should be investigated further—both on other concrete compositions and on on-site cast (“real”) concretes—before changing current conditioning procedures. Based on personal experience a non-frost-resistant concrete will exhibit initial freeze/thaw damage after 28 cycles. Varying the concrete composition, both HUA-3 and HUA-5 are insignificantly affected by 35 freeze/thaw cycles, whereas as expected, the higher w/c ratio and non-air-entrained DBT concretes are not frost-resistant. The results are in agreement with observations on fluorescence impregnated thin sections (Fig. 7). Neither microcracking nor increased capillary porosity are seen (coarsening of pore structure is not detectable) in the HUA-3 concrete, whereas marked damage was observed in both DBT concretes.

Only limited data appear to be available on the effect of moisture content on the rate of absorption [7–13]. Based on background data on capillary suction as a function of RH and sorption isotherms from Partner 1 in the project reported in Ref. [11] (OPC, w/c=0.4, moist-cured 7 days, age 28 days), capillary suction as a function of the initial degree of vacuum saturation has been estimated (Fig. 8). Data from Ref. [12] are also given in Fig. 8. Finally, data from the present study are given. These data show the rate of absorption vs. mean degree of saturation in the outer 10 mm of the samples. Data marked with “○” and “□”

are based on only two measuring points (assuming constant rate of absorption). Values of $R^2 > .96$ were obtained for the other data series of 4, 11, and 11 data points. Apparently, the data fit a square root time function even though the initial moisture content was not uniform. As expected, an increased rate of absorption is observed for drier samples (decreased degree of saturation). Also, a lower rate of absorption is found for the older concrete and silica fume containing concrete. However, it should be kept in mind that the difference in duration of capillary suction would affect the rate of absorption similarly.

Applying the correlation between sorptivity (rate of absorption) and initial moisture content proposed in Ref. [8]: $S_i/S_0 = (1 - 1.08\theta_i)^{1/2}$ (S_0 : sorptivity of dry sample, S_i : sorptivity of sample with degree of saturation at θ_i with uniform moisture content), values of S_0 at approximately 0.01 and 0.001 $\text{kg/m}^2 \text{s}^{1/2}$ are obtained based on the data from Ref. [11] and data from the present investigation, respectively (data are shown in Fig. 8, $R^2 = .89$ and $.74$).

Freeze/thaw exposure in combination with a salt solution exposure appears to increase the rate of absorption. An explanation for observations of increased rate of absorption of freeze/thaw exposed concrete in contact with salt solutions compared to concrete in contact with pure water was suggested in Ref. [14]: “Soluble salts will reduce the freezing point of the liquid. The freezing point of the pore liquid is -1 to -2 °C (-3 °C), whereas a 3% sodium chloride solution freezes at -2 °C. During melting the temperature will only increase after all the ice with the actual freezing point has melted. Thawing of water is accompanied by a volume contraction. If pure ice is present on the surface, when the ice in the pores melts, the concrete may maintain its original degree of saturation. However, if the surface is exposed to a liquid salt solution, some of this solution will be absorbed. The determining factor is thus the difference in freezing point between the pore liquid and the liquid of exposure.”

Applying the finite difference model proposed in Refs. [15,16], the mean degree of saturation measured in the outer 10 mm after drying according to method D2 can be

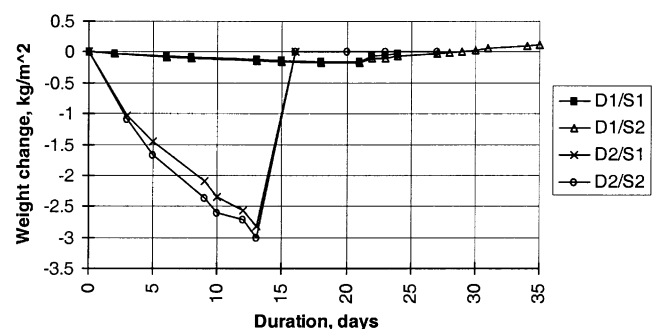


Fig. 3. Effects of selected conditioning on the weight change of samples of HUA-3 concrete.

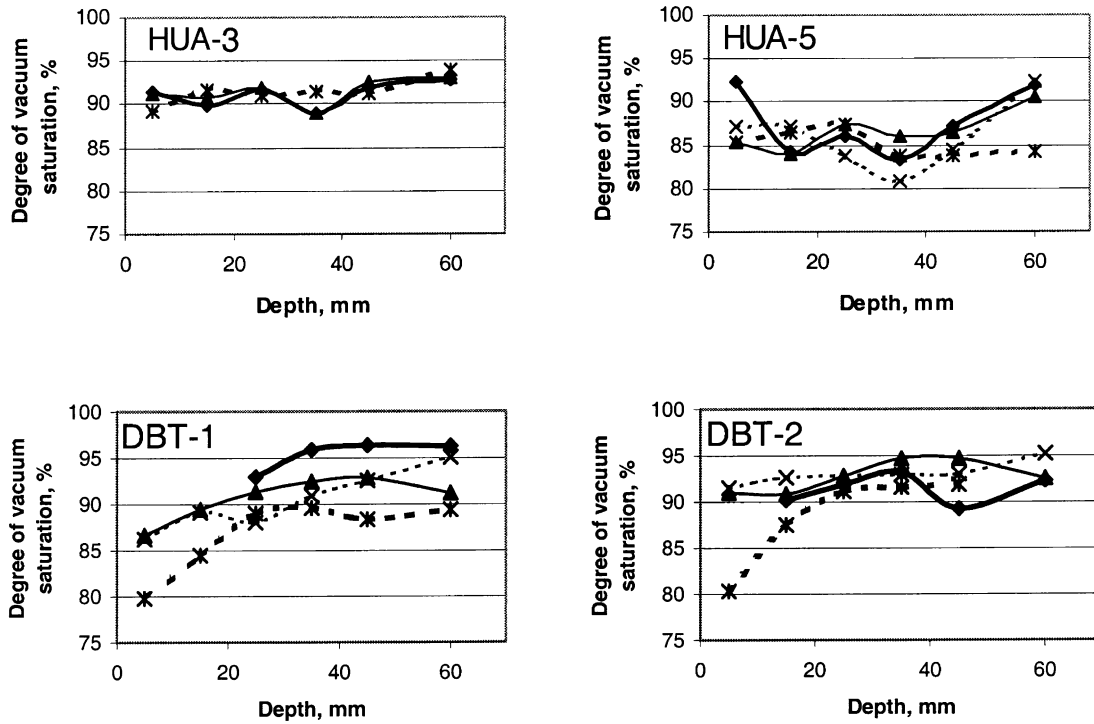


Fig. 4. Effect of conditioning and testing according to SS 14 72 44 (D0: - - -; D1: - - - - D1/S1: — D1 S1/T1: —).

simulated using coefficients of moisture transport (δ) at 5×10^{-8} and 1×10^{-7} m²/s and moisture capacities (dw/dRH) at 10 and 20 kg/m³, respectively. Moisture flow properties (δ) at 2.7×10^{-7} and 1.2×10^{-7} m²/s have been obtained for an almost similar concrete (equiv. w/c=0.38, 400 kg/m³ cement, 5% silica fume) after 0.5 and 3.5 years, respectively (reverted cup method, 65–85% to 100 RH) [13].

4. Conclusions

Only limited effect of conditioning according to SS 14 72 44 (“the Borås method”) is observed on selected HPCs

(equiv. w/c=(w/(c+0.5fa+2sf))=0.35–0.39; 5–8% silica fume and 0–12% fly ash). This is explained by a combined

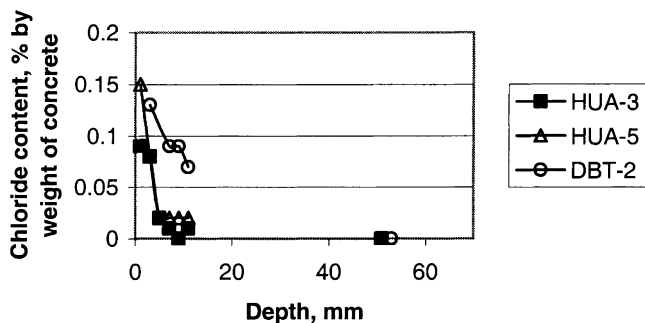


Fig. 5. Chloride content after freeze/thaw exposure (D1/S1/T1).

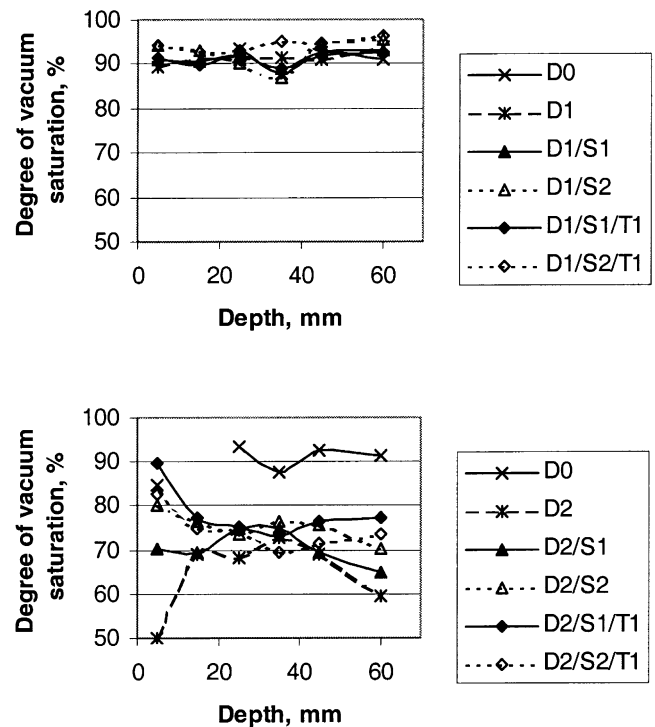


Fig. 6. Moisture profiles (degree of vacuum saturation) in prisms of HUA-3 concrete after exposure to various conditioning and testing conditions.

Table 4
Response to 28 or 35 freeze/thaw cycles according to SS 13 72 44 (two samples of each mix)

Concrete	HUA-3			HUA-5	DBT-1	DBT-2
	D1/S1	D0	D2/S1	D1/S1	D1/S1	D1/S1
<i>Scaling</i>						
28 days, kg/m ²	0.01	0.01	0.00	0.03	2.52	3.31
	0.00	0.01	0.01	0.05	2.37	3.49
35 days, kg/m ²	0.01	NA	NA	0.04	3.64	4.75
	0.00	NA	NA	0.05	2.64	4.72
<i>Expansion</i>						
Top, 35 days, %	0.012	NA	NA	-0.008	0.163	0.470
	0.021	NA	NA	0.051	0.515	0.475
Mid, 35 days, %	0.016	NA	NA	0.015	0.026	0.354
	0.000	NA	NA	0.015	0.516	0.494

low moisture transport coefficient and a small moisture capacity. Moisture transport coefficients (δ) at 0.05×10^{-6} to 0.1×10^{-6} m²/s have been estimated for the concrete with (equiv. w/c=0.39; 8% silica fume).

An increased rate of absorption is observed for drier samples (decreased degree of saturation). Freeze/thaw

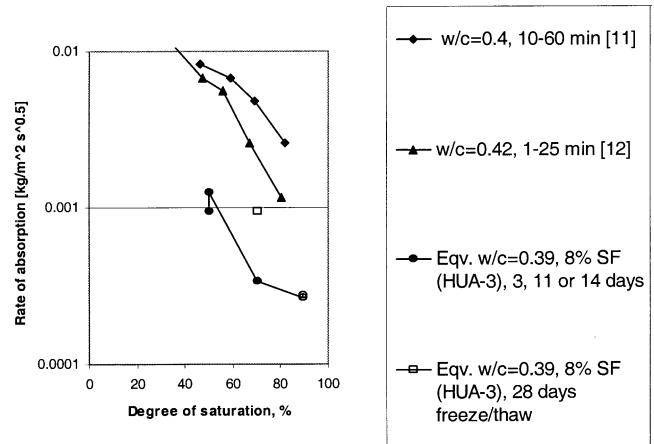


Fig. 8. Effect of initial degree of saturation on the rate of absorption. Data from this study (degree of vacuum saturation) and Refs. [11,12] (estimated degree of vacuum saturation) have been 14 days capillary section, as well as 28 days of freezing and thawing.

exposure in combination with exposure to a salt solution exposure appears to increase the rate of absorption.

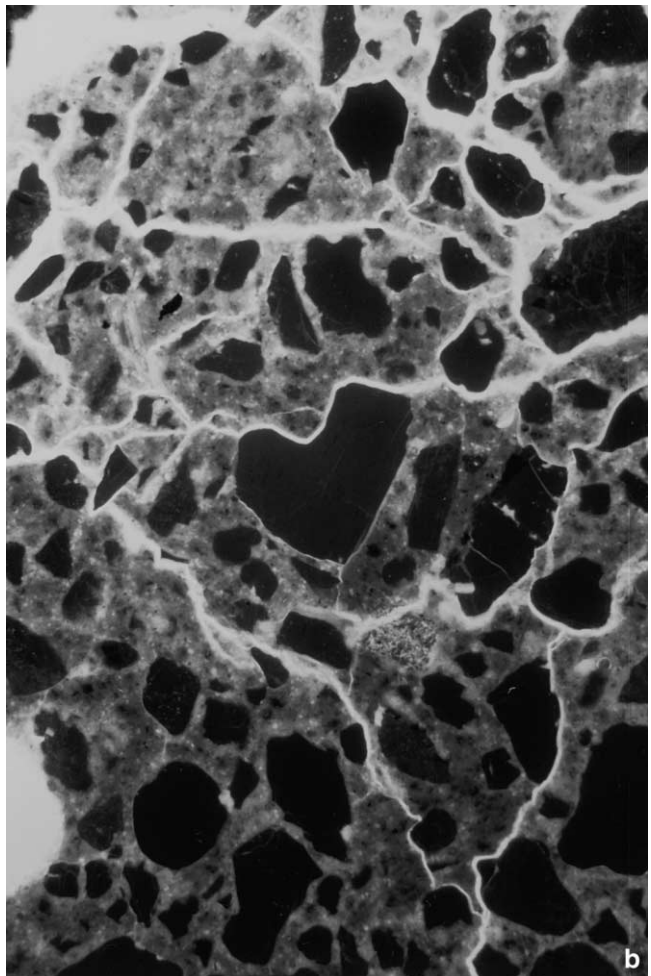
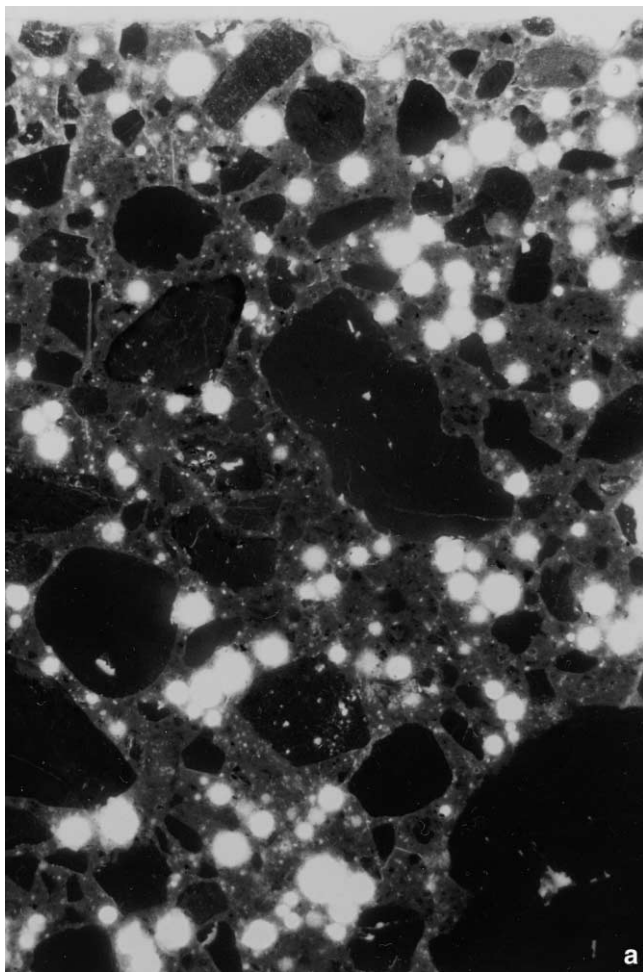


Fig. 7. Close-up of surface fluorescence-impregnated thin sections showing the microstructure of the upper 3 mm of the HUA-3 concrete (left) and upper 2–5 mm of DBT-2 concrete (right) after D1/S1/T1. The uppermost 2 mm of the DBT concrete was fully disintegrated and is not shown.

Acknowledgments

The investigations were made as part of a Research and Development project sponsored by The Danish Agency for Trade and Industry and headed by The Danish Road Directorate. Partners were Dansk Beton Teknik, COWI Consulting Engineers and Planners, G.M. Idorn Consult (RAMBØLL), and Danish Technological Institute.

References

- [1] M. Geiker, P. Laugesen, E.J. Pedersen, N. Thaulow, P. Golterman, J.O. Frederiksen, HETEK, Method for Test of Frost Resistance of High Performance Concrete, Supplementary Research, Report 86, Danish Road Directorate, Copenhagen, Denmark, 1997.
- [2] M. Geiker, S. Rostam, Høj kvalitetsbeton til Udsatte Anlægskonstruktioner. Opgave 7b: Luftindholdsstabilitet (High performance concrete for exposed infrastructure structures, Part 7b: Air void stability), Dansk Betoninstitut, Copenhagen, Denmark, 1995 (in Danish).
- [3] D. Bager, E.J. Sellevold, Ice formation in hardened cement paste: Part II. Drying and resaturation on room temperature cured pastes, *Cem. Concr. Res.* 16 (1986) 835–844.
- [4] RILEM TC 117-FDC, Freeze–thaw and deicing resistance of concrete, *Mater. Struct., Suppl.* (1997) 3–6.
- [5] E. Atlassi, Desorption isotherms of silica fume mortar, 9th Int. Conf. Cem. Chem., New Delhi, 1996.
- [6] SS 13 72 44 Concrete testing—hardened concrete—Scaling at Freezing, Swedish Standards Institution, Stockholm, Sweden, 1995.
- [7] L.J. Parrott, Moisture conditioning and transport properties of test specimens, *Mater. Struct.* 27 (1994) 460–468.
- [8] C. Hall, Water sorptivity of mortars and concretes: A review, *Mag. Concr. Res.* 41 (1989) 51–62.
- [9] S. Millard, Effects of temperature and moisture upon concrete permeability and resistivity measurements, workshop on in situ permeability, Loughborough, 1989, 9 pp., referred to in: J. Kropp, H.K. Hilsdorf (Eds.), RILEM Report 12, E & FN Spon, London, UK, 1995.
- [10] R. Hudd, Effect of moisture content on in situ permeability readings, workshop on in situ permeability, Loughborough, 1989, 6 pp., referred to in: J. Kropp, H.K. Hilsdorf (Eds.), RILEM Report 12, E & FN Spon, London, UK, 1995.
- [11] RILEM TC 116-PCD, Permeability of concrete as a criterion of its durability. Concrete durability—an approach towards performance testing, *Mater. Struct.* 32 (1999) 163–173.
- [12] S.J. DeSouza, R.D. Hooton, J.A. Bickley, A field test for evaluation high performance concrete covercrete quality, *J. Civ. Eng.* 25 (1998) 551–556.
- [13] L.-O. Nilsson, private communication.
- [14] M. Geiker, N. Thaulow, Ingress of moisture due to freeze/thaw exposure, in: S. Lindmark (Ed.), Frost Resistance of Building Materials, Report TVBM-3072, University of Lund, Sweden, 1996, pp. 159–162.
- [15] L.-O. Nilsson, Hygroscopic moisture in concrete—drying, measurements and related material properties, Report TVBM-1003, Lund Institute of Technology, University of Lund, Sweden, 1980.
- [16] L.-O. Nilsson, Fukt och betong, section 14 (Moisture and concrete, section 14), in: C. Ljungkrantz, G. Möller, N. Petersons (Eds.), *Betong handbok, material* (Concrete hand book, materials), Svensk Bygtjänst and Cementa, Solna, Sweden, 1994, 485–524 (in Swedish).