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Preface

This doctoral thesis was submitted to the Norwegian University of Science and Technology (NTNU) in Trondheim for the degree of Philosophiae Doctor (Ph.D.). The research was carried out at the Department of Structural Engineering, Faculty of Engineering Science, NTNU, in Trondheim, Norway.

The Ph.D. project was a part of Work Package 2: "Production and documentation of frost-resistant concrete" within the User-driven Research-based Innovation project "Durable Advanced Concrete Solutions (DaCS). Design and construction for coastal and arctic regions". The work was financed by Norwegian Research Council, Kværner (project leader), Multiconsult, Concrete Structures, Mapei, Skanska, Norbetong, St. Gobain Weber, Norcem, NPRA, NTNU, and SINTEF. The Ph.D. project started in January 2016 and the thesis was submitted in July 2020.

The main supervisor of the Ph.D. project was Professor Dr. Stefan Jacobsen (NTNU, Norway). The co-supervisor was Professor Emeritus Dr. George W. Scherer (Princeton University, USA).

The thesis consists of an extended summary and four appended documents.

The author, Andrei Shpak, declares that this thesis with all its presented work is his own. This thesis contains no material that has previously been submitted for a degree at this or any other university. The author conducted most of the experiments and wrote most of each of the above-listed publications and manuscripts and all other parts of the thesis.

If there are any errors, they are author's and should not tarnish the reputations of the esteemed persons acknowledged in the thesis.

Andrei Shpak
Trondheim, 03.07.2020

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I am very thankful to the project partners of a research project DaCS and Norwegian research council for financing the work and allowing me to take it on.

I would like to express my immense gratitude to my main supervisor, a very good man, Stefan Jacobsen, for his scientific creativity, for giving me enough rope in experimental work, for believing in me and recognizing my achievements, and, not least, taking good care of me and my family.

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I thank my co-supervisor, Professor emeritus George W. Scherer (Princeton University), for donating freeze-warping box, help in assembling and setting up, for fruitful discussions.

I am very grateful to have a very wise group lead, Professor Jan Arve Øverli, who always listened to me mindfully and made me do adjustments in the work plan so I could make it to the end. I mightly appreciate the continuous support and desire to help with the research offered by Professor Mette Geiker. I thank my good friend Dr. Guzel Shamsutdinova who I had a great pleasure to organize an international workshop with. I extend special thanks to Jelena and Elisabeth who I shared not the only the office space with, but victories and losses, life-changing events, who encouraged and supported me all the time. Funny, we all ended up with +1 child during the Ph.D. I would also like to thank the members of the concrete group for all those moments of happiness we shared during my time at university.

This work would not have existed without the major contribution of a good number of master, exchange and summer students I supervised who I had a great pleasure to work with and learned a great deal from, and who did their best to help me in my research. I thank Ole Christian Børsum, MSc for assistance in enhancing the preparation system for salt-frost testing, Margrethe Stensholt, MSc, and, especially, Marte Brun, BSc for processing most of the dilatometry data. Marte also contributed to the processing of AVA and IMA data, which helped to evaluate the methods, and I am mightly thankful for that. I also thank Ole Petter Vimo, MSc, Victor Boyer, BSc, Jørn Hustad, MSc, and Per Øystein Nordtug, Engineer for assistance in salt-frost testing and pilot studies.

The work could not be done without thorough planning and successful production of concrete at SINTEF with Ola Skjølsvold, Knut Lervik, and Erik Johansen, who also performed rapid freeze-thaw tests for me. I would also like to show gratitude to laboratory engineers at NTNU Steinar Seehus, Ove Loraas, Bjørn Strickert Schjølberg, and Terje Petersen for assistance in calibration, setting up equipment, material, and technical support. I extend special thanks to Dr. Alisa Machner and her student-assistants in the period 2018-2019 for thorough planning, stopping hydration, and performing and analyzing TGA.

I am immensely grateful to Erik Sellevold, Professor Emeritus for his comments on an earlier version of the manuscript and to Matthias Müller, Research associate from Bauhaus-Universität Weimar for being a discussion partner even during difficult times of finishing his Ph.D. thesis.

I am very thankful to my friends who I met here in Trondheim, who supported me and my family along the way, looked after kids, made feel at home in Trondheim, and brought a lot of meaning in life. Thank you Alex, Cristina, Massimiliano, Ilaria, Andres, Tobias, Valera, Solveig, Elena, and Francesco.

Finally, special thanks go to my wife, Dasha, and children, Oscar and Aurora, for their love and tremendous patience. Moreover, my wife assisted me a few times with salt scaling and desorption experiments, when I could not get by alone. Thanks also to my parents and relatives who always believed in me.

Abstract

Concrete with moderate replacement levels of fly ash (FA) has been used for decades and considered sustainable in harsh environments. If the replacement levels become high (FA/C > 40…50%), the range of properties from fresh to hardened, including performance in frost testing give often unfavorable results. To reduce the environmental impact of the cement industry, solutions for sustainable frost-resistant concrete with high FA replacements should be developed. Designing such concrete is possible if the exposure is properly characterized, requirements to part materials identified, the guideline for work execution understood and test program agreed upon. Apart from that, the reliable production of frost-resistant concrete with FA should be established and controlled. Eventually, frost-testing results of the concrete should be satisfactory and reproducible for the clients. All these aspects of a "life cycle" of concrete were studied in the present Ph.D. project.

The study was aimed at: (1) reviewing international requirements and recommendations for frost durable concrete from design to execution and testing; (2) developing robust admixture-binder combination for high-volume FA concrete suitable for both onshore and offshore arctic exposure; (3) understand how freeze-thaw performance testing affects high-volume FA concrete. A series of wet freeze-thaw performance tests in presence of freshwater and 3%NaCl were done on high-volume FA concrete. The aim was to investigate the effect of w/b-ratio, air entrainment, extremely low temperature, curing duration, and FA on resistance to the surface and internal damage and understand how surface, internal damage, and liquid transport interrelate for FA-concrete in such tests. Several pilot studies were also carried out to support the main investigations.

Requirements and recommendations for frost durable concrete from standards and specifications in Europe, North-America and Asia, various international organizations and construction projects were reviewed, compared and discussed. This was done based on exposure, material, execution, and tests. Also, some practical examples of the specification together with examples of need of stringency and some occurring peculiarities in testing are given. Finally, the large variation in how frost durability is perceived in different parties of the decision, planning, execution, and commissioning process around the world are discussed and illustrated.

Development of a robust admixture-binder combination resulted in a study on the effect of a sequence of addition for air-entraining (AEA) and super plasticizing (SP) admixtures on air entrainment in high-volume FA concrete (≈45%FA/(FA+C)). The addition of SP before AEA was found to be the most favorable admixture combination for air entrainment in FA concrete, unlike that for OPC (where AEA is added first). Also, Foam Index (common method for evaluation AEA-binder systems) measurements on the same binder materials, admixtures, and dosage sequences were found less useful for studying the effect of admixture combinations. Obtaining a certain air content using the experience with the AEA-SP dosage was found to be an untrivial task if there is a lack of parameter control.

Using the experience of the admixture combination seven concrete mixes were produced: six mixes with 0.52 FA/C and w/b ratios 0.293, 0.40 and 0.45 with and without entrained air, and one OPC mix w/b 0.45, all with 0.06 SF/C. Two of the most used methods, ASTM C666, procedure A for rapid freeze-thaw in water and CEN/TS 12390 for surface scaling in presence of 3 % NaCl solution, were used and extended to investigate how cracking, scaling and saturation progress at standard (-20ºC) and arctic (-52ºC) temperatures in such severe conditions. The results showed that high-volume FA concrete could be produced frost resistant in standardized testing and in arctic exposure when properly air-entrained. Prolonged water curing was found to have a positive effect, except for salt-scaling resistance of air-

entrained FA concrete mixes. Long-term water curing allowed FA concrete with 0.293 w/b without air entrainment to survive a rapid freeze-thaw test in freshwater. Liquid uptake during freeze-thaw was found to be a link, connecting internal and surface frost damage. Air entrainment was found to protect against accelerated liquid uptake during wet freeze/thaw.

The work conducted in this thesis contributes to the understanding of how to treat high-volume FA concrete in production and what to expect of the performance in various freeze-thaw environments.

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Organization of the thesis

The thesis consists of four parts:

I. Extended summary

II. Publications and manuscripts

Report
Shpak A, Jacobsen S: “Requirements and recommendations for frost durable concrete. Test methods. Overview of national and international standards, codes, committees, representative projects”. DaCS project reports, report No.06, 2019, 60 pp.

Paper
Shpak A, Jacobsen S: “Effect of AEA-SP dosage sequence on air entrainment in FA concrete”. Nordic Concrete Research – Publ. No. NCR 61 – ISSUE 2 / 2019 – Article 1, pp. 1-21.

Manuscript 1
Frost testing of HVFA concrete. Part 1. Surface and internal damage, 72 pp.

Manuscript 2
Frost testing of HVFA concrete. Part 2. Strength, hydration and liquid transport, 38 pp.

III. Appendices

IV. Supplementary papers

S-I. Shpak A, Turowski M, Vimo OP, Jacobsen S.: “Effect of AEA-SP dosage sequence on air content and air void structure in fresh and hardened fly ash mortar”. Proceedings of the XXIII Nordic Concrete Research Symposium, Aalborg, Denmark, 2017, pp. 145-148.

S-II. Shpak A, Fossaa KT, Jacobsen S: “Requirements and recommendations to frost durable concrete – an overview”, Concrete in arctic conditions, Proceedings from a Nordic workshop no.16, NTNU, Trondheim, 2019, pp. 35-39.

S-III. Shpak A, Jacobsen S: “Frost testing of HP/HVFA concrete for severe offshore conditions”, Concrete in arctic conditions, Proceedings from a Nordic workshop no.16, NTNU, Trondheim, 2019, pp. 63-66.

S-IV. Shpak A, Fosså KT, Jacobsen S: “Frost testing of HP/HVFA concrete for severe offshore conditions”, Durable Concrete for Infrastructure under Severe Conditions, Proceedings of Lorcenis conference, Ghent, 2019, pp. 187-190.

S-V. Shpak A, Jacobsen S: “Cracking in High Volume Fly Ash Concrete specimens during the European salt-frost slab test: dilatometry measurements and consequence for surface scaling”, Design and construction of sustainable concrete structures: causes, calculation and consequences of cracks, Proceedings from a Nordic workshop no.17, Oslo, 2019, pp. 27-28.

S-VI. Shpak A, Brun M, Fosså KT, Jacobsen S: “Salt frost scaling testing HVFA concrete to -52C: - internal cracking measured with dilatometry”. Submitted to XXIV Nordic Concrete Research Symposium, Sandefjord, Oslo, 2020.

Part I
Extended Summary

1. Introduction

The Ph.D. project is focused on the production and documentation of frost durable concrete with high-volume (HV) of fly ash (FA) as a cement replacement. Introducing HVFA concrete in frost exposure and proving its sustainability will lead to reduced production of cement, responsible for a great share of total CO_2 emission in the world, and increased use of the piling up by-products. Low-heat high-volume FA concrete with "guaranteed frost resistance" can be used for massive structures in the future (pillars and decks for new E39 bridges, construction of dams, offshore concrete structures, etc.) provided that it is well-documented. Being a by-product of coal power plants, FA is presently the most common supplementary cementitious material. Therefore, it was chosen as the available material with the highest potential suitability for large-scale production of frost-resistant concrete. Decades of experience of producing and testing FA concrete and its availability on the world's market played an important role in the choice of the material. The literature studies showed that arriving at HVFA concrete with guaranteed frost resistance is not a trivial task.

At first, one should understand what frost resistance means in general with respect to requirements and recommendations and how to evaluate it in different countries. Then one could focus on concretes with supplementary cementitious materials like FA

Thinking either from the customer's or contractor's perspective the concrete material should satisfy a certain minimum of requirements for a given exposure. To navigate through the pool of standards and recommendations issued by standardization- and engineering organizations a comprehensive review of about 60 regulatory documents from nine countries around the globe that deal with frost-resistance was done [**Report**].

Secondly, to guarantee frost resistance according to some method, the HVFA concrete must be properly designed. That implies the selection of materials and proportions and the production of concrete of stable quality that makes it frost resistant. Controlled air entrainment is a key to stable production, but for HVFA concrete it can be a major issue. Thus far, the production of frost durable FA concrete with a stable and protective air void system has been difficult among practitioners because of the varying quality of FA (carbon content, the inclusion of highly absorbent hollow spheres). In practice, trial mixing is used to obtain predictable air entrainment for the materials and production equipment at hand. Experience with the sequence of addition for air-entraining (AEA) and super plasticizing (SP) chemical admixtures in foam index test with FA slurry showed that SP added before AEA during mixing might be one relatively simple solution to a stable air void system in FA concrete. The sequence of addition for the admixtures has been discussed for a long time, yet no agreement has been reached. In the attempt to solve that we studied the effect of the sequence of addition for the admixtures on air entrainment in FA concrete [**Paper**].

Finally, to understand the limits of HVFA concrete, its behavior in freeze-thaw testing, and how to obtain frost resistance in such concrete mixes, a major experimental program on concretes and corresponding cement pastes was performed [**Manuscripts 1** and **2**]. The main difficulty is that the concrete, at an early age, should sustain severe exposure in testing to be qualified for exposure to frost in the presence of salt or freshwater. With high volumes of FA in the concrete, at an early age, we do not see pozzolanic by-product reacting at all, i.e FA behaves as a filler. Therefore, the amount of water per reacting binder increases, compared to ordinary concrete with Portland cement. The resulting higher effective water/binder ratio then contributes to failing in the freeze-thaw performance testing. We need to understand what decisions contractors and customers should make for concrete so it acquires the ability to withstand the environmental loads. The work on freeze-thaw testing consists of two parts: (1) surface and internal damage, (2) strength, hydration, and liquid transport.

We aimed to provide answers on the following research questions:

- How is frost resistance perceived in the world from the perspective of the standards, regulatory documents, and official guidelines?
- Can we produce frost-resistant high-volume FA concrete?
- How do different freeze-thaw exposures at varying curing ages affect the performance of FA concrete?
- Can FA concrete be frost resistant when non-air-entrained? What is governing (at most) frost resistance for FA-concrete: strength or pore structure?
- How do two of the most used frost performance tests work for HVFA concrete? Under which conditions do internal cracking and surface scaling frost damage relate to or depend on each other? What is the role of transport of liquid and what happens if the temperature is reduced to "arctic" exposure of around -50 °C instead of the usual -20 C°?

The scope of work consist of:

- **Primary studies**
  - analysis of relevant requirements and recommendations for frost durable concrete, including execution and testing;
  - production of frost durable high-volume fly ash concrete: effect of the sequence of addition for air-entraining and water-reducing admixtures during concrete mixing on the quality of macroporosity;
  - development of watertight dam of non-absorbing materials for control of liquid uptake in the European slab salt-scaling test;
  - study the effect of water-to-binder ratio, air entrainment, prolonged water curing, low temperature on resistance to internal cracking and surface damage in salt-frost testing and freeze-thaw testing in freshwater;
  - reciprocity of surface scaling and internal cracking in different freeze-thaw tests;
  - study the effect of degree of reaction on transport properties and frost test performance of FA concrete.

Research is limited to mixes close to the recommended composition in the European standard and mixes with the potential of being frost resistant without air entrainment. However, higher amounts of fly ash as the main supplementary cementitious material were used. Furthermore, all the mixes contain silica fume as a part of standard requirements for frost exposure. As the standard freeze-thaw experiments take a long time, the test program contained selected variations of curing conditions for all the mixes. Along with supplementary studies of various material parameters, a comprehensive experimental program was carried out. During the studies, the work of six master students and two exchange students supervised by the author was incorporated in the context of the present thesis. We also organized an international workshop "Concrete in Arctic Conditions" [1] at NTNU in Trondheim with one full day devoted to discussions and presentations from researchers working with frost durability coming from eight different countries.

2. Methods

2.1. Pilot studies

A number of exploratory studies were launched as input to the development of a suitable research program. This included full-scale work on a ready mix concrete plant as a possible case to study the effect of the dosage sequence of AEA-SP on air entrainment. The first experience with the Air Void Analyzer (AVA) was gained and a new AVA purchased and used in the PhD-work. Also, a pilot study on the effect of water-filled surface air voids on scaling resistance was launched in an effort to study whether such air void filling would lead to special characteristics of the scaling. Furthermore, freeze-warping measurements of inhomogeneously saturated mortar beams were made to investigate whether bi-material bending could help to understand how strain and internal cracking affect salt-scaling in frost testing. Also, sorption studies on cement paste of the same binder as in HVFA concrete mixes used in performance testing were done to assess hydration and freezing point depression in fly ash binders. Studies of shape and size of the collected scaled particles were also carried out and an effort made to compare these to different theories (critical thickness, glue spall) explaining surface scaling. Most of this work was either reported as commercial quality assurance work in full-scale production for clients of the ready mix plant or as project reports or MSc theses of the students. Some parts have been included in the publications of the thesis with the students as co-authors where relevant.

2.2. Review of requirements and recommendations

The review was done to assist the industry with (1) characterization of exposure conditions or “Load” (wetness/saturation/situation, de-icers, frost, etc.), (2) selection of material requirements or “Resistance” (air voids, w/b, binder type, strength, etc.), (3) understanding of existing execution requirements (pumping, casting, finishing, curing, etc.), (4) choice of a suitable test method (air voids, porosity, strength), and (5) examples of requirements from different standards and projects. These five aspects of designing and testing of concrete for the freeze-thaw exposure in nine different countries were tabulated and thoroughly discussed in the report.

2.3. Production – air voids in FA concrete

The work aimed to find the most reliable sequence of addition for AEA and SP in HVFA concrete in terms of air void system and reproducibility. For that, two series of experiments (resulting in two master theses [2,3]) were performed: (1) with two variations of matrix volume, two types of AEA, constant w/b, FA/b, and dosage of AEA, but variable workability and air content; (2) with one AEA, constant matrix volume, w/b, FA/b, and workability, but variable air content. Special mixing procedures were developed with a focus on full activation of AEA during the mixing process. The air void system was characterized by image analysis of polished sections of hardened concrete and the PF method.

2.4. Studies of performance and liquid transport during freeze-thaw testing of HVFA concrete

To follow up on the review of requirements and recommendations and the experiences with the production of air-entrained HVFA concrete, a comprehensive test program was designed to serve the purpose of the study, see Table 2-1. It took over 18 months to perform the tests. All seven concretes had 0.06 SF/C and were produced with slump 200±20mm and air content 4-6% with a spacing factor ranging 0.17 – 0.25 mm, controlled and checked for stability by Air Void Analyser (AVA) in fresh concrete and then verified by Image Analysis on polished sections of hardened concrete [4,5]. The frost testing was done to evaluate resistance to the surface and internal damage during freeze-thaw in the CEN/TS 12390-9 slab test with 3 % NaCl and the ASTM C666 Proc. A rapid freeze-thaw test in freshwater.

Since most of the experiments are based on European salt-scaling slab test CEN/TS 12390-9, within a span of a master project [6] a solution for water- or test-liquid-tight dam under the freeze-thaw testing was developed. It allows obtaining reproducible and accurate values of the scaled mass and liquid uptake per area of the exposed surface. Leakage in such a test has been a major issue in European laboratories, which led to new test methods with the inverted position of the specimen, which we cannot correlate with traditional testing. Also, selected low-temperature “arctic” tests at -52ºC were done on parallel specimens to the standard CEN/TS 12390-9 slab test program.

Internal cracking in the salt-frost test was taken as length change measurements with invar steel dilatometers parallel to the exposed surface with LVDTs at standard (-20ºC) and “arctic” (-52ºC) temperatures. Relative Dynamic Modulus (RDM) was used in ASTM C666. Liquid transport was monitored by mass change measurements during wet curing, pre-saturation after mild drying, and during freeze-thaw. Special care was taken to account for absorption in loosened particles caused by surface damage in the freeze-thaw tests for calculations of liquid uptake in concrete during freeze-thaw. The absorption of concrete cubes during water curing was monitored for more than one year. Filling of air voids during prolonged curing was estimated by comparing non-air entrained and air-entrained specimens with equal paste quality and on the basis of theoretical self-desiccation. Portlandite content and bound water in cement paste of corresponding quality to cement paste in the tested concrete mixes was measured by TGA at the curing (water) age of 14d, 3m, and 1y. Hydration development was characterized by the consumption of calcium hydroxide (CH) and the monitoring of chemically bound (w_n) and hydrate water (w_h).

Table 2-1
The experimental program of HVFA concretes and cement paste in Manuscripts 1 and 2 of Part II

				Concrete									**Cement paste**[2]	
	Material			**Freeze-thaw testing**					**Properties of hardened concrete**				Determination of degree of reaction and pore structure	
				Standard or modified standard testing				Arctic cycle "-50ºC", 28 cycles using CEN12390 preconditioning and cooling rate[3]	Air void structure		Compressive strength	Hardened density, self-desiccation	Portlandite content by TGA	ISO 12571-2013 Desorption[4]
				CEN12390 Standard Freeze-thaw Slab test. "Borås"			ASTM C666 with scaling and liquid uptake, 300 cycles							
Mix code	w/b	FA/b	AEA	Salt scaling 112c. with liquid uptake and UPV	Continuous dilatometry for 56 cycles	Manual meas. of length change[1]			ASTM C457	PF-test				
B	**C**	**D**	**E**	**F**	**G**	**H**	**I**	**J**	**K**	**L**	**M**	**N**	**O**	**P**
0.40-33 A	0,40	0,33	AEA	7d, 3m, 12m		12m	14d, 12m	9m	1y	28d, 3m, 9m	28d, 3m, 12m	1d, 28d, 3m, 12m	14d, 3m, 12m	14d
0.40-33 0	0,40	0,33	0	7d, 3m, 12m		12m	14d, 12m	9m		28d, 3m, 9m	28d, 3m, 12m	1d, 28d, 3m, 12m		
0.45-33 A	0,45	0,33	AEA	7d	7d		14d, 20m[4]		11m	28d	28d, 3m, 20m	1d, 28d, 3m, 12m	14d, 3m, 12m	14d
0.45-33 0	0,45	0,33	0	7d	7d		14d			28d	28d, 3m	1d, 28d, 3m, 12m		
0.293-33 A	0,293	0,33	AEA	7d, 4m	7d	4m	14d	4m	4m	28d	28d, 3m	1d, 28d, 3m	14d, 3m, 12m	14d
0.293-33 0	0,293	0,33	0	7d, 4m	7d	4m	14d, 14m[4]	4m		28d	28d, 3m, 14m	1d, 28d, 3m		
0.45-0 A	0,45	0	AEA	7d		7d	14d, 12m[4]			28d	28d, 12m	1d, 28d, 3m	14d, 3m, 12m	14d
				Duration of water-curing [days or months]										

[1] Manual length change is measured on 2 parallel specimens
[2] Non-air-entrained. Cast using a rotated curing device for homogeneity
[3] Continuous dilatometry for min 28 cycles. One cycle lasts 36 hours, the same cooling and heating velocities as in CEN 12390.
[4] A part of miscellaneous / pilot studies

3. Main conclusions

3.1. Requirements and recommendations

The state-of-the-art on requirements and recommendations for frost durable concrete gives an overview of test methods for frost resistance worldwide. It illustrates the complexity of documenting frost resistance, which requirements to use and test methods to choose worldwide.

Various regulatory requirements and recommendations from Europe, Asia, and North America (a total of nine countries) were summed up in Figure 1 in [**Report**]. Differences between the countries and sometimes within the countries in characterizing exposure, selecting material and execution requirements, and choosing the testing procedures are illustrated. Concerning SCM and particularly FA, the report revealed that little information about the use of FA in concrete is available in the official documents when it comes to design, execution, and testing. It means that special requirements for conditioning and interpretation of the results for such concretes, which may behave fundamentally different from OPC concrete, in such tests barely exist. Lack of studies on the agreement between the methods for evaluating frost resistance and on the connection between performance in the lab and at field contributes to the complexity of the topic. An illustration is given of how frost resistance is defined around the globe. In addition, practical examples of how to select and design frost durable materials for varying types of exposure were given. The study reported in **Manuscript 1** and **2** was launched to fill out knowledge gaps of HVFAC identified in the review.

3.2. Production – air voids in FA concrete

The results showed that the most favorable sequence of addition of AEA and SP during HVFA concrete production is when SP is added before AEA. The polycarboxylate SP seems to shield AEA from being adsorbed and becoming less efficient. At the same time, SP does seem to lose its active ingredient, keeping workability constant at constant dosage independent of the AEA-SP dosage sequence.

This combination provided the best air void system with the lowest air content (least strength losses), meeting stringent requirements to the quality of macroporosity required for frost resistance. Yet, the performance of AEA agents should be considered when performing trial mixing. We found that of those few AEAs studied, a synthetic AEA based on olefin sulfonate showed superior performance compared to natural-synthetic AEA. The particular AEA could allow ignoring differences in the AEA-SP dosage sequence when the needed dosage is found and reproducibility achieved. Finally, the trial mixing of concrete is a much safer way to assess the efficacy of AEA-SP combinations in FA concrete compared to Foam Index testing.

3.3. Surface and internal damage

The results show the importance of distinguishing between surface scaling and internal damage in freeze-thaw testing. There is surprisingly high surface damage in many of the studied specimens in the ASTM C666 test in freshwater even without internal damage. In the salt scaling tests, many specimens with low/acceptable scaling had large internal damage well beyond fracture tensile strain. Prolonged water curing improves resistance to both scaling and internal damage for non- and air-entrained (AE) concretes in the freshwater rapid freeze-thaw test. On the contrary, in the slab test, prolonged curing was found to reduce salt scaling resistance for air-entrained concrete. In one case, it made the cumulative scaling even exceed that of the non-AE companion. We have yet no clear explanation for this effect. Exposure to -52ºC increased the amount of cumulative scaling and, especially, the residual dilation of concrete compared to in standard testing. Dilatometry measurements showed that at such low temperatures internal damage in non-air-entrained HVFA concrete can be initiated in the first cycle. Surprisingly the exposed non-AE FA concretes were expanding gradually, in one case up to 12000 με, showing some sort of ductile behavior. The progression of scaling was quantified by an acceleration factor and analyzed from the features of the scaling rate curves. We found that for all curing ages, surface damage accelerates more for non-AE concrete in the slab test and for AE concrete in ASTM C666. The reduced resistance to salt scaling at prolonged curing of AE concrete also caused an increased acceleration of surface damage. Concrete with internal damage often has an acceleration of the surface damage in both frost tests. A general relation between surface and internal damage can, however, not be seen for all concrete mixes in salt scaling tests. In ASTM C666, however, surface damage is increasing with internal damage. Non-AE HVFA concrete could be made resistant to internal frost damage in both the severe ASTM C666 procedure A and the severe salt-frost scaling slab test with sufficiently low w/b (=0.293) and long curing time in water (14 months).

3.4. Strength, hydration and liquid transport

Measurements of the absorption during curing show that 8-22% percent of the entrained air voids become waterfilled during long term curing of concretes with w/b 0.293, 0.40 and 0.45 with 33%FA/b and 4%SF/b. Hydration studies on the cement paste, strength development curves, and absorption measurements show that the development of reaction in ternary blends with FA is considerably stronger than the development of reaction for an OPC+SF mix. Hydration studies indicate that about 50% of all added FA in the mixes reacted after 1 year of curing. The absorption of water in concrete during wet curing could roughly be predicted by TGA measurements of the cement paste of corresponding quality, showing clear self-desiccation in the FA-reaction. Absorption during pre-saturation after mild drying in the standard procedure for salt scaling test was found to correlate with the first measured scaling after seven freeze-thaw cycles. Observations of lower Liquid Uptake (LU) during freeze-thaw per unit surface in ASTM C666 proc A than in the salt scaling test indicate that the mechanisms of surface damage, and possibly also of transport, are different in these two tests. LU during rapid freeze-thaw correlated with measurements of internal cracking for concrete that did not survive the test. Superficial damage in the same test correlates with LU, especially for well-cured concrete. No general correlation between LU and salt scaling was found in the Borås test. For all concrete mixes and test procedures, air entrainment reduces LU during freeze-thaw. Exposure to Arctic temperatures increases both LU and salt scaling for internally damaged concretes comparing to standard Borås cycle, but it does not affect LU during freeze-thaw for well-hardened HVFA concretes without internal damage. The reduced Liquid Uptake during freeze/thaw (or reduced "pumping effect") by effective air void systems, therefore, appears to be the main protective effect in the investigated HVFA concretes exposed to these 3 different frost test procedures.

4. Miscellaneous pilot studies – selected results

The pilot studies were launched following reviews of practice and literature to map out possible laboratory and field studies. The work was partly done in cooperation with master students and industry. The sorption studies were made on the same cement paste mixes used in [**Manuscript 2**], and reported as a student project report [7].

4.1. Field studies of air entrainment: trials with AVA on RMC plant and QA-work on larger production (30.000 m^3) of air-entrained FA concrete

AVA (Air Void Analyser)-studies in full-scale RMC (Ready Mix Concrete) production. Air entrainment still is the basic tool in the production of frost-resistant concrete and therefore initial work on producing and documenting the air void system in production was done with AVA. The scientific objective was to investigate to what extent the effect of the AEA-SP dosage sequence can be used to control the air void system in full-scale production at Norbetong's new RMC plant in Ilsvika in Trondheim. A field lab was established and the AVA set up on-site but it was found difficult to vary the dosage sequence with the production equipment of the RMC plant. Instead, the study was continued in the lab and resulted in two MSC these [2,3] and [**Paper**]. Quality assurance (QA) work on the stability of air entrainment throughout the thickness of placed concrete was completed with the industrial partner Norbetong AS. Air void analysis of hardened concrete and the PF porosity characterization was done at four different depths from the cast surface. The results are publicly available in [8].

4.2. Studies of the effect of surface saturation of air voids on salt scaling. Shape characterization of scaling mass

The accelerated Liquid Uptake (LU) during freeze-thaw could cause the air-voids to become water-filled at the surface with time. Thereby protection could be lost and instead more damage than in non-AE concrete could happen. A pilot study was performed of salt scaling measurements on specimens that were pressure-saturated before the test commenced.

Scaling measurements showed that one day of the exposed surface under 5atm of water column increased cumulative scaling after 50 cycles for AE concrete from 0.2 to 2.4 kg/m^2, but reduced scaling for non-AE – from about 4 to 1.4 kg/m^2. It was also found that scaled-off material became thicker (over 1mm contra common from 0.05-0.3mm flakes [9] presumably because of some bursting of the air-voids at the surface. The number of specimens was however limited, and there were some variations in concrete qualities. See Appendix P-I for details.

Simplified measurements of scaling mass of the main concrete samples [**Manuscript 1**] showed that the thickness of scaling mass (flakes with one facet larger than 1mm) of AE specimens are within a range of 0.2 - 0.3 mm independent of exposure, curing duration, and the number of cycles. Consequently, in the acceleration phase of AE concretes we simply have more scaling mass of "standard" thickness, i.e. not thicker flakes because of some "burst" air voids. For non-AE concrete thickness ranges 0.5 - 0.9 mm, independent of oscillations of the scaling rate. The thickness of the flakes happens to coincide fairly well with the air-void spacing factor in hardened concrete. The most recent work [9] revealed that the particles of Borås are coarser and their thickness more uniform and less dependent on size than in ASTM. In ASTM thickness is more clearly a function of size. In both ASTM and Borås air-entrained concrete appears to give thicker particles, with some uncertainty though.

4.3. Studies of freeze-warping

Since it is expected that internal cracking can increase surface scaling, it was of interest to study how fluid ingress during freezing affects the stresses and strains near the surface. A freeze-warping box developed by J. Valenza and G. Scherer at Princeton University in 2003-2005 and donated to NTNUs concrete lab in 2014 and was used. Observations were made of the bi-material deflection of a mortar specimen during a freeze-thaw cycle, caused by a thermal mismatch between moist and dry concrete. It took a work of one master student to develop a robust experimental set up that would be in line with

Scherer/Valenza's bi-material bending model and to produce an operator manual for that demanding experiment. All details about experimental set up are given in [10]. Further, we attempted to study the effect of a controlled amount of moisture at the surface on deflection during freezing and thawing (1 cycle +5…-20ºC…+5 in 6 hours). Figure 4-1 shows deflection (after removing drift and deformation of the equipment) of a dry concrete beam (110x20x10mm^3) after dripping 2g of water on the surface of a severely (105 °C) predried specimen and freezing it while it absorbs water.

The figure shows that the concrete sample (presumably with AE) bends immediately up at freezing, possibly due to internal damage. Then it continues warping up even more during the thawing phase, possibly due to permanent cracking and further filling by suction during thawing. Permanent upward warping is seen after one freeze-thaw cycle. The figure shows the capabilities of the experimental setup when conditions (control of room temperature, vibration, timely calibration) permit. The work was stopped at the trial stage due to the high sensitivity of the measuring equipment, a series of breakdowns of the important elements, and time restrictions compared to the other parts of the study.

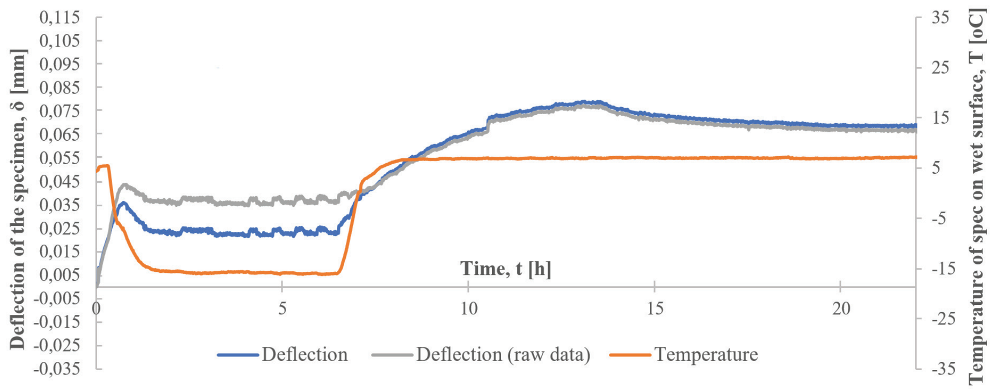

Figure 4-1. *Deflection of room dried concrete specimen after spreading 2g of water on the top surface. Trial*

4.4. Sorption studies

Exposure of concrete to the Arctic temperature cycle (+20…-52ºC) means more freezable pore water in the concrete samples. Desorption [7] experiments on cement paste were intended to assess pore structure and quantify freezable water for the different binder qualities, curing periods, and exposure temperatures used in Manuscripts 1 and 2. We assume that the first desorption resembles the processes in the cement paste of concrete exposed to the first freezing being never (as in ASTM C666 proc. A) or slightly dried (test surface in salt scaling tests is dried for 7 days at 65%RH), as in real concrete structures.

Method. Cement pastes for desorption studies replicated the pastes in concrete mixes tested for frost resistance in this study. The 14 days old paste cylinders were sliced so that five discs from each mix were put in one of the glass desiccators with 11%, 43%, 75%, 94%, 97%, and 100% RH. Three drying methods were used to relate evaporable water content in desorption experiments to (1) drying at 105ºC, (2) drying over a silica gel (about 0% RH), (3) drying by double solvent replacement with subsequent drying over silica gel.

Results. Solvent replacement procedure was thought to be the most efficient way to preserve microstructure and remove only evaporable water ($\boldsymbol{w_e}$). However, Figure 4-2 shows negative values of

w_e at low RH indicating that the part of the solvent remained in the porosity, even after subsequent long-term drying over silica gel (0.45-00-06). The same amount of w_e at 11%RH and 43%RH shows us that w/c-ratio was very similar in both mixes and that amount of hydration products/sorption sites is nearly the same (see RH0), hence FA has not reacted. First strength measurements on concrete also confirm that.

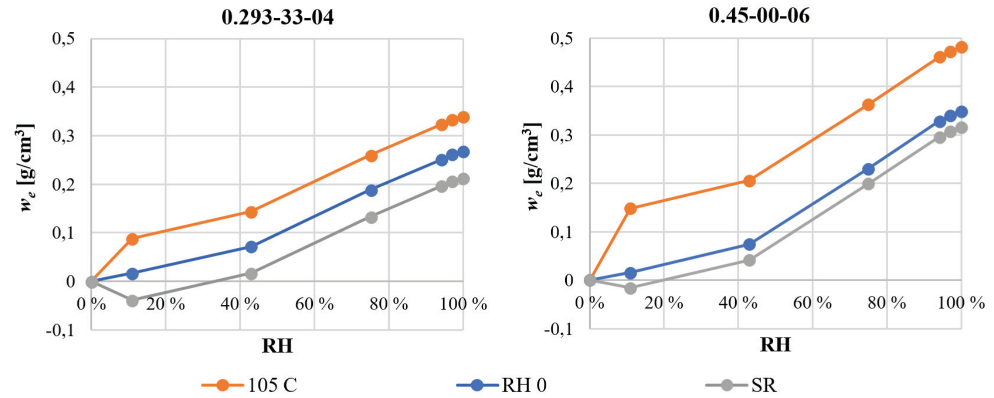

Figure 4-2. *Desorption isotherms for cement paste mixes with w/(c+SF)≈0.45. Effect of drying procedures*

5. Future research

Based on the accomplished work, the following research questions can be further investigated:

- Do water-filled air-voids at the test surface increase superficial damage? Will there still be a "pessimal" concentration of NaCl solution when air voids are water-filled at the surface?
- Influence of additional exposure (50ºC drying-re-saturation) on well-cured FA concrete with respect to F/T-resistance.
- Sustainable marine concrete mixes with high volume fly ash. Including the splash zone.
- AEA-SP admixture combination. Further studies, involving surface chemistry and full-scale testing
- Bi-material modeling using freeze-warping and dilatometry measurements with varying the degree of saturation in concrete. Modeling of degradation in the outermost layer under FT cycles.
- Desorption studies performed during the Ph.D. time to quantify freezable water in the cement paste discs and to relate to liquid transport and presumingly to the types of freeze-thaw damage.
- Arctic cycle (-52 °C) in salt scaling set up after standard Borås curing, considering the negative effect of additional curing on scaling resistance of AE HVFA concrete. In Manuscript 1 freezing dilatometry studies of the internal damage during the very first frost cycle detected much higher internal damage compared to that in "standard" temperature (-20ºC) in non-AE concrete, while the actual air void system effectively protected AE concrete. Can air voids turn negative also for internal damage after a long time cure?
- We could have used more rapid and severe methods to identify frost resistance of concrete (rather than long-lasting CEN/TS 12390-9), which correlate very well in relation to water uptake-surface scaling. Maybe dilatometry during the first cycle like in Manuscript 1 or perhaps Arctic exposure for 5% of NaCl solution, which is used in Russian severe freeze-thaw test (third accelerated test procedure with freezing to -50ºC)? The use of 5% NaCl both on the surface and in the pore system in the Russian standard is in line with Wang *et al*. [11], who found that 5% NaCl combined with low temperature gives the most rapid damage. This is contrary to the European standard, which uses saturation of the pore system with pure water followed by freeze-thaw with 3 % NaCl. This procedure was selected

because experience shows that that concentration gives maximum damage in standard testing with temperature not going below -24ºC [12].

Participation in ongoing and possible future projects:

- New RILEM TC FTC 2018-2023 and FIB TG8 - WP4 Freeze/Thaw deterioration of concrete contributing to a new *fib* Model Code 2020. Joint work on studying multiple degradations by combined actions of accelerated liquid uptake, chloride penetration, cracking. Contribution to the development of practical recommendations for sustainable structures exposed to severe environments.
- Nanocem CP14 joint effort on experiments and modeling. Water filling of air voids - method and effect(s) in frost and/or salt-frost exposure and modeling internal damage (hydraulic pressure, crystallization pressure, cryosuction, etc.) vs surface damage (glue spall effect) including advanced poromechanical modeling.
- Use of FA concrete in development projects for Norwegian Public Road Administration under "E39 project"

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Part II
Publications and manuscripts

Report

Requirements and recommendations for frost durable concrete. Test methods.
Overview of national and international standards, codes, committees, representative projects

Shpak, A., Jacobsen, S.
DaCS project reports, report No.06
Trondheim, 2019

DaCS
Durable advanced Concrete Solutions

Report No. 06

Requirements and recommendations for frost durable concrete. Test methods.

Overview of national and international standards, codes, committees, representative projects.

WP 2. Production and documentation of frost durable concrete: air entrainment, cracking and scaling in performance testing

Author(s)
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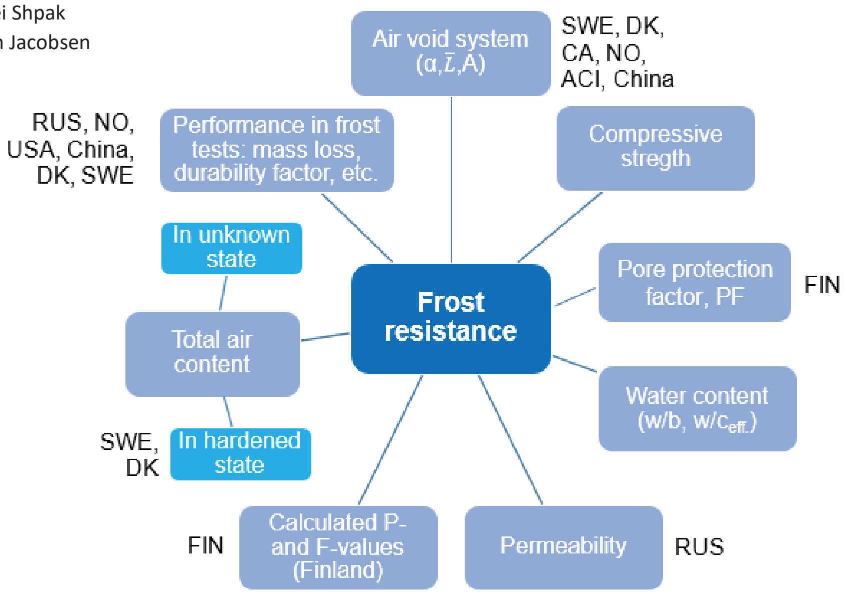

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ABSTRACT

The complexity of the ways the standards are organized in most of the reviewed countries contributes to immense efforts that the design and construction institutions should take on for selecting the requirements for a structure in a particular environment.
Requirements and recommendations to frost durable concrete can be roughly divided into Exposure or Load, Material requirements or Resistance, Execution, Tests and Acceptance Criteria. Each component is discussed in the present report, which is entirely based on more than 60 available to the author regulatory and recommendative documents in Europe, Russia, and North America and partly China as well as specific requirements in selected organizations and projects.

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Preface

This report and the related work have been carried out within the research project "Durable advanced Concrete Solutions" (DaCS). The project started in 2015 and is a 4-years' research program with a focus on concrete structures for severe conditions. The main R&D objective is to enable the production of sustainable and durable concrete structures for coastal and offshore arctic applications, considering both production and service life phases.

Multiple researchers from the Norwegian University of Science and Technology, SINTEF and industry partners, together with 3 PhD-students and a number of MSc-students, work on four focus areas:

- WP 1: Early age cracking and crack calculation in design
- WP 2: Production and documentation of frost-resistant concrete
- WP 3: Concrete ice abrasion
- WP 4: Ductile, durable Lightweight Aggregate Concrete

The industry partners are leading multinational companies in the cement and building industry, together with Norwegian engineering companies and offshore industry. Together our aim is to improve the concrete material quality to produce environmentally friendly and durable concrete structures for future arctic offshore and coastal applications. Combining the existing knowledge and experience across industries with the recognized research capabilities of NTNU and SINTEF provides a good basis for both high quality and industry relevant research. Achieving the overall research objectives will strengthen the Norwegian industry's relevance, attractiveness, and competitiveness.

The DaCS project partners are: Kværner AS (project owner), Axion AS (representing Stalite), AF Gruppen Norge AS, Concrete Structures AS, Mapei AS, Multiconsult AS, NorBetong AS, Norcem AS, NPRA (Statens Vegvesen), Norges Teknisk-Naturvitenskapelige Universitet (NTNU), SINTEF Byggforsk, Skanska Norge AS, Unicon AS and Veidekke Entreprenør AS. The project has received a financial contribution from the Norwegian Research Council.
For more information, see https://www.sintef.no/projectweb/dacs/.

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I am also very thankful to my colleagues at Institute of Structural Engineering (Institutt for konstruksjonsteknikk) at NTNU for helping me out and spending hours with translation of the documents in Chineese, Finnish, German, namely Senbo Xiao, Sakari Tapani Pallaspuro, Alisa Machner and Elisabeth Leite Skare.

The present report would not exist without help and technical support of Peng Zhang from Qingdao University of Technology in China, Kjell Tore Fosså from Kværner AS and UniS in Norway, Frank Spörel from Bundesanstalt für Wasserbau/BAW in Germany, Matthias Müller from Bauhaus-Universität Weimar in Germany, Ole Mejlhede Jensen from DTU in Danmark, Martin Strand from Lund University in Sweden, Miguel Ferreira from VTT in Finland and Mette Rica Geiker from NTNU in Norway.

Summary

The report gives an overview of requirements and recommendations to frost durable concrete in standards and specifications, available to the author from a limited selection of standardization and engineering organizations in Europe, Russia and North America, and partly China.

In order to compare requirements and recommendations from different documents we have decided to divide them into Load, Resistance, Execution, Tests and Acceptance Criteria. Each criteria is organized in detailed tables for the best possible comparison. The results provide an overview for how the frost durability is perceived in standards and regulatory documents in different parts of the world. The results show a great variability in the definition of freeze-thaw resistance Most of the countries limit requirements to the mass-ratio, binder type and content, and the total air content in fresh or hardened concrete. Some set requirements to the air-void system and air content in hardened concrete, while others use requirements based on freeze-thaw tests optionally combined with permeability-related tests and remaining compressive strength.

Requirements and recommendations from different international projects show that the industry normally uses the most stringent freeze-thaw test methods (scaling with 3% salt solution, internal cracking with rapid freeze-thaw cycles in water) to meet Clients' requirements. Yet it is not clear how these types of test methods correlate with real exposure in actual structures. The differences could lie in mismatch between, for example, age of concrete before the test and age of the structure, exposure to salt and water during freeze-thaw cycles of specimen in the test and of real structure, test surface (sawn or formed) and finished surface for actual covering of a roadway, or sidewalk, or formed surface or similar.

There is a valuable exposure station project in Sweden, which has provided with a relatively well-correlated data comparison between the European salt-scaling test and frost damage after exposure of samples along salted motorway[1]. However, based on Canadian experience (Thomas[2]), the results from North-American salt-frost scaling test and performance of the same concretes at field did not correlate, especially for concrete with high volume of fly ash. In addition to the abovementioned differences between the test and exposed structure, scaling in salt-frost testing has shown to be sensitive to curing, pre-conditioning and ageing before testing. Compared to OPC concrete, carbonation can cause increased scaling for concrete with high content of slag, whilst concrete with fly ash requires longer curing period in order to perform as good as OPC concrete.

All in all, most standards are not updated with recent research results, do not account for the steady development of concrete materials (especially towards SCM) and technologies, machinery, construction, and testing techniques.

[1] Helsing E., Utgenannt P. The salt-frost resistance of concrete with supplementary cementitious materials (SCM), Nordic Concrete Research. Proceedings of the XXIII Nordic Concrete Research Symposium, 2017

[2] Thomas, M. Optimizing the use of fly ash in concrete. (2007)

Sammendrag

Rapporten gir en oversikt over krav og anbefalinger til frostbestandig betong i standarder og spesifikasjoner tilgjengelig for forfatteren fra et begrenset utvalg av standardiserings- og ingeniørorganisasjoner i Europa, Russland og Nord-Amerika og delvis Kina.

For å kunne sammenligne krav og anbefalinger fra ulike dokumenter har vi valgt å dele inn krav og anbefalinger til frostbestandig betong i last-, motstands-, utførelses-, test- og godkjennelseskriterier. Hvert av kriteriene er organisert i detaljerte tabeller for enklest mulig sammenligning. Resultatene gir dermed en oversikt over hvordan frostbestandighet oppfattes i standarder og kravdokumenter i ulike deler av verden. Resultatene viser stor variasjon i definisjonen av fryse-tine-motstand. De fleste land begrenser kravene til krav til masseforhold, bindemiddeltype og –innhold og totalt luftinnhold i fersk eller herdet betong. Noen stiller krav til luftporesystemet og luftinnholdet i herdet betong, mens andre bruker krav basert på fryse-tine-tester og eventuelt permeabilitets-relaterte tester og gjenværende trykkfasthet.

Krav og anbefalinger fra ulike prosjekter internasjonalt viser at industrien normalt bruker de strengeste fryse-tine-testmetodene (avskalling med 3 % saltløsning, oppsprekking ved raske fryse-tine-sykler i vann) for å møte kundenes krav. Hvordan denne typen testmetoder relaterer til virkelig eksponering i de aktuelle konstruksjonene er imidlertid ofte uklart. Forskjellene gjelder f.eks. aldring før fryse-tine-forsøket begynner sammenlignet med aldring i konstruksjonen før frosteksponering, tilgang til salt og vann i testprøven i forhold til konstruksjonen under frysing og tining, prøveflater i testprøven (sag- eller formflate) kontra bearbeidet overflate i veidekke, fortau, forskalingsflate i konstruksjon og lignende.

I Sverige finnes et verdifullt felteksponeringsprosjekt som har gitt en mengde data med relativ god korrelasjon mellom den europeiske salt-frost-avskallingstesten og frostskader etter eksponering av prøver langs en saltet motorvei[1]. Basert på kanadiske erfaringer (Thomas[2]) korrelerer imidlertid ikke den nord-amerikanske salt-frost-avskallingstesten med feltytelsene til de samme betongene, spesielt for betong med et høyt volum av flyveaske. I tillegg til forskjellene mellom testprøven og eksponert konstruksjon nevnt ovenfor, har avskalling i salt-frost-prøving vist seg å være følsom for herding, forbehandling og aldring før testing. Sammenlignet med OPC-betong kan karbonatisering gi økt avskalling på betong med høyt slagginnhold, mens betong med flyveaske trenger lengre herdetid for å klare seg like bra som OPC-betong.

Alt i alt, de fleste standarder er ikke oppdatert med nylige forskningsresultater, tar ikke hensyn til kontinuerlig utvikling av betongens delmaterialer (spesielt mot SCM) og teknologi, maskineri, konstruksjon, og testteknikker.

[1] Helsing E., Utgenannt P. The salt-frost resistance of concrete with supplementary cementitious materials (SCM), Nordic Concrete Research. Proceedings of the XXIII Nordic Concrete Research Symposium, 2017
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Table of contents

APPENDICES

List of abbreviations (Standards)

ACI 555	American Concrete Institute. Code or Standard Specification
ACI 555**R**	American Concrete Institute. Guide
ASTM	American Society for Testing and Materials. Standard
AASHTO LRFD	The American Association of State Highway and Transportation Officials. Specification, based on Load and Resistance Factor Design philosophy
CSA	Canadian Standards Association
BNQ	Bureau de normalisation du Québec / Bureau of standardisation in Québec, Canada
MTO	Ministry of Transportation, Ontario, Canada. Laboratory testing manual
NS-EN 206	Norwegian national annex to EN 206
SS EN 206	Swedish national annex to EN 206
SIS-CEN/TR	Swedish technical report
DS	Danish standard
DIN	German Institute for Standardization, National standards
GOST	Russian regulatory requirements (for all sorts of products, services)
SP (new SNiP)	Russian building rules (and regulations, for SNiP – is being superseded)

List of abbreviations (Concrete, parameters, and constituents)

SCM	Supplementary cementitious materials (silica fume, fly ash, blast furnace slag)
SF	Silica fume
FA	Fly ash
BFS	Blast furnace slag
AEA	Air-entraining admixture
SP	Water-reducing or super-plasticizing admixture
w/c	Water-to-cement ratio
w/b	Water-to-binder ratio
T.A.C	Total air-void content (Total air content), %
D_{max}	The highest diameter of the aggregate particle, mm
Rel. Dyn.E-Modul	Relative dynamic modulus of elasticity

1 Introduction

This DaCS project report represents an overview of requirements and recommendations for frost durable concrete, embracing the data from over 60 standards, regulatory and recommendatory documents (see Table 2). The collected data is included in the author's Ph.D. project "Production and documentation for frost durable concrete". Over the years many workers have reviewed the topic concrete frost durability from various perspectives; for Rilem committees, for textbooks, as part of PhDs, to understand or develop models to explain frost damage such as the critical degree of saturation and the glue-spall theory etc. We have in this document limited the review to standards and recommendations issued by standardization- and engineering organizations for production of frost durable concrete.

It is important to distinguish, that in that overview the documents under the category "Requirements" mean national (f.ex. NS-EN, SS, DIN), regional (CEN) and international standards (ISO, ASTM), whilst specifications, codes, technical standards, or any other documents, developed by technical committees (ACI, AASHTO, RILEM, HETEK, SVV, ZTV), will further be called for "Recommendations".

Note: The word "standard" (mentioned 120 times in this document) is understood differently as it can mean either a compulsory (norm or "law") or a recommendative document. For instance, ACI means that they issue standards, codes, specifications, all in one, but the organization has zero liability for the consequences their "standards" could lead to. Interesting to note that ASTM international standards often refer to requirements given in ACI documents.

The words "requirement" (repeated 110 times in the document) and "specification" (used 30 times) can also be misleading as it is clearly seen in the following example from the Wikipedia: Specification or Requirement specification is a technical standard, developed by a technical committee in a private organization for a product, a structure, or a particular work. So, depending on the perspective one interpret the document from, a specification is a requirement and a recommendation at the same time.

The scope of this work is to give an overview over the documents (available to author) relevant for frost exposure requirements and recommendations with a slight focus on the introduction of supplementary cementitious materials (Fly Ash or FA in particular) in concrete, pointing to basic similarities and differences, ending with some examples of requirements to frost durable concrete structures.

Frost durability for concrete is its ability to withstand repetitive freeze-thaw cycles throughout a defined life of the structural element without damage due to surface scaling or internal cracking. The concrete is usually exposed to periodical wetting and drying, deicer salts and other different aggressive matters along with the freeze-thaw.

It is the country standards, norms and regulations for concrete that stipulate:

- the exposure (wetness/saturation, chlorides, frost etc.) to base the design on,
- the material requirements (air void requirements, w/b-ratio, binder composition, strength etc.) to select for that exposure,
- the production techniques and rules (placing arrangements, finishing, curing) to apply for making the concrete frost resistant and
- the test methods (air voids, frost tests, porosity, strength etc.) for the final product to confirm compliance.

The approach can vary significantly from country to country. Numerous committees and unions with their sets of recommendations in addition to national standards can make it difficult even within the country to agree on what the frost durable concrete is and how to produce it.

Selection of the reviewed documents in this course report is limited to available to author standards and recommendations from the USA (ACI, AASHTO, ASTM), Canada (CSA, BNQ), Norway (NS-EN), Sweden (SS, SIS), Danmark (DS), Germany (DIN, ZTV, BAW), Russia (SP, GOST, SNiP) and China (GB/T).

The present overview did not include among other documents a review of the concept of equivalent durability of concrete CEN/TR 16563. These technical recommendations were adopted by a number of European countries. It targets connecting field experience with laboratory results, developing a database of reference mixes and exposures. At present, the concept cannot be used in most of the European countries under the directive of EN 206 [20], as it requires some established national system for reference concretes.

Table 1. List of the main tables in the document

	Table 2	Overview of the documents included in the review
Load	Table 3a	Classification for freeze-thaw exposure conditions. LOAD
	Table 3b	Summary of exposure classes from the reviewed standards and specifications
Resistance	Table 4	Material requirements. RESISTANCE
Execution	Table 5	Production and execution of concrete works. Requirements and recommendations
Tests	Table 6a	Tests for frost durability – material characterization
	Table 6b	Tests for frost durability – freeze-thaw tests
	Table 7	Overview of requirements for frost durable concrete

Table 2. Overview of the documents included in the review

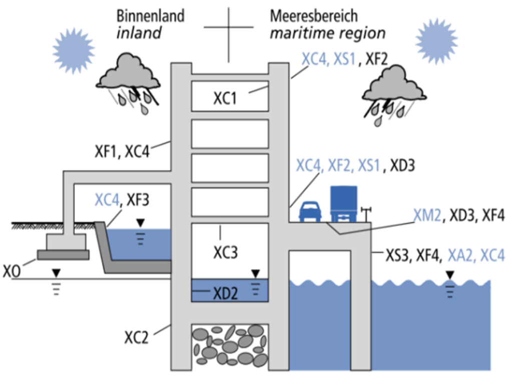
	Europe					Asia		North America	
	Norway	**Sweden**	**Denmark**	**Germany**	**Finland**	**Russia**	**China**	**USA**	**Canada**
Exposure classes, Material requirements	NS-EN 206+NA NS-EN ISO 19903	SS EN 206 SS 137003	DS 2426 (DS 411) DS/EN 1992-1-1 HETEK, Danish Road Directorate	DIN 1045-2	B4, Liite 3, SFS-EN 206-1	GOST 31384-2017 SP 35.13330.2011 GOST 26633-2012	GB 50476-2008	AASHTO LRFD Bridge design spec. ACI 201.2R-01 ACI 302.1R-96 ACI 318-14	CSA A23.1-09-A23.2-09
Production and execution of concrete works	EN 206:2013 EN 13670:2009					GOST 7473-2010 GOST 10181-2014 GOST 30459-2008 SP 70.13330.2012		ASTM C172/C172M ACI 304.2R-00 ASTM C94/C94M-16a	
	NS-EN 13670:2009/NA NS-EN 14487-1+NA NS-EN ISO 19903 NCA Pub. 25 SVV Prosesskode 2	SS 137003		DIN 1045-2 ZTV-ING				ACI 212.3R-2010 ACI 302.1R-96 ACI 304R-00 ACI 318-14 PCA. Volume 19/1 AASHTO LRFD Bridge constr. spec.	CSA A23.1-09-A23.2-09
Tests for frost durability – freeze-thaw tests	CEN/TS 12390-9					GOST 10060-2012	GB/T 50082—2009	ASTM C672 ASTM C666	
		SS 137244 SIS-CEN/TR 15177		Bunke cube test RILEM TC 117-FDC/CDF					MTO LS-412 BNQ NQ 2621-900

2 Exposure characteristics

Most of the standards begin with the description of exposure classes, emphasizing by that a great importance of selecting it properly. Exposure class represents an imposed environmental **Load** that a structural element or a whole structure will oppose to throughout its lifecycle.

Exposure class (further in the text – class) selection for designing of the concrete mix for the structures that undergo freeze-thaw cycles depends on varying saturation conditions, presence of de-icing agents (f.ex. chlorides) and sulfates (seawater).

Combination of environmental loads is often a case when selecting the most appropriate class of the load for a particular element. Some organizations develop free software (ex. Svensk Betong [59]) or guidelines (see an example in figure 1 below), helping end-users to choose the most suitable class for further design.



Expositionsklassen (Umwelteinwirkungen, „Angriffe") *Exposure classes (environmental effects, "attacks")*				Betontechnische Maßnahmen („Widerstände") *Concrete technology measures ("resistances")*			
Klassenbez. *class designation*		Einwirkung *effect*	und *and* Beanspruchung *stress*	Max. w/z *max. w/c*		Min. z *min. c*	f_{ck}, cube *f_{ck}, cube*
XO		kein Angriff *no attack*	kein Betonangriff *no concrete attack*	keine Anforderung *no requirement*		keine Anforderung *no requirement*	C8/10 *C8/10*
XC	1	Carbonatisierung *carbonation* (H_2O, CO_2)	trocken *dry*	0,75		240	C16/20
	2		ständig nass *constantly wet*	0,75		240	C16/20
	3		mäßig feucht *moderately moist*	0,65		260	C20/25
	4		nass / trocken *wet / dry*	0,60		280	C25/30
XD/ XS	1	Chlorid *chloride* (Cl, H_2O)	mäßig feucht moderately moist	0,55		300	C30/37
	2		ständig nass *constantly wet*	0,50		320	C35/45
	3		nass / trocken *wet / dry*	0,45		320	C35/45
XF	1	Frost /+ Salz *freeze-thaw /+ salt*	mäßige Wassers. o. T. *moderate water saturation (o.T.)*	0,60		280	C25/30
	2		mäßige Wassers. m. T. *moderate water saturation (m.T.)*	0,55	+ LP	300	C25/30
				0,50		320	C35/45
	3		hohe Wassers. o. T. *high water saturation (o.T.)*	0,55	+ LP	300	C25/30
				0,50		320	C35/45
	4		hohe Wassers. m. T. *high water saturation (m.T.)*	0,50	+ LP	320	C30/37
XA	1	Chem. Angriff *chemical attack* (Säure *acid*)	schwach angreifend *weakly corrosive*	0,60		280	C25/30
	2		mäßig angreifend *moderately corrosive*	0,50		320	C35/45
	3		stark angreifend *strongly corrosive*	0,45		320	C35/45
XM	1	Verschleiß *wear*	mäßiger Verschleiß *moderate wear*	0,55		300	C30/37
	2		starker Verschleiß *severe wear*	0,45		320	C35/45
	3		sehr starker Verschleiß *very severe wear*	0,45		320	C35/45

Figure 1. Guidelines for exposure class and material parameters selection [60]

Reprinted from "The new German concrete standards DIN EN 206-1 and DIN EN 1045-2 as basis for the design of durable constructions," by Horst Grube, Beatrix Kerkhoff., Concrete Technology Reports, VDZ, Düsseldorf, 2001-2003, p.22

For example, if we design a structure in Sweden, exposed to seawater, and freezing and thawing cycles, the governing exposure classes would be XS3 and XF4 – for the splash zone and XS1 and XF2 – above the area of active splashes (exposed to seawater spray). Consequently, the requirements for concrete should meet the limits described in the national standard for both classes in either case. Normally XF-classes (in Europe) set stricter requirements and, hence, serve as dominant exposure classes for selection of the mix design parameters.

However, there are other practices, regulating requirements and recommendations towards application for some particular structures or its components, which overrule in a loose sense the use of standard requirements. That could be an industrial specification or code of various committees, for instance, for road and highway structures (ex. [14], [15], [24], [38], etc.).

In addition, particular projects like some of the concrete offshore platforms or bridges may have their own set of requirement specifications to concrete (and its constituent materials) in the face of either extensively harsh environment, prolonged design life or construction solutions beyond any other specifications, or simply taking into account stringent Clients' design considerations.

Table 3a presents an overview of how standards in different countries specify exposure classes. **Table 3b** shows an example of a criterion-based comparison of the overviewed standards.

Observations based solely on Tables 3a and 3b (see the Tables below):

1. Europe. EN 206 and modifications of this European standard, i.e. NOR [20], SWE [28], GER [35], DK [33], RUS [48]:

- Neither Norwegian nor Danish standards consider high saturation condition for De-icer-free exposure, differentiating class XF1 from XF3 only by exposed surface orientation. Recommendations from HETEK [34] solve the mismatch for Danish Road Directorate. Norwegian road authorities follow the letter of the standard.
- Both German and Swedish standards do not specify saturation conditions for class XF1
- Only Danish and Swedish standards seem to provide with a full description of exposed surfaces and structures for the classes, while other three national standards give very vague and general definitions.
- German standard display no division to horizontal and vertical surfaces between the classes of exposed structures
- Sweden – the only standard distinguishing the transition from a vertical to a horizontal surface by introducing the threshold requirement for the slope (30deg.).

2. North America

- A substantial difference can be observed in exposure classes characterization between reviewed ACI [1,3,6] and AASHTO [14,15] documents, despite the fact that exposure characteristics of the latter two specifications should logically fall under broader ACI 201.2R Guide for durable concrete and ACI 318 Building Code Requirements for structural concrete.
- ACI 201 [1] and ACI 302 [2] are well aligned.
- AASHTO specifications [14,15] introduce the classes for air-entrained (AE) and high-performance concretes (HPC) of elements in bridge construction. There are many requirements given particularly for class A (HPC) (see Table 4), but the exposure conditions are not defined.
- Canadian standard [17] offers two pure frost classes for horizontal and vertical surfaces without exposure to chlorides (similar to XF1 and XF3 in variations to EN 206 standard). Pertaining exposure to chlorides and to other aggressive matters, like manure, the standard does not put emphasis on the impact of freeze-thaw on requirements for concrete, meaning that other exposure criteria overrule it (see Appendix A, Table A.1).
- There is a mismatch between ASTM C94/C94M [7] for fresh concrete and the building code ACI 318 [6]. When referring to ACI code, ASTM standard wrongly categorizes exposure class F2 as a severe exposure (see Note 5, section 6.1.4 in ASTM C94) with exposure to de-icing salts, while in the ACI F2 is for exposure to only fresh water.

3. Asian approach:

- Both Chinese [51,53] and Russian [48] standards define exposure by lowest temperatures in the freeze-thaw cycle. The temperature ranges vary significantly, depending on the target structural groups: SP 35.13330 is for the bridges and pipes whereas GB/T 50476 is for general civil construction.
- Russian GOST standards are being revised and updated. The new versions resemble European standards, and they refer to them more often. However, the building norms SP (or SNiP) seem to remain independent of that tendency.

None of the standards gives a definition of moderate saturation conditions, splash zone, sea spray exposure, airborne de-icing agent.

Table 3a. Classification for freeze-thaw exposure conditions. LOAD

Country	Standard, Code, Guide	Exposure class	Exposed structures, surfaces classification	Water saturation, exposure	De-icing agents (incl. chlorides)	Temperature	Reference
Norway, NO	NS-EN 206:2013+NA:2014	XF1	Vertical concrete surfaces	Rain, moderate saturation		Freezing and thawing cycles	Table 1, p. 21 [20]
		XF2	Vertical concrete surfaces of road structures	Moderate saturation	De-icing agent (airborne)		
		XF3	Horizontal concrete surfaces	Rain, moderate saturation			
		XF4	Road and bridge decks, splash zone of marine structures and other concrete surfaces	High saturation	De-icing agent or direct spray with de-icer, Sea water		
	NS-EN ISO 19903:2006	Severe	Reinforced and pre-stressed concrete above splash zone	Moderate saturation	Sea spray, rain	Freezing and thawing cycles	Clause 8.3.3.1 [23]
		Very severe	Reinforced and pre-stressed concrete in splash zone	High saturation	Sea water		
Canada, CA	CSA A23.1-09-A23.2-09	F-1	Pool decks, patios, tennis courts, freshwater pools, and freshwater control structures.	Saturated condition		Freezing and thawing cycles	Table 1, p.121 [17]
		F-2	Exterior walls and columns.	Unsaturated condition			
		C-1	Structurally reinforced concrete. Bridge decks, parking decks and ramps, portions of marine structures located within the tidal and splash zones, salt-water pools and other concrete structures		Chlorides, seawater spray		
		C-2	Non-structurally reinforced. Garage floors, porches, steps, pavements, sidewalks, curbs, and gutters.		Chlorides		
		C-XL	Structurally reinforced, highly durable concrete (with higher durability performance expectations than the C-1, A-1, or S-1 classes)		Chlorides or other severe environments		
		A-1	Structurally reinforced concrete exposed to severe manure and/or silage gases. Concrete exposed to the vapor above municipal sewage or industrial effluent. Reinforced elements over manure pits/silos or in contact with effluents				
		A-2	Structurally reinforced concrete exposed to moderate to severe manure and/or silage gases and liquids. Exterior walls and slabs of manure tanks.				
		A-3	Structurally reinforced concrete exposed to moderate to severe manure and/or silage gases and liquids in a continuously submerged condition.				

Country	Standard, Code, Guide	Exposure class	Exposed structures, surfaces classification	Water saturation, exposure	De-icing agents (incl. chlorides)	Temperature	Reference
USA	ACI 201.2R-01	Moderate	Certain exterior walls, beams, girders, and slabs not in direct contact with soil	Occasional contact with moisture		Freezing and thawing cycles	Clause 1.4, Table 1.1 [1]
		Severe	Concrete of thin sections (bridge decks, railings, curbs, sills, ledges, and ornamental works)	Almost continuous contact with moisture	De-icing salts	Freezing and thawing cycles	
	ACI 302.1R-96	Moderate	Concrete floors and slabs			Freezing and thawing	Clause 6.2.3 [3]
		Severe	Concrete floors and slabs	High saturation	De-icing chemicals		
			Reinforced concrete		Brackish water, seawater, de-icing chemicals, other aggressive materials		
	AASHTO LRFD Bridge design and construction specifications	Class A (AE),	Concrete for all elements of structures		Saltwater (in or above), De-icing salts	Alternate freezing and thawing	C5.4.2.1 [14], Table 8.2.2-1 [15]
		Class A (HPC)	Cast-in-place construction where performance criteria should be specified separately				
		Class B (AE)	Concrete for footings, pedestals, massive pier shafts, and gravity walls		De-icing salts, saltwater		
		Class C (AE)	Concrete for thin sections, such as reinforced railing less than 10cm thick, for filler in steel grid floors				
	ACI 318-14	F1	Exterior walls, beams, girders, and slabs not in direct contact with soil.	Limited exposure to water.		Freezing-and-thawing cycles	Tables 19.3.1.1 and R.19.3.1 [6]
		F2	Members that will be subject to snow and ice accumulation, such as exterior elevated slabs. Horizontal and vertical members in contact with soil. Foundation or basement walls extending above grade.	Frequent exposure to water, possible saturation. Snow and ice accumulation against surface			
		F3	Horizontal members in parking structures. Foundation or basement walls extending above grade.	Frequent exposure to water, possible saturation. Snow and ice accumulation against surface	De-icing chemicals		
Germany	DIN 1045-2	XF1	Outdoor components			Freezing and thawing cycles	Table 1 [35]
		XF2	Elements in spraying or splashing areas of treated traffic areas that do not fall under XF4. Concrete elements in the seawater spray area	Moderate saturation	De-icing agent, Sea water spray		

Table 3a continued

Country	Standard, Code, Guide	Exposure class	Exposed structures, surfaces classification	Water saturation, exposure	De-icing agents (incl. chlorides)	Temperature	Reference
Germany	DIN 1045-2	XF3	Structural components in the area with varying fresh water level (direct translation - "water exchange zone")	High saturation		Freezing and thawing cycles	Table 1 [35]
		XF4	Traffic areas treated with de-icing agents. Mainly horizontal surfaces in the spraying area of treated traffic areas. Reservoirs of clear water; Marine structures in the tidal zone	High saturation	De-icing agents, sea water		
Sweden	SS EN 206:2013 (with additional clarification from Heidelbergcement group)	XF1	Vertical surfaces exposed to rain or freezing. Outdoors with slopes greater than 30 degrees. Facades, indoors in uninsulated buildings, escape routes, as well as heated and ventilated side spaces in road tunnels			Freezing and thawing cycles	[28]
		XF2	Vertical surfaces exposed to freezing Parts of bridges under the ground. Otherwise traffic space in road tunnels.	Moderate saturation	De-icing agent (airborne)		
		XF3	Horizontal surfaces exposed to rain and freezing. Outdoors with slopes lower than 30 degrees. Constructions in fresh water with / without one-sided pressure or exposed to splash. Outdoor water pools, balconies without risk for de-icers, dam structures, parts of bridges / tunnels that are not in the road environment	Rain, high saturation			
		XF4	Road and bridge decks, surfaces directly exposed to splashes of de-icing agents. Splash zone of marine structures. Decks and joints of outdoor parking houses, garage driveways, ground concrete, concrete pavements, stairs / ramps, decks in timber drying areas, upper surface of the bridge deck parts of bridges / tunnels that belong to road environment	High saturation	De-icing salts, sea water		
Denmark	DS 2426 (DS/EN 1992-1-1 DK NA:2011, which replaced DS 411)	XF1 (moderate)	Vertical concrete surfaces. Foundation piles and foundations partly above ground, external walls, columns and facades, external beams with structurally protected upper side, balcony railings, installation channels, elevator pits.	Rain, moderate saturation		Freeze-thaw cycles	Clause 4.2 [33]
		XF2 (aggressive)	Vertical concrete surfaces of road structures. Retaining walls, exterior stairs, basement exterior walls partially above ground	Moderate saturation	De-icing agent (airborne)		

Table 3a continued

Country	Standard, Code, Guide	Exposure class	Exposed structures, surfaces classification	Water saturation, exposure	De-icing agents (incl. chlorides)	Temperature	Reference
Denmark	DS 2426 (DS/EN 1992-1-1 DK NA:2011, which replaced DS 411)	XF3 (aggressive)	Horizontal concrete surfaces Outside deck, external beams without structurally protected upper side, exterior stairs	Rain, moderate saturation		Freeze-thaw cycles	Clause 4.2 [33]
		XF4 (extra aggressive)	Road and bridge decks, splash zone of marine structures and concrete surfaces, exposed to direct spray containing de-icer and freezing. Balconies and their elements, parking decks, swimming pools, bridge columns, edge beams on bridges.	High saturation	De-icing agent or direct spray with de-icer, Sea water		
	HETEK committee, Danish Road Directorate	1	Splash zone structures, pavement slabs, edge beams, decks, continuously moisture exposed columns and vertical walls, back-filled support and retention walls and decks without membrane	Exposed to water, high saturation	With or without salt	Freezing and thawing cycles	Report No. 97, 1997, p.13 [34]
		2	Decks with intact membranes, crash barriers, columns and vertical walls, not exposed to capillary suction, but less than 1,5m from splash zone	Periodical water exposure	With or without salt		
		3	Vertical surfaces – back-filled retention walls with membranes, sheltered columns	Rare exposure to water			
Russia	GOST 31384-2017[1]	XF1	Vertical surfaces of the buildings and structures	Rain, moderate water saturation		Freezing and thawing cycles	Table A.1 [48]
		XF2	Vertical surfaces of transport works	Moderate water saturation	De-icing agents		
		XF3	Horizontal surfaces of the roads and other structures	High water saturation			
		XF4	Horizontal surfaces of roads and bridges, outdoor staircases, etc. Marine structures	High water saturation	De-icing agents, incl. sea water		
	SP 35.13330.2012[2]	Severe	Surfaces of massive structures and thin walls (less than 0.5m thick)	Underwater (0.5m below ice exposed surfaces), under- and over-ground flood-free zones		-10…-20 C°	Table 7.5 [50]
		Extra severe	Surfaces of massive structures, thin walls, external and internal concrete blocks	Varying water level (splash / tidal zone)	Sea water?	Below -20 C°	
		De-icing salts	Elements listed for severe and extra severe exposure		De-icing agents	Below -10 C°	

Table 3a continued

Country	Standard, Code, Guide	Exposure class	Exposed structures, surfaces classification	Water saturation, exposure	De-icing agents (incl. chlorides)	Temperature	Reference
China	GB/T 50476-2008 [51]	II-C	Components of water level variation zone, horizontal surfaces	High. Frequent rain, water level variation zone	/	-3...+2,5	Peng Zhang [53]
			Vertical surfaces	Moderate. Rain	/	-3...-8 and below -8	
		II-D	Components of water level variation zone, horizontal surfaces	High. Frequent rain, water level variation zone	/		
			Components of water level variation zone, horizontal surfaces	High. Frequent rain, water level variation zone	Chlorides	-3...+2,5	
			Vertical surfaces	Moderate. Rain		-3...-8 and below -8	
		II-E	Components of water level variation zone, horizontal surfaces	High. Frequent rain, water level variation zone			

Table 3a continued

[1] Exposure classes for concretes in the aggressive environment, inducing corrosion by the action of alternating freezing and thawing.

[2] Standard specifies the requirements for minimum frost resistance class in particular exposure conditions

Table 3b. Summary of exposure classes from the reviewed standards and specifications

Saturation and additional criteria	Hetek	NS-EN ISO 19903	DS 2426	NS-EN 206	GOST 31384	SS EN 206	DIN 1045-2	ACI 318	ACI 302	ACI 201	CSA A23	GB/T 50476	AASHTO	SP 35.13330	De-icing agents
	DK	NOR	DK	NOR	RUS	SWE	GER	US			CAN	CHI	US	RUS	
High saturation, sea splash	Class 1	Very severe	XF4							Severe	C-XL	II-D, II-E	Class A (HPC)		De-icing salts
High saturation					XF3			F2			F1	II-C, II-D		Severe	No de-icing salts
Moderate. Horiz. surfaces	Class 2		XF3					F1 / F2	Moderate		F2				
Moderate saturation, sea spray		Severe	XF2					F3			C1	II-D	Class A, Class B		De-icing salts
Moderate saturation Vertic. surfaces				XF1				F1		Moderate		II-C		Severe	No de-icing salts
Rare contact with water	Class 3					XF1			Moderate						
Parking (garage floors), ground concrete			XF4		XF4			F3			C2		Class B		De-icing salts
Thin walls, sections										Severe			Class C	Extra severe	
NA									Severe		A1-A3				Other aggressive matters

Note: The table attempts to summarize collected information in a simple manner. More criteria could be included into that comparison table: direct contact with ground, reinforced or not, etc.

3 Material requirements

It is not that ambiguous that the main parameters for designing frost durable concrete remain the same as for regular concrete: water-to-cement (w/c) or water-to-binder (w/b) ratios with or without efficiency factors for SCM, compressive strength and air-void content. However, when we mean frost–durable or –resistant concrete, there are other material requirements to be involved – air-void spacing factor, binder proportions, and limitations, permeability and frost resistance for aggregate and concrete.

All the above-mentioned material requirements represent **Resistance** of the structure or the element against the **Load** in the form of environmental exposure, determined in accordance with Table 3a.

Table 4 shows the presence of given controlled parameters for concrete (mix design, its fresh and hardened state) in various national standards and specifications. The requirements are given to ensure a minimum of 50 years service life (or more, depending on concrete cover), unless other specified.

Observations:

1. Air-void parameters:

- In Europe, it is only Sweden and Denmark, who set the importance of air void parameters (spacing factor) in the main concrete standard (EN 206).
- It is unclear in which state of the concrete (fresh or hardened) it should meet the requirements for air content. T.A.C is documented in hardened concrete only in Denmark, among all the reviewed standards; however, Sweden presumably does the same despite no clarity in the standard text. Both standards also measure T.A.C in the fresh state.
- Swedish standard avoids setting a requirement for T.A.C and air-void spacing factor for the most severe exposure XF4.
- Even though the Canadian standard [17] sets general requirements for the average air void spacing factor of 230 µm (no single result greater than 260µm), it recommends (I.3.8, Clause 4.3.1) especially for highly workable HPC concretes using a target maximum allowed spacing factor of 170 µm.
- The Air Void Analyzer or AVA (acc. to chapter C4.6. [57]) can be considered for use in monitoring the variability of the air void system in fresh concrete during production. Although its correlation with other test methods is still to be proven, AVA stands as an alternative test method for air content (Table C1.0-2 [57]) in line with standards ASTM C231 and ASTM C457.
- Russia.
  Interesting that GOST 26633-2012 for heavyweight concrete [46] had clear requirements to air-void content varied with D_{max} and w/c-ratios (see the Table below), accepting only the concrete with F/T-resistance over F_1200 (F_1 – in acc. with Basic first method< see Table 6b). Whereas the newest revision, called GOST 26633-2015, exhibits far less information and sets a general requirement for air content at 4% for all concretes with F_1200 (F_2100) independent of D_{max} and w/c, and the table below is no longer there or in any other standards.

Copy of Table B.7 (Б.7 - in Russian) [46]

Max size of aggregate particles (D_{max}), mm	Air-void content for fresh concrete, %		
	w/c ≤0,41	w/c = 0,41…0,50	w/c > 0,50
10	3…4	3…5	5…7
20	1…3	2…4	4…6
40		1…3	3…5
80			2…4

Worth mentioning that valid standards in Russia often refer in the text to old expired documents. Therefore, it makes it harder to pick a right selection of standards to follow and makes it easier to make a mistake.
Looking at the numbers, the Russian standard from 2012 does not seem to require air-entraining agents for concretes with w/c below 0.41, which is, best to our knowledge, the only standard, defining the lowest w/c-ratio limit for the need of air entrainment. This particular case requires rather thorough review of Russian standards.
Same GOST 26633-2015 limits w/c to 0.45 and the air content – to 5-7 % for topcoat for roads and airfield pavements [46 – 2015 version].

- Chinese standard focuses on requirements for air content (for certain aggregate size) and the spacing factor as a function of the degree of water saturation and the presence of de-icing salts (see Appendix B.1)

2. Supplementary cementitious materials (SCM):
    - German standard DIN 1045-2 sets requirements for a separate category of slow hardening concretes (with fly ash) when identifying limiting values for concrete that is to be exposed to freeze-thaw (see Table 6).
    - Canadian standard suggests using SCM in case of combined sulfate and chloride attacks.
    - Russian GOST R 55224-2012 [64] (Clause 5.11.2) accepts blast furnace slag as the only main mineral addition or cement replacement in concretes for road topping and airfield pavements, for reinforced elements and bridge structures.
3. Durability parameters:
    - Norwegian national annex introduces durability classes, which unify requirements for concrete under six (6) different sets for corresponding exposure classes (Table NA.11 in [20]). It makes it possible to simplify the process of selecting the requirements in the event of combined actions.
    - Finnish standard [63] uses unique P- and F-values as key parameters for concretes in the presence of de-icing agents (XF2, XF4) and without (XF1, XF3) respectively. The F-value depends on w/c-ratio and T.A.C (for D_{max} -16mm), while for P-value, in addition, binder composition and curing (see Appendix B.2) have meaning.
    - In Russia, the frost resistance class is assigned to particular types of structures and exposure conditions with special focus on the severity of freeze-thaw cycles (different requirements for lowest temperatures), see GOST 31384-2017 [48] (Table E.1 in the standard) and Appendix C2, Table C2.1. Building regulations and rules (known as SNiP or superseding it - SP), which the design of new works is based upon, provide own guidance on which parameters the concrete should meet (see Note 3 for Table C2.1, Appendix C2).
4. Other requirements:
    - Efficiency factors:
        - Both in Canada and in the USA, there are no efficiency factors (k_{eff}) used for SCMs. W/CM = Mass of Water / Total mass of PC + SCM.
        - There is no agreement in Europe on k_{eff} value for fly ash, used when calculating the so-called effective water-to-cement ratio (w/c_{eff}) or mass-ratio. However, most of the standards suggest using w/c_{eff} as a key parameter for concrete mix design (see a definition of c_{eff} in a footnote 1 under Table 4)
    - Only German and Russian *main concrete standards* specify particular acceptance criteria for frost resistance of aggregate.

DIN 1045-2 [35] (tables F 2.2. and U1), however, introduces two different frost resistance categories, referring to DIN EN 12620 (Tables 18 and 19 in that standard). Aggregates for XF1 and XF3 exposure is tested in fresh water (EN 1367-1), while for the exposures XF2 and XF4 (de-icing agents and seawater) aggregates are tested either by a procedure for determination of magnesium sulfate value (EN 1367-2), so-called “direct method”, or in the presence of de-icing salts (1%NaCl or urea). However, it should be mentioned that no clear relationship exists between the frost resistance of the aggregate as determined by EN 1367-1 and how the aggregate will perform in concrete during freeze/thaw.

- Norway puts an emphasis on the need for frost-resistant aggregate only for XF4 class, while Danish standard does not mention this requirement at all in the *main country concrete standard* [32].
- Russian SP 41.13330.2012 [81] recommends which type of admixtures for concrete to use as a function of structure type and exposure characteristics (see Annex B, Tables B.1 and B.2 in the standards).

Note:

It is often the calculated physical load that influences the material requirements, making concrete mix design process rather complicated and comprehensive.

For example, when we have a combined environmental load XF4 (European standard) and physical abrasive load from ice drift, the requirement for concrete strength increases from a standard value of C30/37 for XF4 to C70/85 (class B70). However, frost exposure demands certain air content, say 5%, which by thumb rule for high strength concrete would reduce compressive strength (in comparison with non-air entrained concrete – around 2.5% T.A.C.) without any extra measures by 5-10% for each volume % of additional air. In order to compensate for that strength loss, concrete technologist should reduce w/b-ratio to 0.26-0.28 or use different measurements to meet the required strength level.

Another point is the difficulty could be when one should combine multiple material and execution requirements.

For example, for an offshore structure the frost exposure adds up a challenge for mix design, having at the same time requirements for 500 cycles F/T resistance in ASTM C666 (proc. A) [61], high corresponding compressive strength of C60/75, 5-8% for total air-void content, increased open time of fresh concrete, massive casts, suitability for slipforming, hence highly workable and SCM containing concrete with low w/c [62].

Examples above show that keeping high quality (=high degree of fulfillment of all the requirements) during the production of this kind of concretes is very demanding in practice without a comprehensive, time-consuming and expensive prequalification program.

Table 4. Material requirements. RESISTANCE

Country	Standard	Exposure class, exposed structure	Controlled parameters: Mix design, fresh concrete: Max w/c (effective w/c)	Min (effective Ceff[1]) binder content. kg	Min cement content (when SCM used)	Max (Min) SF content, %	Min strength class for cement	Min (Max) V+D+S[3], %	Max FA content[2] (Max Fa/C-ratio), %	Min air content (for aggregate D_{max}, mm), %	Durability class	Frost resistant aggregate (Class for aggregate)	Hardened concrete: Min comp. strength, MPa or class	Hardened air-void content, %	Max air-void spacing factor (specific surface)	Permeability class	Frost resistance (Class for frost resistance)
General, Europe, EU	EN 206:2013	XF1		(+)		+			(+)				+				
		XF2, XF3, XF4		(+)		+			(+)	+			+				
Norway, NO	NS-EN 206:2013+NA:2014	XF1	+	(+)		(+)		+	+		+		EU				
		XF2, XF3, XF4	+	(+)		(+)		+	+	+	+	+ (XF4)	EU				
	NS-EN ISO 19903:2006	Severe	(+)	+	+[6]	+			+	+, (+)			+				
		Very severe	(+)	+	+	+			+	+, (+)			+		+, (+)		
Sweden, SE	SS 137003:2015	XF1	(+)	(+)		+	+		+			+	EU				
		XF2, XF3	(+)	(+)		+	+		+	+, (+)		+	EU		+		+
		XF4	(+)	(+)		+	+		+			+	EU				+
Germany, GER	DIN 1045-2:2008-08	XF1	(+)		+, (+)							(+)[7]	+				
		XF3	(+)		+, (+)					+, (+)		(+)[7]	+				
		XF2,XF4	(+)		+, (+)					+, (+)		(+)[7]	+				+
Denmark, DK	DS 2426 (DS 411)	XF1	(+)		+	+	+		+, (+)	+			+				
		XF2, XF3, XF4	(+)	+	+	+	+		+, (+)	+			+	+	+		
Finland, FI	B4, Liite 3, SFS-EN 206-1	XF1, XF3	+		+	+		(+)	(+)								
		XF2, XF4				+		(+)	(+)								
Canada, CA	CSA A23.1-09-A23.2-09	A-1, A-2, A-3, F-1, F-2, C-1, C-2, C-XL	+[4]							+, (+)			+[5]		+		
Russia, RU	SP 35.13330.2011	Bridge										(+)				+	(+)
	GOST 26633-2015[8]	Road surface layer	+							+		(+)					

Country	Standard	Exposure class, exposed structure	Controlled parameters														
			Mix design, fresh concrete										Hardened concrete				
			Max w/c (effective w/c)	Min (effective C_{ef}[1]) binder content. kg	Min cement content (when SCM used)	Max (Min) SF content, %	Min strength class for cement	Min (Max) V+D+S[3], %	Max FA content[2] (Max Fa/C-ratio), %	Min air content (for aggregate D_{max}, mm), %	Durability class	Frost resistant aggregate (Class for aggregate)	Min comp. strength, MPa or class	Hardened air-void content, %	Max air-void spacing factor (specific surface)	Permeability class	Frost resistance (Class for frost resistance)
USA	AASHTO LRFD Bridge construction specification	Classes A(AE), B (AE), C (AE)	+		+	+		(+)	+	+, (+)			+				
		Class A (HPC)	+		+	+		(+)	+	+, (+)			+				
	ACI 201.2R-01		+							+, (+)					+, (+)		
	ACI 318-14	F1, F2	+							+, (+)		+	+				
		F3	+	+		+		(+)	+	+, (+)		+	+				
China	GB/T 50476-2008	II-C, II-D, II-E	+							+, (+)			+		+		+

Table 4 continued

Note: **(+)** means that a given standard uses a material requirement, written in **(**parentheses**)** in the table heading, f. ex. SS 137003:2015 specifies requirements for effective w/c, while ACI – for max acceptable w/c.

[1] C_{eff} =C+∑ki×pi, where Pi - puzzolana (FA, SF, GBFS)

[2] Requirements for FA content usually come along with the requirements for slag content.

[3] V - fly ash, D - silica fume, S – slag

[4] The maximum water-to-cementing materials ratio of the HVSCM-1 (high volume of supplementary cementitious materials) concrete, exposed to freezing and thawing, shall be reduced by 0.05 in all exposure classes.

[5] Minimum strength requirement for HVSCM-1 concrete (min 40% FA) and for exposure class C-XL should be given for 56 days.

[6] Requirement depends on D_{max}

[7] Standard specifies separate requirements for aggregates in concrete, depending on water saturation and exposure to chlorides or de-icing salts. Consequently, aggregates for concretes in classes XF2 and XF4 to be tested in salt (1%NaCl), XF1 and XF3 – in water. Limits of weathering or mass loss are different for each exposure class

[8] Retrieved from Appendix B. Additional requirements to concretes for various types of structures

4 Requirement to production and execution of concrete works

A general principle for the reviewed standards and recommendations can be embraced by the following quote from Hooton [65]: “Durable concrete can be made for most aggressive exposures provided appropriate materials, mix designs, and construction practices are followed. Blended cements and SCMs will improve the durability of concrete, but as with any cementing material, it cannot guarantee durability if it is not used in good quality concrete or if concrete is poorly placed and cured” (matured).

Practical understanding of how to obtain frost resistance and frost durability of severely exposed concrete boils down to composition and execution (mixing/workability/placement/execution/curing). Particularly the air entrainment is important. All the factors that may prevent the formation of the so-called adequate air-void system of the exposed parts of a concrete structure should be taken into consideration during production and execution phases.

Table 5 is focused on the production and execution parameters, which influence frost resistance of concrete.

Observations based on the reviewed standards:

0. General:

- The connection between a specification for production and execution and certain exposure characteristics depends on the specifics of the issuing organization. For instance, reviewed road authorities (AASHTO, SVV, ZTV) deal with similar exposure when they design roads, bridges, top coats, namely, saturated conditions, de-icing agents or sea spray. Therefore, their requirements are inseparably associated with exposure. Among the national standards reviewed, Canadian [17] and Russian [71] also connect production and execution with the exposure classes.

1. Mixing of concrete is scarcely mentioned in any of the standards.

- Recommendations to mixing time seem to exist only in North American standards [4,7,14]. AASHTO recommends increased mixing time for saltwater exposure, supposedly to ensure homogeneity of the mixture and activation of air-entraining agents used in their concrete mixes.
- Two documents of the selection [7, 38], namely ASTM standard and German construction guidelines from the road authorities, surprisingly offer a possibility for the addition of water after mixing without providing clear instructions on how to implement that. This we consider a lack of these two standards.
- None of the documents inform about effects of SCM in combination with admixtures, nor enlighten on the best practices for a combination of admixtures in the concrete mix. This is a lack since it is known that the effectiveness of the admixtures will vary with type and quality of pozzolans, with variation in the sequence of addition for admixtures and with their compatibility [4, clause 4.5]. It was clearly demonstrated in the study how varying the sequence of addition for admixtures can affect the air void system [75].

2. Transportation and delivery of fresh concrete.

- Russian standards [41,44,47] require that retention of workability is checked, compared to the actual transport time.
- Particular requirements do not seem to exist in the reviewed European documents.

- In North America, if we consider ASTM [7] requirement as a general limitation for transportation from A to B (max 1.5 hours), then for highways in AASHTO standard [15] it includes in the same timeframe placement and consolidation (practically reducing maximum allowed transportation time by ca 30 min). At the same time, the guide ACI 304R [4] sets even stricter requirements.

3. Fresh concrete measurements

Air void content in concrete. Sampling:

- European requirements for control of the air content were not found, either in concrete specification nor in the standard for execution. This lack of specification on where and when to measure fresh air content and workability can we consider a serious lack.
- The European standard for execution EN 13670, Chapter 8. Casting [ref. 21, with Norwegian National Annex], seems to be kept on a general level assuming that the contractor/executing party has all the detailed competencies necessary to ensure that specified quality is reached in the final concrete structure.
- In the USA, T.A.C is controlled at the concrete batch plant [7] and upon delivery of the truck on site [6]. Impact of a subsequent delivery, especially in the case of concrete pumping, should be taken into consideration during mix design in accordance with PCA and ACI recommendations [4,16].
- The Norwegian and Russian national standards [21,44,47] do not require to control T.A.C after transportation and use of delivery equipment, whereas Norwegian road authorities [24] and Canadian, Swedish and German national standards [17, 29,35] will have concrete checked immediately before placing.
- The frequency of the tests under production varies significantly. CSA [17] requires high reproducibility for concretes that are to be in saturated condition and exposed to de-icing agents, whereas other documents do not seem to have such a requirement.

Air content. Verification tests:

- Russian standards offer retention tests or test of the stability of technological parameters (air content, temperature, workability, fresh density, bleeding). However, there is no uniformity of the description for required properties retention found between GOST 7473 [41], GOST 30459 [47] and GOST 10181 [44], which entails a contradiction between producers and users. Even though GOST 7473 for fresh concrete is logically the first to check the requirements, a majority of the producers is prone to follow GOST 30459 for admixtures, since they are interested mainly in workability retention. However, GOST 10181 for test methods sets its own list of technological requirements to fresh concrete that should be retained in time.
- American ACI 304.2R [5] for concrete pumping and ASTM C172 [9] for fresh concrete suggest making a verification test before and after pumping to account for air loss and change in air-void structure in the mix design. At the same time, ACI 212 [2] suggests verification by using unit weight in addition to the air meter test.
- SVV [24,25] require reproducibility of T.A.C-value 3 times in a row with pressure meter with circa 25% allowable tolerance.
- The specifications do not stipulate verification tests for concrete containing SCM in terms of stability of air entrainment.

W/C-ratio control during production seems to be of interest only for the road authorities [24, 38]

Temperature control.

- The standards agree on the minimum allowable fresh concrete temperature of 5ºC. It is the road authorities [15, 38] that are more concerned about maximum concrete temperature to avoid high (over 70ºC) peaks in hardened concrete, which are likely to jeopardize concrete durability due to thermal crack formation, coarsened pore structure, delayed ettringite formation etc.
- Stability of the concrete temperature is very important because the variations in temperature entail deviations in T.A.C and air-void parameters [4, 72].

4. Placing and finishing

- ACI 302 [3] for slab and floor is the only document among all reviewed that offers finishing techniques and rules in order to prevent surface scaling under freezing from happening.
- ACI 304.2R [5] with a reference to [56] says that ordinary pumping has a minor effect on the spacing factor and, hence, frost resistance of the concrete. However, Jacobsen et al. 2012 [72] found that different researchers discovered a strong impact of pumping on an air-void system of concretes with low w/c-ratio, which is contrary to work presented in ACI standard.

5. Surface protection and curing

- There is a big difference between European and Russian codes for requirements for surface strength before first freezing (5MPa versus minimum 70% of design strength).
- Only AASHTO and CSA standards draw attention to curing precautions for concretes that are to be subjected to de-icing agents.
- AASHTO assigns a requirement for minimum strength level for concrete before exposure, while other documents from North America simply require a number of days and conditions for curing independent of concrete mix design.
- For offshore concrete structure NS-EN ISO 19903 [23] imposes the requirements to strength development (degree of hydration) and climatic conditions when curing (see a copy of the table from the standard below).

Reproduced from NS-EN ISO 19903 [23], table 7, page 66
Minimum values of a ratio for the strength of concrete at the end of curing to 28d strength

Climatic conditions when curing			**Strength proportion**		
			Submerged zone	Splash zone	Other zones
H	Humid	RH > 80%	0,5	0,6	0,5
M	Moderate	65% < RH ≤ 80%	0,6	0,7	0,6
D	Dry	45% < RH ≤ 65%	0,6	0,7	0,6
VD	Very dry	RH ≤ 45%	0,7	0,8	0,7

Note: Alternatively, curing duration can be estimated by calculating maturity based on appropriate function, suitable for selected binder combination. It is suggested that surface permeability or strength of concrete cover can also prove the equivalence of curing.

Table 5. Production and execution of concrete works. Requirements and recommendations

Country	Standard / Document	Execution parameters	Requirements / Recommendations
Europe	EN 206:2013 [20, p. 42]	Min temperature of fresh concrete	5 °C at the time of delivery
		Determination of curing time	Determination by a strength development curve at 20°C between 2 and 28 days or acc. to Table 16. Then curing class is determined by Table 4 in EN 13670 [21] with a subsequent duration determined in acc. with Annex F [21]
	EN 13670:2009 [21, p.27]	Curing regime	The concrete temperature should not fall below 0°C until the concrete surface compressive strength is min 5MPa
		Peak concrete temperature	Shall not exceed 70°C upon hydration, unless specified and proved otherwise
Norway	NS-EN 13670:2009/NA:2010 [21]	Total air content. Frequency of tests	Measured on starting and every 50 m^3 and at least every 3 hours
	NS-EN 14487-1+NA:2012 [22]	Total air content control	No requirements for control of air content for sprayed concrete
	Kværner AS [54] NCA Pub. 25 [26]	Protection against surface frost damage of young concrete during slipforming	Use insulation mats (with possible heating), covering young concrete under the form until it gained sufficient strength (ca 2-3 days old) to withstand F-T cycle, in addition to compulsory curing compound protection. Insulation should be evaluated for every case.
	NS-EN ISO 19903 [23]	Surface protection in freezing conditions	Concrete slabs and other parts that can become saturated shall be protected from the ingress of external water for at least 7 days after casting. Differential temperature across a section should not be allowed to exceed 10°C per 100mm to minimize early age cracking. Peak concrete temperature max 70°C. Concrete should always stay above 0°C until it reaches 5MPa
	SVV Prosesskode 2 [24]	Total air content and temperature (when cold or hot weather). Frequency of tests	Measured (just before placing, i.e. after pumping) every 50 m^3 or at least every 3 hours
		Stability of air content	Air content is stable if 3 consecutive measurements are within tolerance of ±1,5%
		Mass ratio	Tested for every 2000 m^3, produced by the plant (acc. to [25])
Sweden	SS 137003:2015 [29]	Total air content. Time of sampling	Immediately before placing
Germany	DIN 1045-2 [35] ZTV-ING [38]	Total air content. Time of sampling	In-situ just before placing
	ZTV-ING [38]	Maximum fresh concrete temperature	25 °C - for tunnel elements, 30 °C - for other structures
		Addition of water after mixing	Planned subsequent addition of water is permitted (after Client's consent) for concrete for pavements in-situ.
		W/c determination in fresh concrete	Within 1 hour after mixing. Microwave oven method [70] - 5.000±1g sample baked for at least 20min.

Country	Standard / Document	Execution parameters		Requirements / Recommendations
Russia	GOST 7473-2010 [41]	Maximum transportation time		Controlled by workability retention category[1]
	GOST 10181-2014 [44]	Retention of technological parameters: workability, fresh density, total air content, bleeding, fresh temperature		Two tests to be compared: within 15 min after production and within 20 min after delivery to site
	GOST 30459-2008 [47]	Total air content (when AEA[2] is used)		Immediately and 1 hour after mixing
		Properties retention - slump		Immediately and every 30 min after mixing
	SP 70.13330.2012 [71]	Curing regime	General	Protection against evaporation and meteorological precipitation until concrete reaches 70% of designed strength with subsequent maintenance of temperature-humidity conditions. In winter, it is curing during 5-7 days with concrete temperature 5-10°C
			For concretes exposed to freeze-thaw in saturated conditions	The concrete temperature should not fall below 0°C until the concrete reaches 80% of designed compressive strength
North America	ASTM C172/C172M [9] ACI 304.2R-00 [5]	Total air content and workability control		A correlation test is suggested by comparing concrete entering the hopper and that discharged at the end of the pipeline
	ASTM C94/C94M-16a [7]	Mixing time (without mixer performance test)		Not less than 1 min for mixers of 1yd³ (0,76m³). Increase by 15 seconds for each cubic meter (or cubic yard) of increased mixer capacity. It gives about 2 min of mixing time for 4m3 batch plant mixer
		Addition of water after mixing		A one-time addition of water is not prohibited, but it shall be completed within 15 min from the start of the first addition (ref. Clause 12.7 [7]). In that case, the drum of the mixer shall be turned min 30 additional revolutions.
		Maximum transportation time		Max within 1,5 hours after water is added into the mix (or cement introduced to the aggregates)
		Maximum fresh concrete temperature		32°C (90°F)
		Workability, temperature, density, total air content. Frequency of tests		At least every 115 m³ (150yd³). Sampling to be done after the discharge of not less than 0.25 m³ of concrete.
Canada	CSA A23.1-09- A23.2-09 [17]	Curing regime. General (see Appendix 1, Table A1)		Additional curing (7d, +10°C) – mainly for structurally reinforced concrete (except for elements submerged in manure) and saturated condition. Basic curing (3d, +10°C) for other elements and unsaturated conditions.
		Total air content. For exposure classes: F-1, C-XL, C-1, C-2 (Table 2)	Time of sampling	Measured from the first portion of the concrete prior to placement, i.e. after the delivery.
			Frequency of tests	Every load or batch of concrete shall be tested until satisfactory control of the air content is established. Whenever a test falls outside the specified limits, the testing frequency shall revert to one test per load or batch until satisfactory control is re-established.
		Curing regime. For exposure classes: C-XL, C-1, C-2		Recommended for concrete to air-dry for at least a month after the end of the curing period, before exposure to de-icing chemicals

Table 5 continued

Country	Standard / Document	Execution parameters	Requirements / Recommendations
USA	ACI 212.3R-2010 [2]	Total air content. Verification	Air content readings should be verified on site by unit weight (density) test, ASTM C138/C138M
	ACI 302.1R-96 [3]	Finishing rules (to avoid scaling)	Air-entrained concrete should not receive a troweled finish. Magnesium floats should be used. Any finishing operation performed while there is excess moisture or bleed water on the surface is strictly forbidden
	ACI 304R-00 [4]	Maximum transportation time (for trucks with and without an agitator)	30-45 min (with possible corrections on weather conditions)
	ACI 318-14 [6]	Total air content. Time of sampling	As the concrete is discharged from a mixer to the conveying equipment, transferring concrete to the forms.
	AASHTO LRFD Bridge construction specification [15]	Mixing time for concrete Exposure to saltwater	More than 2 min, but less than 5 min (without concretizing the volume of the mix)
		Maximum fresh concrete temperature. Bridge decks. Exposure to saltwater	80°F (ca 26,5C°) at time of placement
		Maximum time for placement and consolidation	Prior to initial set and in no case more than 1.5 h after the cement was added to the mix
		Curing regime	Maintain the temperature of concrete above 45F (ca 7,2 C°) at least 6 days, when up to 25% of cement is replaced by pozzolana or up to 50% by slag – at least 10 days until concrete reaches 70% of designed strength
		The time required before exposure to salt water	Prevent salt water from coming in direct contact with the concrete for a period of not less than 30 days after placement
	PCA. Volume 19/1 [16]	Air loss when pumping concrete	Suggests to account for the reduction in air content from 2 to 3%

Table 5 continued

[1] Producers for simplification classify workability retention in 3 classes C1 – < 20 min, C2 – 20…60 min, C3 - > 60 min, which are connected to binder properties, temperature, etc.

[2] AEA – air entraining agent

5 Overview of test methods for freeze-thaw resistance. Requirements to frost durable concrete

An overview of freeze/thaw test methods is given. Testing is mainly done to understand what material characteristics that matter the most in the performance testing for frost resistance and to compare mixes with acceptance criteria.

Requirements and tests should ideally focus on documenting that with due curing conditions composition (especially mass-ratio) and material quality come out as specified in the finished concrete structure. This includes the entrained air voids that must survive the entire concrete production cycle to give the right air void system in the hardened structure. However, not all the reviewed documents follow this logic.

Table 6a shows material characterization and performance criteria, which we can relate to frost resistant concrete. The main material parameter measured is air void content.
Main observations:

- (Ultra) High-performance concretes in accordance with practice for offshore concrete structures [62] require only results from ASTM C666, procedure A to judge about frost durability of the structure. Whereas road authorities require in the first place a salt-scaling test to be done, even though the exposure can be as severe as for offshore structures.
- Russian freeze-thaw classes F for concrete use a compressive strength as a material characterization. The other standards that set requirements for the lowest strength for concrete surface before it is exposed to first freezing (see **Table 5**), could supposedly consider strength as a performance criterion, but at an early stage.
- Most of the specifications that do not require analysis of hardened air-void structure, offer other methods to assess potential to frost resistance like pore protection factor or protective pore ratio (known as PF) [24,25], and permeability [45].

Table 6b provides an overview of test methods for freeze-thaw resistance, which is one of the most controversial topics of production and documentation of frost durable concrete.
Even though a great deal of work has been done with clear separation between the two main deterioration forms, surface scaling and internal cracking, the discussion about the relation to real exposure and acceptance criteria are found to often cause disagreement and difficulties. This is partly due to effects of preparation, curing and aging of specimens, to what extent both scaling and cracking occur in the tests, and, last but not least, the relation between the tests and the field performance/service life. Well-known examples and uncertainties are:

1. Improved salt scaling durability by accelerated carbonation in OPC concrete while for slag concrete it can worsen the performance in lab tests. Unclear how carbonation will contribute to field performance and to the service life of the structure from frost resistance perspective.
2. Concrete that will be deemed durable in a severe deicer salt scaling test CEN TS 12390-9 may not pass a severe internal cracking test such as ASTM C666, procedure A.
3. The uncertainty of how long an air void system will be protective when frozen and thawed with liquid at the surface, i.e. what happens if/when air voids get water-filled.
4. How low w/b-ratio is needed for different binders and frost tests to obtain frost durability without entrained air voids of a specific spacing factor under varying curing- and exposure conditions (see 4^{th} bullet point on page 18 on Russian old requirements for highest w/b-ratio of 0.41 for concrete without air entrainment).

The lack of understanding of how test methods work in such cases is a part of the challenge to find reliable specifications for concrete based on frost testing.

"Calibration" of frost test methods versus behavior of small specimens in field exposure stations has been done for many years, pointing to a probable reasonable connection between the European CEN/TS 12390-9 [67] salt-frost slab test and field exposure along heavily deicer exposed roads. However, there are some ageing-/microstructure issues and problems with excessive internal cracking compared to reinforced structures. Fast solutions to practical recommendations for a wide range of new binder types and exposed structures based on calibrated frost testing have therefore not been found yet. The time-consuming practice of field exposure stations and the transfer of experience from these to realistic acceptance criteria still needs updating for new binders.

D. Hooton [58] embraces salt-scaling standards in North America, concluding that:

- ASTM C 672 does not correlate well with field performance and appears to show fly ash and slag concrete at a disadvantage.
- Air-entrained concrete with up to 30% fly ash or 35% slag should be resistant to deicer salt scaling, provided that sufficient maturity is gained.
- The standard MTO LS-413 (almost the same as ASTM C672 but with mass loss measurements) is overly severe to SCM concretes unless additional maturity beyond the standard 14-day cure is attained prior to the test. However, in practice, there is no time to wait for concrete to mature for proving frost durability of a given structural element. Hence, SCMs are usually deemed unacceptable.

Some additional observations:

- ASTM C672 as a qualitative test requires an expert opinion on what the rate of scaling is. Table C1 (Appendix C1) shows a suggestion for more precise evaluation [58].
- Only Russian test standard [42] among all the other tests reviewed here comply with severe exposure, such as exposure to arctic and seawater, low-temperature frost cycles and exposed to chlorides surface. It is understood that GOST 10060 also allows user to choose between standard and accelerated freeze-thaw tests (see Appendix C2, Table C2.2).
- Russian tests do not offer any of the qualitative nor quantitative methods for evaluation of surface damage during freeze-thaw experiments, even though they put emphasis on following up surface scaling and cracking and that the test is to be interrupted when the surface is damaged enough.

VTT Technical Research Centre of Finland issued a comprehensive literature review of freeze-thaw testing in 2012 [80], where they also gave description of the test methods that had been withdrawn from use, for example, standard test method ASTM C671-94 for determining dilation caused by freezing-thawing cycles. However, conservative clients such as Exxon Mobil that build marine concrete structures still require using withdrawn ASTM C671 along with ASTMC666, procedure A [62].

Observation for laboratory works:

Studying mixing procedures for laboratories in Norway (SVV Method 411 in R210), it was discovered that ASTM C192/C192M (par. 8.1.2) suggests a completely different sequence of addition of part materials and a procedure for preparation of mixing equipment: “8.1.2. Prior to starting rotation of the mixer add the coarse aggregate, some of the mixing water, and the solution of admixture when required…. When feasible, disperse the admixture in the mixing water before addition. Start the mixer, then add the fine aggregate, cement, and water with the mixer running.” While in Norway, it starts with misting of the mixer and its parts, followed by sequential addition of sand, coarse aggregate, cementitious material; mixing water and admixtures in a given sequence of addition is added 60 seconds after the dry mixing.

Variability in the lab routines leads to additional complications in correlating the results between countries, and different sequences of admixture dosage (AEA then SP, SP then AEA, AEA and SP together) can largely affect the resulting air void system [75].

Table 7 shows a selection of requirements to frost durable concrete both from standards, recommendations, and from some major projects. It also gives understanding of how complete the reviewed documents are when it comes to the all-round requirements for concrete in a given exposure.

Examples of requirements from project specifications with brief descriptions for Øresund Bridge (Danmark/Sweden), Hebron and White Rose gravity base structures (Canada, Newfoundland) are presented in Appendix E.

Table 6a. Tests for frost durability – material characterization

Country	Standard	Material characterization	Performance testing
Norway	NS-EN 206:2013+NA:2014	Total air-content, compressive strength, w/c_{eff}	Salt scaling – Slab test acc. to NS-CEN/TS 12390-9:2006: Lab or pre-testing: M_{56} < 0,2 kg/m³ or M_{56} < 0,5kg/m³ and M_{56}/ M_{28} < 2.
	SVV Prosesskode 2	Total air-content, compressive strength, w/c_{eff}; Pore protection factor - PF method, method 426 [25] Water-to-cement ratio – microwave	Salt scaling – Slab test acc. to NS-CEN/TS 12390-9:2006 Lab or pre-testing: M_{56} < 0,2 kg/m³ or M_{56} < 0,5kg/m³ and M_{56}/ M_{28} < 2.
	NS-EN ISO 19903:2006	Total air-content, compressive strength, w/c_{eff}; Air-void system in acc. with NS-EN 480-11:2005	Internal cracking, ASTM C666, procedure A
Sweden	SS 137003:2015	Total air-content, compressive strength, w/c_{eff} Air content / air-void system ASTM C457/C457M	Frost resistance: XF2, XF4 – SS137244, method A XF3 – SS137244, method B
Denmark	HETEK committee, Danish Road Directorate	Pore protection factor (only exp. class 1)[1] Req. - Min. 25%	Salt scaling – "Borås method", SS 147244: Lab or pretesting: M_{56} < 0,2 kg/m³ or M_{56} < 0,5kg/m³ and M_{56}/ M_{28} < 2. In situ: M_{56} < 1,0 kg/m³ Internal cracking, ASTM C666-A (only exp. class 1) Lab.: $expansion_{300}$ < 0,05 % (0,1% - in-situ)
	DS 2426	Total air-content, compressive strength Air content / air-void system in acc. with ASTM C457/C457M	Internal cracking by ASTM C666 – Good
Finland	B4, Liite 3, SFS-EN 206-1	w/c (XF1, XF3), P - (XF1, XF3) and F(XF2, XF4) – values Protective pore ratio (similar to PF-method [25]): SFS 4475 (1980)	Salt scaling – Slab test acc. to CEN/TR 15177: Transit time ≥75% or relative flex. or splitting tensile strength ≥67% [80]
Germany	DIN 1045:2:2008-08	Total air-content, compressive strength, w/c_{eff} Water-to-cement ratio – microwave	Frost resistance and durability by DIN CEN/TS 12390-9:2006-08
	ZTV-ING		Frost resistance and durability by CDF test (BAW Code of practice [39])
Russia	SP 35.13330	Air content, compressive strength Concrete impermeability, GOST 12730.5-84	Frost resistance by GOST 10060 + Compressive strength
Canada	CSA A23.1-09-A23.2-09	Air content: The pressure method CSA A23.2-4C The volumetric method CSA A23.2-7C Compressive strength Air content / air-void system: ASTM C457/C457M (magnification 100…125) – cylindrical forms	Salt scaling (owner selects a method) - recommendations: ASTM C672/C672M BNQ NQ 2621-900 MTO LS-412
USA	ACI 201.2R-01, ACI 212.3R-10	Air content: The pressure method ASTM C231 The volumetric method – ASTM C173/C173M The gravimetric method (or the Unit weight test) – ASTM C138/138M Air content / air-void system ASTM C457/C457M	De-icer salt scaling ASTM C672
China	GB/T 50476	w/c, total air-content, compressive strength Air-void system (Spacing factor)	Frost resistance in acc. with GB/T 50082-2009

[1] Vuorinnen J., Om skyddsporförhållandet hos betong [About pore protection factor in concrete]. DBT publication nr. 22, Nordisk Workshop Beton & Frost, Køge, 1984

Table 6b. Tests for frost durability – freeze-thaw tests

Country	Type of frost resistance experiment		Proce-dure name	Samples			Freezing/thawing cycles		Test setup			Expression of test results				Exceptions / Limitations / Notes
	Scaling test	Internal cracking test		L x W x D, mm (Min surface area, m²)	Min. # of speci-mens per series	Curing plan	Duration of 1 cycle	Temp. range, C° (Where measured)	Test liquid / Setup	Freezing	Thawing	Parameter	Number of F/T cycles	Unit	Passing criteria	
Sweden, Europe	SS 137244 [30], CEN/TS 12390-9 [67]		Slab test or Borås method	150 x 150 x 50	4	1d - 20°C, 95%RH 6d - water, 20°C 21d - 20°C, 65%RH 3 d - 3mm deionized water on a test surface	24 h (16h/8h)	-18...-22 / +16...+24 (in the middle of test liquid above a specimen)	Method A [30] - 3%NaCl 3mm of FM on TS, one-sided Method B [30] – fresh water	Chamber controlled		Scaled material (gathered by brushing of loose material from TS)	56 (112 - with SF)	kg/m²	M_{56}<1 kg/m² M_{56}/ M_{28}<2	14±1 weeks in 65%RH, 20C deg for concrete with Slag content ≥ 35%
Sweden, Europe		SIS-CEN/TR 15177 [31]	Beam test	400 x 100 x 100	3	1d - 20°C, 95%RH 6d - sealed, 20°C 21d - water, 20°C	12 h (8h/4h)	-18...-22 / +22...+4 (center of a specimen)	Deionized water		in water +5...+21°C	Rel. Dyn.E-Modul	56	%		Visual assessment (cracks, scaling from aggregate particles, leakage of freezing medium) before the start and after the specified number of cycles should be reported
Sweden, Europe			Slab test	150 x 150 x 50	4	1d - 20°C, 95%RH 6d - water, 20°C 21d - 20°C, 65%RH 3 d - 3mm deionized water on a test surface	24 h (16h/8h)	-18...-22 / +16...+24	De-ionised water or 3%NaCl / 3mm of FM on TS	Chamber controlled		Length change Rel. Dyn.E-Modul	56	% %		
Sweden, Europe			CIF-test	150 x 150 x 70	5	1d - 20°C, 95%RH 6d - water, 20°C 21d - 20°C, 65%RH 7 d - test surface is dipped into 3%NaCl	12 h (7h/5h)	-19,5...-20,5 / +19...+21	3%NaCl / Inverted positioning with TS immersed in FM. 5 mm FM under TS	Chamber controlled		Length change Water uptake Rel. Dyn.E-Modul	56	%		
Germany, Europe	Bunke [68], CEN/TS 12390-9		Cube-Test	100 x 100 x 100	4	1d - 20°C, 95%RH 6d - water, 20°C 20d - 20°C, 65%RH 1d - submerged in the test liquid	24 h (16h/8h)	-13...-17 / +18...+22 (in the middle of reference specimen)	Submerged cubes in 3%NaCl, all sides	Chamber. Rapid thawing in 3 h from -15 to +20, then constant +20 for 5h		Scaled material (gathered by brushing of loose material from TS)	56 (100 - Bunke, DIN 1048)	%	M_{56} / M_0 < 3%	
Germany, Europe	RILEM TC 117-FDC/CDF [69], CEN/TS 12390-9		CF-/CDF-Test	150 x 140 x 50 (0,08)	5	1d - 20°C, 95%RH 6d - water, 20°C 21d - 20°C, 65%RH 7d - capillary suction of 5mm test liquid	12 h (7h/5h)	-19,5...-20,5 / +19...+21 (liquid-cooling under the container and test liquid)	One-sided, capillary suction of 5mm CF - deionized water CDF - 3% NaCl	Chamber controlled		Scaled material (gathered by ultrasonic bath)	14 (CDF) 28(CF)	kg/m2	M_{56}<1,5 kg/m² M_{56}/ M_{28}<3	

Country	Type of frost resistance experiment		Proce-dure name	Samples			Freezing/thawing cycles		Test setup			Expression of test results				Exceptions / Limitations / Notes
	Scaling test	Internal cracking test		L x W x D, mm (Min surface area, m²)	Min. # of speci-mens per series	Curing plan	Duration of 1 cycle	Temp. range, C° (Where measured)	Test liquid / Setup	Freezing	Thawing	Parameter	Number of F/T cycles	Unit	Passing criteria	
North America	ASTM C672 [13]			A x B x 75 (0,045)	2	1d - 23±2oC 13d - moist storage, 23±2oC, 95%RH 14d - 23±2oC, 45-55%RH	22-26 h (16-18h / 6-8h)	-15…-21 / +21…+25 (in the chamber)	ca 3,87%CaCl2 (4g of CaCl2(H2O)x for 100ml of solution) / 6mm of liquid on TS	air +21…+25oC, 45-55%RH		Rating of scaling, photo documentation	50	0-5 (no - severe scaling)		No thermal elements suggested. No prevention against evaporation. No side insulation. No conditioning before F-T experiments. Water is added instead to maintain 6 mm FM.
North America		ASTM C666 [61]	Proc. A	275…405 x 75…125 x 75…125	3	1d - 23±2oC 14d - water, 23±2oC From structure: 2d - saturated lime water, 23±2oC	2-5 h (50% / 50%)	-16…-20 / +2…+6 (center of the specimen)	Immediately after curing bring spec in temp -1…+2oC, make measurements and start cycling in water	water	water	Rel. Dyn.E-Modul and durability factor Length change Mass loss	300	%		
			Proc. B							air	water					
Canada, Ontario	MTO LS-412 [19]			300 x 300 x 75	2	1d - 23±2oC, 95%RH 13d - 23±2oC, 95%RH 14d - 23±2oC, 45-55%RH	22-26 h (16-18h / 6-8h)	-19,5…-20,7 / +19…+21	3%NaCl / 6mm of liquid on TS		air 23±2oC, 45-55%RH	Scaled material	50	kg/m2	<0,8 kg/m²	Wooden supports for air circulation and temperature distribution.
Canada, Quebec	BNQ NQ 2621-900 [18]					1d - 23±2°C 13d - moist storage, 23±2°C, 95%RH 14d - 23±2°C, 45-55%RH 7d – resaturation of TS with 3%NaCl	24 h (16±1h/8±1h)	-15…-21 / +5…+28 (at the interface of FM and TS)	3%NaCl		chamber controlled	Scaled material	56	kg/m2	<0,5 kg/m²	Recommended test by ICON/CANMET, Canada for concretes with SCM
China		GB/T 50082—2009	Slow FT	100 x 100 x 100	3	24d - climate room 4d - water storage	> 8 h (1,5-2…4h/4h)	-18…-20 / 18…20 (water temp)		In air	Water, temp more than 10C for first 30 min, then 18-20 C	Mass loss Compressive strength loss	min 25	%	not more than 5% not more than 25%	
China			Fast FT	100 x 100 x 400	3	24d - climate room 4d - water storage	2…4h (1…2h/1h…2h) Lowest temp. is held for max 10 min	-16…-20 / 3…7 (temp in the center of the sample)		In water	Water	Weight change Rel. Dyn. E-Modulus Basic frequency, ʃ	min 25	%	<5% E reduced less than to 60%	The box for FT - 500 x 115 x 115 F/T cycles cannot be stopped for longer than the duration of 2 cycles when samples are not kept frozen, and not more than 2 times, whether samples are frozen or not

Table 6b continued

Country	Type of frost resistance experiment		Proce-dure name	Samples			Freezing/thawing cycles		Test setup			Expression of test results				Exceptions / Limitations / Notes
	Scaling test	Internal cracking test		L x W x D, mm (Min surface area, m²)	Min. # of speci-mens per series	Curing plan	Duration of 1 cycle	Temp. range, C° (Where measured)	Test liquid / Setup	Freezing	Thawing	Parameter	Number of F/T cycles	Unit	Passing criteria	
China	GB/T 50082—2009		One-sided FT	150 x 110 x (70±2)	5	1d - 20±2°C 6d - water, 20±2oC (cutting) 21d - 20±2°C, 65±5%RH 7 d - resaturation in 3%NaCl (10±1 mm column), TS down	12 h (4h + 3h constant / 4h+1h constant)	-19…-21 / 20 cooling rate - 10 C /h (in the air)	3%NaCl / Inverted positioning with TS immersed in FM. 5 mm FM under TS	In a freezing medium, surrounding test dish with specimens. Chamber controlled		Water adsorption Scaled mass Rel. Dyn.E-Modulus, transition time	min 4		<1,5 kg/m² E reduced less than to 80%	CIF-cycle and type of test. Butyl tape is used for insulation of cube facets other than test surface. Sealing - 2-4 days before the end of conditioning Ultrasound test lasts 3 min for each measurement
Russia	GOST 10060-2012		First method F_1	100 x 100 x 100	12 (for FT) + 6 (reference)	1d - 20±2oC 27d - moist storage, 20±2°C, 95%RH 4d - gradual saturation in water	> 4 h (> 2,5h / >1,5…2,5h)	-16…-20 / +18…22 (air/water temperature)		In air	Water	Mean compressive strength loss Mass loss Visual check Rel. Dyn.E-Modulus, Transition time (UPV) Deformation	15…800	%	ca < 10% (< 15% for LWA) strength loss < 2% mass loss No cracks, chips, spalling of ribs < 25% loss of E-Modul < 15% reduction of transition time < 0,1% average deformation	All concretes, except for concretes used for road and airfield pavement in the presence of mineralized water
				150 x 150 x 150			> 6 h (> 3,5h / >2,5…3,5h)									
			Second method F_2	100 x 100 x 100		1d - 20±2oC 27d - moist storage, 20±2oC, 95%RH 4d - gradual saturation in 5%NaCl	> 4 h (> 2,5h / >1,5…2,5h)	-16…-20 / +18…22 (air / solution temperature)			In 5%NaCl, 18…20C		50…800			Concretes used for road and airfield pavement in the presence of mineralized water
				150 x 150 x 150			> 6 h (> 3,5h / >2,5…3,5h)									
			Second rapid	100 x 100 x 100			> 4 h (> 2,5h / >1,5…2,5h)						20…300			All concretes, except for concretes used for road and airfield pavement in the presence of mineralized water and light-weight concretes lighter than 1500 kg/m3
				150 x 150 x 150			> 6 h (> 3,5h / >2,5…3,5h)									
			Third rapid	100 x 100 x 100	6 (for FT) + 6 (reference)	1d - 20±2oC 27d - moist storage, 20±2oC, 95%RH 4d - gradual saturation in 5%NaCl	> 8,5…10,5 h (2,5…3h + 2,5…3h constant / 1…2h in chamber down to -10C, in solution - min 2,5h)	-48…-52 / 18…22 (air / solution temperature)		5%NaCl			2…35 (Structure type 1) 5…205 (Structure type 2)			Structure types: 1. All concretes, except for concretes used for road and airfield pavement in the presence of mineralized water and light-weight concretes not heavier than 1500 kg/m3; 2. Concretes used for road and airfield pavement in the presence of mineralized water
				150 x 150 x 150			> 9,5…11,5 h (2,5…3h + 2,5…3h constant / 1…2h in chamber down to -10C, in solution - min 3,5h)									

Table 6b continued

Table 7. Overview of requirements for frost durable concrete

Country / Organization, Project	Exposure class, area, decisive parameter	Material requirements: Max w/c (effective w/c)	Min cement (binder) content, kg/m³	Max (Min) SF content, %	Max FA content (Max Fa/C-ratio), % or kg/m³	Min air content in fresh / *hardened*** concrete (for aggregate D_{max}, mm), %	Max electric conductivity, Coulombs	Frost resistance, cycles (frost res. class or min. durability factor,%)	Quality of macro-porosity in hardened concrete: Min specific surface, mm²/mm³	Quality of macro-porosity in hardened concrete: Max spacing factor (single results) L, mm	Min concrete comp. strength (strength for cement), MPa or class	Water impermeability class (Durability class)	Laboratory tests: Porosity, air content, parameters of air voids (other requirements)	Laboratory tests: Frost resistance and other tests	Execution: Placing, Pumping, Curing
General Europe EN 206:2013	XF1	0,55	(300)*	11	(33)						C30/37				
	XF2	0,55	(300)*	11	(33)	4					C25/30				
	XF3	0,50	(320)*	11	(33)	4					C30/37				
	XF4	0,45	(340)*	11	(33)	4					C30/37				
Norway NS-EN 206:2013 +NA:2014	XF1	0,60	(250)*		(35)							M60			
	XF1	0,45	(300)*		(35)							M45			
	XF1	0,40	(330)*	(6)	(35)							M40			
	XF2, XF3	0,45	(300)*		(35)	4						MF45			
	XF4	0,40	(330)*	(6)	(35)	4						MF40			
Norway NS-EN ISO 19903:2006	Very severe, splash zone	0,40	400	10	35	4,0 (40) 5,0 (20)		$M_{s,56}\leq$ 0,50kg/m²	25	0,25	40				Max vertical drop – 2,0m Protection from the ingress of external water for min 7 days after casting
Norway Road Authorities Prosesskode 2	SV-Standard concrete	0,40	(350_{eff})	3…5 class 1	30 – class A	3…6% for B45 2…5% for over B45[1]				0,2	(42,5… 52,5)	(MF40 or M40)		NS-CEN/TS 12390-9 – F/T, slab test EN 480-11	

Country / Organization, Project	Exposure class, area, decisive parameter	Material requirements: Max w/c (effective w/c)	Min cement (binder) content, kg/m³	Max (Min) SF content, %	Max FA content (Max Fa/C-ratio), % or kg/m³	Min air content in fresh / *hardened*** concrete (for aggregate D_{max}, mm), %	Max electric conductivity, Coulombs	Frost resistance, cycles (frost res. class or min. durability factor,%)	Quality of macro-porosity in hardened concrete: Min specific surface, mm²/mm³	Quality of macro-porosity in hardened concrete: Max spacing factor (single results) L, mm	Min concrete comp. strength (strength for cement), MPa or class	Water impermeability class (Durability class)	Laboratory tests: Porosity, air content, parameters of air voids (other requirements)	Laboratory tests: Frost resistance and other tests	Execution: Placing, Pumping, Curing
Sweden SS137003: 2015	XF1	0,60	(200)*	10	(35)						(42,5)				
	XF2	0,45	(200)*	10	(35)	4,0 (>16) 4,5 (>8; ≤16)					(42,5)			SS 137244, method A – with 3%NaCl	
	XF3	0,46	(200)*	5	(35)	5,0 (≤8)					(42,5)			SS 137244, method B – with fresh water	
	XF4	0,45	(200)*	5	(20)						(42,5)			SS 137244, method A – with 3%NaCl	
Denmark/ Sweden ASO Group, Skanska, Øresund Bridge [77,79]	Splash zone -3.0 to +6.0 m	0,42	270 (340)	6 (2)	20	4			25		45		(max Cl content 0,1% of powder content. Na_2O_{eq}≤3.0kg/m³ with a mortar content of 60 vol.% Cores for air content drilled every 5000 m³)	SS 137244, method A – with 3%NaCl Dilation method. Water saturated concrete should withstand 1 cycle – no cracks allowed	Avoid exposure to chlorides before concrete is 1 year old
Denmark DS 2426	XF1 (M - moderate)	0,55	150	11	35 (33)	4,5					(42,5) C25/30				
	XF2 (A - aggressive)	0,45	150 (375)	11	20 (33)	4,5/3,5**				0,2	(42,5) C35/45		Air void analysis DS/EN 480-11 2 samples 100x150mm2 with min age of 7 days	SS 137244, method A – with 3%NaCl	
	XF3 (A - aggressive)	0,45	150 (375)	11	20 (33)	4,5/3,5**				0,2	(42,5) C35/45				
	XF4 (E - extra aggressive)	0,40	150 (375)	11	20 (33)	4,5/3,5**				0,2	(42,5) C40/50				

Table 7 continued

Country / Organization, Project	**Exposure class, area, decisive parameter**	Material requirements											Laboratory tests		Execution
		Max w/c (effective w/c)	Min cement (binder) content, kg/m³	Max (Min) SF content, %	Max FA content (Max Fa/C-ratio), % or kg/m³	Min air content in fresh / *hardened*** concrete (for aggregate D_{max}, mm), %	Max electric conductivity, Coulombs	Frost resistance, cycles (frost res. class or min. durability factor,%)	Quality of macro-porosity in hardened concrete: Min specific surface, mm²/mm³	Quality of macro-porosity in hardened concrete: Max spacing factor (single results) L, mm	Min concrete comp. strength (strength for cement), MPa or class	Water impermeability class (Durability class)	Porosity, air content, parameters of air voids (other requirements)	Frost resistance and other tests	Placing, Pumping, Curing
Finland SFS-EN 206-1, liite 3	XF1	0,60	270	11	(30)			(F = 1,0)							
	XF2			7	(30)			(P = 25)							
	XF3	0,50	300	11	(30)			(F = 1,5)							
	XF4			7	(30)			(P = 40)							
Germany ZTV-ING	XF2, tunnel walls (airborne De-icer)	0,50	(300)	10	80 kg/m³	4,0…6,0(16)[3]					C30/37				
	XF4, horizontal surfaces (snow, De-icer)	0,50			[4]			$M_{s,28} \leq 1,50$ kg/m²			$C25/30_{28}$			BAW Code of practice	
Germany DIN 1045-2:2008-08	XF1	0,60	280 (270)								C25/30				
	XF2	0,55[5] / 0,50[5]	300 / 320[5] (270[5])								C25/30 or C35/45[6]			RILEM TC 117-FDC/CDF	
	XF3	0,55 / 0,50	300 / 320[5] (270)								C25/30 or C35/45[6]				
	XF4	0,50	320 (270[5])								C30/37			RILEM TC 117-FDC/CDF	
Canada KKC, Hebron GBS Project	Splash zone	0,36	(400)	8	15	5,0…8,0 (22)	1000	500 (90%)		0,25 (0,30)	B55 after 56 days		ASTM C457	NS 3473 – comp. strength ASTM C1202 – El. Conductivity NT Build 443 – Chloride diffusion ASTM C666, proc. A – rapid F/T	Max vertical drop – 1,5m Min 7d of moist curing to gain min. 70% strength Max cooling rate for concrete surface – 20°C per 24h.

Table 7 continued

Country / Organization, Project	Exposure class, area, decisive parameter	Material requirements											Laboratory tests		Execution
		Max w/c (effective w/c)	Min cement (binder) content, kg/m³	Max (Min) SF content, %	Max FA content (Max Fa/C-ratio), % or kg/m³	Min air content in fresh / *hardened*** concrete (for aggregate D_{max}, mm), %	Max electric conductivity, Coulombs	Frost resistance, cycles (frost res. class or min. durability factor,%)	Quality of macro-porosity in hardened concrete: Min specific surface, mm²/mm³	Quality of macro-porosity in hardened concrete: Max spacing factor (single results) L, mm	Min concrete comp. strength (strength for cement), MPa or class	Water impermeability class (Durability class)	Porosity, air content, parameters of air voids (other requirements)	Frost resistance and other tests	Placing, Pumping, Curing
Canada Confederation bridge [78]	Class A concrete Main piers and T-beams	0,34	416	7,5	-	6	300_{28d}				82_{28d}				Target slump – 200mm
	Class C concrete Massive foundations	0,37	285	7,5	32	7	420_{28d}				50_{28d} 76_{91d}				Target slump – 185 mm
USA Portland Cement Association	De-icer exposed HPC for bridges, severe exposure	0,45	(335)	10	25	6,0 (16-22)[2]	1500	(90) Visual rating 1, scaling	24	0,2			ASTM C457	ASTM C666, proc.A ASTM C672 ASTM C1202	Curing begins within 15 min after finishing Wet curing for 7 days and until comp. strength of 22 MPa is reached
USA ACI 201.2 ACI 212.3R ASTM C457	Severe exposure	0,45				4,5…7,5 (25,4)			24	0,2					
USA ACI 318-14	F3, De-icing salt, saturation, reinforced concrete	0,4		10	35	4,5…7,5 (25,4)					34,5				
Russia GOST 26633-2015	Road surface layer, bridges	0,45				5,0…7,0		($F_2$200) GOST 10060			B30	W8			
Russia GOST 31384-2017	XF1	0,55	300								B20				
	XF2	0,55	300			4					B35				
	XF3	0,50	320			4					B25				
	XF4	0,45	340			4					B35				

Table 7 continued

Country / Organization, Project	Exposure class, area, decisive parameter	Material requirements											Laboratory tests		Execution
		Max w/c (effective w/c)	Min cement (binder) content, kg/m^3	Max (Min) SF content, %	Max FA content (Max Fa/C-ratio), % or kg/m^3	Min air content in fresh / *hardened*** concrete (for aggregate D_{max}, mm), %	Max electric conductivity, Coulombs	Frost resistance, cycles (frost res. class or min. durability factor,%)	Quality of macro-porosity in hardened concrete: Min specific surface, mm^2/mm^3	Quality of macro-porosity in hardened concrete: Max spacing factor (single results) L, mm	Min concrete comp. strength (strength for cement), MPa or class	Water impermeability class (Durability class)	Porosity, air content, parameters of air voids (other requirements)	Frost resistance and other tests	Placing, Pumping, Curing
Russia Kværner, Sakhalin I GBS Project	Shaft concrete / splash zone	0,4	360	8	-	5,0…8,0 (20)	1000	($F_2$500) GOST 10060	25	0,25	C70/85 (52,5N)	W12	EN 12350-7 – air content NS-EN 480-11 – Air quality of hardened concrete	EN 206-1 – comp. strength GOST 12730.5 - Permeability GOST 10060 – rapid freeze-thaw tests ASTM C666, proc. A – rapid F/T	Max vertical drop – 1,5m. Min 3d of moist curing. Max cooling rate for concrete surface – 20ºC per 24h. Exposure to F/T when min 80% of strength is attained

Table 7 continued

Note: **(+)** means that a given standard uses a material requirement, written in **(**parentheses**)** in the table heading, f. ex. SS 137003:2015 specifies requirements for effective w/c, while ACI – for max acceptable w/c.

[1] It serves as a requirement for frost resistance, it the last is not documented by any other means

[2] Based on criteria of required 9% air in the mortar phase of concrete

[3] Air content varies from stiff (4,5±0,5%) to flowable (5,5±0,5%) consistency within one aggregate size

[4] FA use in XF4 concrete is not permitted unless agreed separately with the customer

[5] Additions, excluding fly ash, may not be taken into account for the calculation of water-cement ratio and min. cement content

[6] For slow hardening concretes

[7] 0,55 for binders with min 80% PC-clinker

* Effective binder - Ceff =C+∑ki×pi, where Pi - puzzolana (FA, SF, GBFS)

6 Conclusive remarks and future work

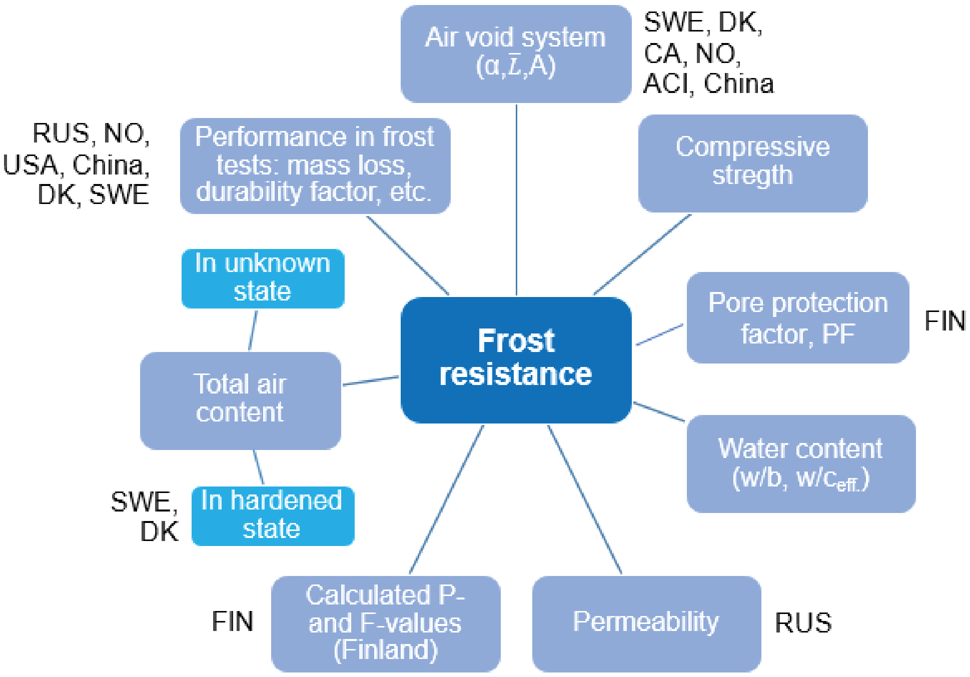


Figure 2. The meaning of frost resistance in different countries

If one should list down the requirements for concrete in a certain exposure from material requirements and testing to execution, quality assurance and handover, the results in different countries will vary in parameters, numbers and level of detailing.

Concerning material requirements, Figure 2 shows how variable the definition of frost resistant concrete can be, depending on which country one is going to design and build the structure in. As for execution requirements, it starts out with uncertainties about mixing procedures, especially when concrete contains SCM. Then there come requirements to transportation and delivery, which we found no description about in European documents. Another question is how to ensure that concrete put into the structure is of right quality, where to measure air and when, what is the requirement to concrete temperature (Dodson[3] stressed that air content is very dependant on fresh concrete temperature).

Practicalities that affect the quality of end product like placing, finishing and curing surprisingly are rarely described. For example, in the USA level of detailing in the regulatory documents allows people at supervisor level to understand how a task before him/her should be tackled.
In Norway, the system of standards and regulations is mainly oriented for a managerial level, providing only general information used as a tool to control processes. The system holds the management responsible by requiring a “Central approval” that is given based on proof of competence (see also the Norwegian hierarchy of acts and standards [76] in Appendix D). The Contractor is approved by the

[3] Dodson, Vance H. “Concrete admixtures”, Chapter 6: “Air entraining admixtures”, Van Nostrand Reinhold, New York, ISBN 0-442-00149-5 (1990)

Control Council for Concrete [kontrollbetong.no], The Directorate of Building Quality [dibk.no] or some other deputy or nominee for the particular field of work.
This difference between the end users of requirements can be an obstacle for international teams and projects.

Kukko and Kuosa [74] in 1999 reviewed the standards that define the requirements for frost resistant concrete in North America (Canada, USA) and Europe (Finland, Denmark, Norway, Sweden, Germany) with a slight focus on freezing and thawing without de-icing salts. At that time, they concluded that variations in exposure classification and frost resistance requirements were considerable. Today, 20 years after that review, we could stand by every word of their conclusion. However, there has been a positive change towards unifying environment classification in Europe, and that one day will make European (and maybe Russian) contractors speak “the same language”.

As per today’s knowledge, frost salt-scaling resistance of concrete has a direct relationship with its air-void system, the formation of which an air-entraining agent is responsible for. Contractors should always perform a certain number of trial batches and also use mockups to verify that the ingredients and procedures used would result in satisfactory and reproducible air-void system in the concrete as placed [57]. It turns out there is a lack of information on how to combine chemical admixtures in a given binder system [75]. Therefore, concerning frost resistance, the standard requirements for chemical admixtures and practice of combining them in the industry requires a thorough revision.

The present report is published in Open Access as a DaCS project report.

The document will also serve as a contribution in a rather major future literature review by newly (October 2018) established RILEM Technical Committee FTC[4]: Durability and service life of concrete under the influence of freeze-thaw cycles combined with chloride penetration; Cluster B.

[4] Chair: Prof. Dr. Dr.-Ing h.c. Folker H. Wittmann. Deputy chair: Dr. Peng Zhang

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Appendices

Appendix A. Canadian standard requirements for concrete in freeze-thaw exposure conditions

Table A1. Requirements for concrete, exposed to F/T, given by CSA A23.1-04 – Concrete Materials and Methods of Concrete Construction, Canadian Standards Association International

Reference		A-1	A-2	A-3	F-1	F-2	C-2	C-1	C-XL
Table 1, s.121	Exposure classes	Manure and/or silage gases exposure with/without F/T conditions			F/T exposure without chlorides		Exposure to chlorides with/without F/T conditions		
4.1.1.3, Tables 2 and 4	Description	Structurally reinforced concrete exposed to severe manure and/or silage gases, with or without freeze-thaw exposure. Concrete exposed to the vapour above municipal sewage or industrial effluent, where hydrogen sulphide gas might be generated. Examples: reinforced beams, slabs, and columns over manure pits and silos, canals, and pig slats; and access holes, enclosed chambers, and pipes that are partially filled with effluents.	Structurally reinforced concrete exposed to moderate to severe manure and/or silage gases and liquids, with or without freeze-thaw exposure. Examples: reinforced walls in exterior manure tanks, silos and feed bunkers, and exterior slabs.	Structurally reinforced concrete exposed to moderate to severe manure and/or silage gases and liquids, with or without freeze-thaw exposure in a continuously submerged condition. Concrete continuously submerged in municipal or industrial effluents. Examples: interior gutter walls, beams, slabs, and columns; sewage pipes that are continuously full (e.g., forcemains); and submerged portions of sewage treatment structures.	Concrete exposed to freezing and thawing in a saturated condition, but not to chlorides. Examples: pool decks, patios, tennis courts, freshwater pools, and freshwater control structures.	Concrete in an unsaturated condition exposed to freezing and thawing, but not to chlorides. Examples: exterior walls and columns.	Non-structurally reinforced (i.e., plain) concrete exposed to chlorides and freezing and thawing. Examples: garage floors, porches, steps, pavements, sidewalks, curbs, and gutters.	Structurally reinforced concrete exposed to chlorides with or without freezing and thawing conditions. Examples: bridge decks, parking decks and ramps, portions of marine structures located within the tidal and splash zones, concrete exposed to seawater spray, and salt water pools.	Structurally reinforced concrete exposed to chlorides or other severe environments with or without freezing and thawing conditions, with higher durability performance expectations than the C-1, A-1, or S-1 classes.
	Concrete requirements								
Table 2, p.122	Max w/c-ratio[1]	0,4	0,45	0,5	0,5	0,55	0,45	0,4[3]	0,37
Table 2, p.122	Min strength[2], Mpa / age, d	35/28	32/28	30/28	30/28	25/28	32/28	35/28	50/56
	Air content category	1	1	2	1	2	1	1	1
Table 4, p.124	Air content, max agg size 22mm (14-20)	5-8%	5-8%	4-7%	5-8%	4-7%	5-8%	5-8%	5-8%

Table A1 continued

Reference	Concrete requirements	A-1	A-2	A-3	F-1	F-2	C-2	C-1	C-XL
Table 2, p.122	Curing type, normal concrete	2	2	1	2	1	1	2	3
Table 20, p. 134	Description of curing procedure	Additional curing. 7d at +10C	Additional curing. 7d at +10C	Basic curing. 3d at +10C	Additional curing. 7d at +10C	Basic curing. 3d at +10C	Basic curing. 3d at +10C	Additional curing. 7d at +10C	Extended wet curing 7d at +10C
4.3.3.3, a	Average air-void spacing factor / max value, µm	230/260	230/260	230/260	230/260	230/260	230/260	230/260	230/260
4.3.3.3, b	Average air-void spacing factor / max value for w/c lower than 0,36, µm	250/300	250/300	250/300	250/300	250/300	250/300	250/300	250/300
4.3.3.3, note 2	Target max spacing factor, considering large variations in ASTM C457/C457M, µm	170	170	170	170	170	170	170	170

[1] Clause 8.7.3. The maximum water-to-cementing materials ratio of the HVSCM-1 concrete, exposed to freezing and thawing, shall be reduced by 0.05 in all exposure classes.

[2] Clause 8.7.3. Minimum strength requirement for HVSCM-1 concrete (min 40%FA) should be given for 56 days

[3] Clause 8.7.3. For HVSCM-1 concrete max w/c should be 0,35.

Appendix B1. Chinese requirements for frost durable concrete

- Air-void characteristics

Table B1.1. Chinese standard requirements to air-void parameters

Conditions / Air content / Maximum aggregate (mm)	High water content	Moderate water content	With salt/De-icer
10	6.5	5.5	6.5
15	6.5	5.0	6.5
25	6.0	4.5	6.0
40	5.5	4.0	5.5
Spacing factor (pac	250	300	200

- Water / cement ratio, strength and other parameters

Table B1.2. Chinese standard design requirements for concrete

Service life / Environment			100 years			50 years			30 years		
			Comp. strength grade	Max. w/c	Min. cover	Comp. strength grade	Max. w/c	Min. cover	Comp. strength grade	Max. w/c	Min. cover
Slab, wall, other surface structural elements	II-C, no salt		C45 ≥C50 Ca35	0.40 0.36 0.50	35 30 35	C45 ≥C50 Ca35	0.40 0.36 0.55	30 25 30	C40 ≥C45 Ca30	0.45 0.40 0.55	30 25 25
	II-D	No salt	Ca40	0.45	35	Ca35	0.50	35	Ca35	0.50	30
		Salt									
	II-E, salt		Ca45	0.40		Ca40	0.45		Ca40	0.45	
Beam, column, other strip structural elements	II-C, no salt		C45 ≥C50 Ca35	0.40 0.36 0.50	40 35 35	C45 ≥C50 Ca35	0.40 0.36 0.55	35 30 35	C40 ≥C45 Ca30	0.45 0.40 0.55	35 30 30
	II-D	No salt	Ca40	0.45	40	Ca35	0.50	40	Ca35	0.50	35
		Salt									
	II-E, salt		Ca45	0.40		Ca40	0.45		Ca40	0.45	

Requirements for concrete cover in the presence of De-icing salts belong to a separate durability / exposure class IV.

Appendix B2. Finnish standard. Definition of F- and P-values

$$\boldsymbol{F} = \frac{1}{-4{,}0 + 7{,}2 \times \frac{(\frac{w}{c})^{0{,}45}}{(a-1)^{0{,}14}}} \quad (1)$$

$$\boldsymbol{P} = \frac{46 \times \boldsymbol{k_{jh}} \times \boldsymbol{k_s}}{\frac{10 \times (\boldsymbol{WAS_{RED}})^{1{,}20}}{\sqrt{a}} - 1} \quad (2)$$

where

$\boldsymbol{k_{jh}}$	curing factor
$\boldsymbol{k_s}$	binder factor
$\boldsymbol{WAS_{RED}}$	reduced water-air-binder ratio
a	total air content, %

$$\boldsymbol{k_{jh}} = 0{,}85 + 0{,}17 \times \log_{10}(t_{jh}) \quad (3)$$

where

t_{jh}	curing time, days

$$\boldsymbol{k_s} = 1 - \left(\frac{Q_{vesi}}{\boldsymbol{Q_{sid}}}\right)^{1{,}5} \times (0{,}05 \times sil + 0{,}02 \times kuona + 0{,}01 \times lt) \quad (4)$$

where

Q_{vesi}	effective water content, kg/m³
$\boldsymbol{Q_{sid}}$	effective binder content, kg/m³
$sil, kuona, lt$	silica fume (SF/b), slag (BFS/b), fly ash (FA/b) contents in binder respectively, %

$$\boldsymbol{Q_{sid}} = Q_{sem} + 2{,}0 \times Q_{sil} + 0{,}8 \times Q_{kuona} + 0{,}4 \times Q_{lt} \quad (5)$$

where

$Q_{sem}, Q_{sil}, Q_{kuona}, Q_{lt}$	cement, silica fume, slag and fly ash contents respectively, kg/m³

$$\boldsymbol{WAS_{RED}} = \frac{Q_{vesi} + 10 \times (a-2)}{\boldsymbol{Q_{sid}}} \quad (6)$$

Appendix C1. Tests of frost durability. Interpretation of scaling rating in Canada

Equivalency Chart Relating Visual Scaling to Mass Loss			
	CSA Scaling Rating	Mass Loss Range g/m2	Visual Characteristics of the scaling surface
Equivalency Rating	0	0 - 50	No significant scaling observed
	1	51 - 210	Very slight scaling 3mm (1/8") depth, max, no coarse aggregate visible and no popouts present
	2A	211 - 500	Slight to moderate scaling and/or presence of a few popouts
	2B	211-500	Slight to moderate scaling and/or presence of many popouts
	3	501 - 1300	Moderate scaling of mortar with some exposed coarse aggregate
	4	1301 - 2100	Moderate to severe scaling: the coarse aggregate is clearly exposed and there is significant scaling of the surface mortar
	5	>2100	Severe scaling: coarse aggregates are visible over the entire surface

Table C1. Recommendation for transition from qualitative rating of scaling to quantitative mass loss. [Reprinted from 58, page 44]

Figure above shows an attempt of how to interpret CSA's scaling rating in physical mass loss, g/m^2. It, however, remains unclear how rating #1 with 51-210 g/m^2 was translated to 3mm scaling depth in CSA A23.2-22C Scaling Test Visual Ratings, because, following a simple calculation of concrete with density of 2400 kg/m^3, we get 7.2 kg/m^2 of mass loss corresponding to 3mm.

Appendix C2. Requirements and tests of frost durability. The relationship between different frost tests in Russia

Table C2.1. Requirements for frost resistance of concrete structures, working in alternating temperatures (Translated and reproduced from GOST 31384-2017, Table E.1, page 36)

Exposure	Calculated ambient winter temperature[2], °C	Frost resistance class requirement[1]
XF2	Below -40	$F_1$300
Moderate water saturation, atmospheric action	-20 … -40	$F_1$200
	-5 … -20	$F_1$150
	Above -5	$F_1$100
XF3	Below -40	$F_1$400
High water saturation in fresh water	-20 … -40	$F_1$300
	-5 … -20	$F_1$200
	Above -5	$F_1$150
XF4	Below -40	$F_2$450
High water saturation in presence of seawater, de-icing agent, mineralized or subpermafrost water	-20 … -40	$F_2$300
	-5 … -20	$F_2$200
	Above -5	$F_2$100

[1] F_1 and F_2 – minimum frost resistance class by first and second respectively basic method (see Table C2.2 below)

[2] Calculated on the basis of average ambient temperature of the coldest five-day stretch (with max variability 8%)

Notes (selected):

2. For structures with variable degree of saturation for different parts, for example foundations for ETL, columns, etc., frost resistance class is based on the most severely exposed element.
3. Frost resistance classes for structures of water supply systems, bridges and pipes, airfields, roadways and hydraulic structures should be stipulated by country codes and standards of the receiving party. In Russia it is:
   - SP 31.13330.2012 SNiP 2.04.02-84* «Water supply. Outdoor systems and structures»,
   - SP 35.13330.2011 SNiP 2.05.03-84 «Bridges and pipelines»,
   - SP 121.13330.2012 SNiP 32-03-96 «Airfields»,
   - SP 34.13330.2012 SNiP 2.05.02-85* «Automobile roads»,
   - SP 41.13330.2012 SNiP 2.06.08-84 «Concrete and reinforced concrete hydraulic structures».

Table C2.2. The relationship between a number of test cycles and a grade for frost resistance of concrete
(Translated and reproduced from GOST 10060-2012, Table 4, page 5)

Methods		Types of concrete	Grades for frost resistance of concrete $\mathbf{F_1}$ or $\mathbf{F_2}$ and a number of cycles when intrermediate tests are performed (abobe the line) and a number of cycles corresponding to a grade for frost resistance of concrete (under the line)												
Basic	First	All concretes, except for concretes used for road and airfield pavement in the presence of mineralized water[1]	$F_1$25	$F_1$35	$F_1$50	$F_1$75	$F_1$100	$F_1$150	$F_1$200	$F_1$300	$F_1$400	$F_1$500	$F_1$600	$F_1$800	$F_1$1000
			15/25	25/35	35/50	50/75	75/100	100/150	150/200	200/300	300/400	400/500	500/600	600/800	800/1000
	Second	Concretes used for road and airfield pavement in the presence of mineralized water[1]	-	-	-	$F_2$75	$F_2$100	$F_2$150	$F_2$200	$F_2$300	$F_2$400	$F_2$500	$F_2$600	$F_2$800	$F_2$1000
			-	-	-	50/75	75/100	100/150	150/200	200/300	300/400	400/500	500/600	600/800	800/1000
Accelerated	Second	All concretes, except for concretes used for road and airfield pavement in the presence of mineralized water[1] and light-weight concretes lighter than 1500 kg/m³	-	-	$F_1$50	$F_1$75	$F_1$100	$F_1$150	$F_1$200	$F_1$300	$F_1$400	$F_1$500	$F_1$600	$F_1$800	$F_1$1000
			-	-	-	—/8	—/13	—/20	20/30	30/45	45/75	75/110	110/150	200/300	300/450
	Third	All concretes, except for concretes used for road and airfield pavement in the presence of mineralized water[1] and light-weight concretes not heavier than 1500 kg/m³	-	-	-	$F_1$75	$F_1$100	$F_1$150	$F_1$200	$F_1$300	$F_1$400	$F_1$500	$F_1$600	$F_1$800	$F_1$1000
			-	-	-	2	3	4	5	8	12	15	19	27	35
		Concretes used for road and airfield pavement in the presence of mineralized water[1]	-	-	-	-	$F_2$100	$F_2$150	$F_2$200	$F_2$300	$F_2$400	$F_2$500	$F_2$600	$F_2$800	$F_2$1000
			-	-	-	-	5	10	20	37	55	80	105	155	205

[1] Seawater is one of the types of mineralized water

Appendix D. Standards hierarchy in Norway

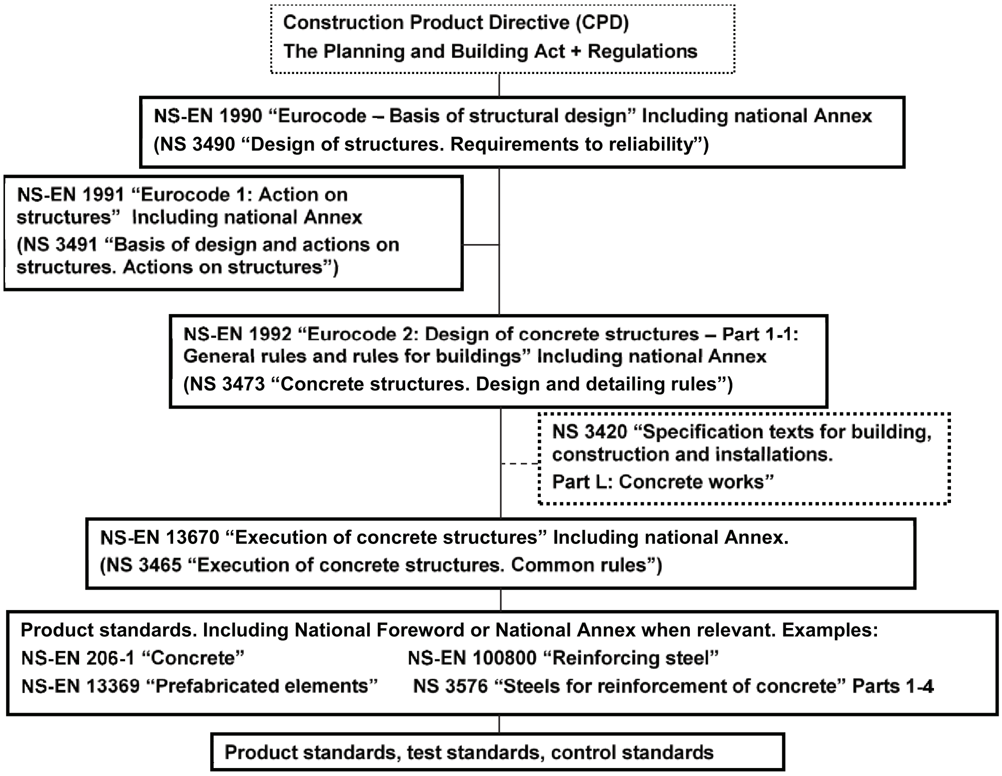


Figure D1. Hierarchy of acts and standards in Norway as it is autumn 2010. (Reprinted from [76], figure 2.1, page 2-4)

Appendix E. Examples of requirements for frost durable concrete

E1. Requirements to concrete of Confederation Bridge

Figure E1. Confederation Bridge (retrieved from https://www.vinci-construction-projets.com/en/realisations/confederation-bridge/)

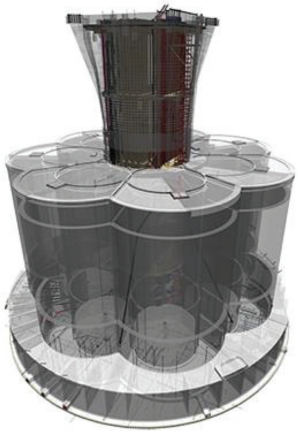
12.9 km long Confederation Bridge upon completion in 1997 connected Prince Edward Island with the main land of Canada, across the Northumberland Strait. It consists of 44 main spans of 250 m length each, massive main pier shaft and foundation elements fabricated on land.

Class A concrete (see Table E1) required a minimum of 55 MPa compressive strength and a maximum of 1000 coulombs chloride permeability (ASTM C 1202 test) at 28 days. Piers with an abrasion resistant ice shield required 80 MPa concrete. Class C concrete was used for pier foundations and some mass-concrete sections. It contained ca 32% fly ash as a cement replacement material. The requirements for Class C concrete were 30 MPa and 40 MPa minimum compressive strength at 28 and 90 days, respectively. All concrete also contained 7.5% silica fume by mass of the total binder content.

Table E1. Mix design and requirements for concrete of Confederation Bridge

Mix Proportions, kg/m^3	Class A Concrete for Main Piers and T-beams	Class C Concrete for Massive Foundations	Abrasion-resistant Ice Shield Concrete
Portland cement	416	285	478
Silica fume	34	22	42
Fly ash, Class F	-	133	60
Fine aggregate	737	744	650
Coarse aggregate	1030	1054	980
Water	153	159	142
Superplasticizer	3	2	6
W/cm	0.34	0.37	0.25
Properties			
Entrained air, %	6.1	7.0	-
Slump, mm	200	185	-
Compressive Strength, MPa			
1-day	35	9.7	-
3 days	52	27.4	-
28 days	82	50.0	100
91 days	-	76.0	-
Rapid chloride permeability, Coulombs (AASHTO T277)			
28 days	300	420	-
90 days	-	-	-

E2. Requirements to concrete of Hebron and White Rose gravity base structures



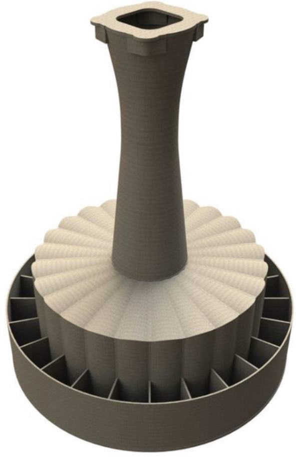


Figure E2. Offshore gravity base concrete structures:

- to the left - Hebron GBS (retrieved from https://www.kvaerner.com/Products/Concrete-structures-for-offshore-platforms/Hebron-GBS-Project/)
- to the right - West White Rose GBS (retrieved from http://westwhiteroseproject.ca/)

Table E2.1. Key parameters for the gravity base structures

Key parameters	Hebron GBS	White Rose GBS
Water depth (mean sea level)	93 m	120
Height of GBS	120 m	145 m
Diameter of GBS base	130 m	122 m
Concrete volume	130 000 m3	76 000 m3
Rebar	40 000 t (325kg/m3)	
Post tensioning steel	3 400 t	
Mechanical outfitting	5 500 t	
Completion	2016	2022

Note: Sources of information: https://www.kvaerner.com/, http://westwhiteroseproject.ca/, http://wwrp.huskyenergy.com/

Table E2.2. Requirements for concrete and prequalification tests

Parameters	Hebron GBS	White Rose GBS
Concrete grade	B65, NS3473	60MPa (28d shaft), EN1992
Exposure class	-	Class C-1 (Shaft), Class C-3 (subm.), CAN/CSA A23.1
Chloride diffusion coefficient	<4.0·10^{-12}m^2/s	
Electric conductivity	< 1500 Coloumbs (submerged) < 1000 Coloumbs (splash), ASTM C1202	< 1500 Coloumbs (splash)
Freezing and thawing	500 cycles, ASTM C666 (Splash)	CAN/CSA A23.2-24A
Air-void system	Spacing factor < 0.25mm ASTM C457	Spacing factor < 0.23mm (splash) Specific surface > 25 mm^{-1}
W/c-ratio	< 0.40	< 0.40 shaft, < 0.45 submerged
Max chloride content	0.10%	0.10%
Cement content per m^3	Min 400 kg (shaft), min 340 kg (submerged)	400-475 kg (shaft) 360-450 kg (submerged)
Abrasion resistance	-	< 10mm loss
Air content, splash zone	5-8%	5-8%
Dmax aggregate	20mm	
Cementitious materials	Cement: C_3A 5-10% Fly ash: type F (low carbon) – max 30% replacement Silica fume: max 8%, SS 15-30 m^2/g, SiO_2 > 85%	Cement: C_3A 5-10% Fly ash: type F (low carbon) – max 35% replacement Silica fume: 5-10% (only shaft) CAN/CSA A23.1
Concrete temperature	Max peak - 70 °C Thermal gradient over 300mm – 20°C Cooling rate - 20°C per 24h	Max peak - 75 °C Thermal gradient over cross section – 20°C Cooling rate - 20°C per 24h

Paper

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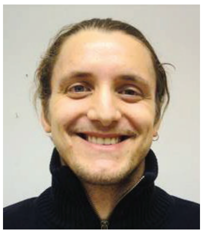
Effect of AEA-SP Dosage Sequence on Air Entrainment in FA Concrete



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ABSTRACT
Laboratory measurements show that varying the dosage sequence of air-entraining agent and co-polymer in the mix (SP added before, after or together with AEA) greatly affects air entrainment in fresh and hardened fly ash concrete. Image analysis shows a somewhat lower specific surface when SP is added together with AEA. Foam Index measurements on the same binder materials, admixtures, and dosage sequences were therefore found less useful for studying the effect of admixture combinations. Obtaining a certain air content using the experience with AEA-SP dosage was found to be an untrivial task if there is a lack of parameter control. Finally, examples of successful mixing procedure for air entrainment in a series of high-volume fly ash concrete are shown.

Keywords: Air entrainment, Dosage sequence, Fly ash, Admixtures

1. INTRODUCTION

Most of the studies on air entrainment and air-void stability in fly ash (FA) concrete focus either on the effectiveness of certain admixtures or on the influence of different types of FA. And there is a general agreement on that properly air-entrained FA concrete simply requires a higher dosage of AEA to compensate for the loss of active ingredient to unburned carbon in FA. However, thus far, the production of frost durable FA concrete with a stable and protective air void system has still proven to be difficult. The problem has been ascribed to the variable carbon content in the fly ash causing variations in the required dosage of an air-entraining agent (AEA) [1]. Additionally to the carbon, other contributors to adsorption of AEA can be hollow FA spheres (cenospheres) and FA spheres filled with numerous small spheres (plerospheres) [2]. A common measure of the increase in dosage of AEA to compensate for the loss of the active ingredient to carbon cannot take variations in fly ash properties into account, and that entails unwanted variations in air entrainment [3]. Trial mixing to ensure quality output is therefore unavoidable even for batch-to-batch variations in fly ash.

The problem can hypothetically be resolved by reducing the number of sorption sites on carbon before the AEA encounters them. Justnes and Ng [4] stated that adsorption of the active ingredient of AEA by carbon in FA can be solved by increasing AEA dosage or add some "sacrificing admixtures that will preferentially adsorb to the carbon". They also assume that it is likely that carbon may preferentially adsorb other organic admixtures like (super-) plasticizers. Plasticizing admixtures will still be attracted the most by AFt phases and alite to disperse the cement, and also FA particles (the glass phase) have some interaction with the admixture, though weaker than with cement and weakest among all the SCMs [5].

Therefore, we think that superplasticizer (SP) could block access of AEA to some carbon in one of the AEA-SP combinations.

Previous measurements have shown large effects on foaming in OPC (ordinary Portland cement) -fly ash water slurries of various SP/AEA combinations and dosage sequences [6]. The foam study indicated that a combination with SP drastically affects the adsorption kinetics. The same materials from that study [6] were also used to investigate the effect of the addition of the admixtures on air entrainment [7].

The sequence of the addition of SP and AEA in concrete has been debated among practitioners for a long time, but the authors do not know any experimental studies of SP-AEA dosage sequence <u>in FA concretes</u> in the literature. For OPC concrete, some authors [8, 9], suggest adding AEA after blending SP in the mix to give a stable air-void spacing factor; others [10–13] say that SP should be added after AEA, providing time for AEA to precipitate.

No standards, committees, or guidelines specify the AEA-SP interaction [14]. Moreover, there is no documentation provided by concrete admixture producers about the compatibility of AEAs and SPs. According to specialists from a Norwegian admixture producer [15], all admixtures get pre-qualified separately from other admixtures. In the company standard, for example, AEA is tested in OPC concrete, targeting slump at 50mm and 4-6% total air content. It is not understood how the admixture producers announce the compatibility of admixtures without providing the meaning of it. In addition, we know of only two studies [16, 17] which revealed the composition of air-entraining agents, and it makes it impossible to assess the performance of AEA without trial mixing.

In the industry, SP-AEA dosage sequence practice varies due to the limitations of the concrete plant, economic reasons, or the producer's or client's established practice. The concrete producers reviewed in this study recommend that AEA is added either before or simultaneously with SP in the concrete mixes containing either pre-blended or separately added FA. Also, the mixing time varies from one to two minutes depending on strength and durability class, and in case of sampling – it increases to 3 minutes. Variability of the production parameters, the inexistence of the regulations and maybe some inaccuracy of the concrete producers reduce chances to control air entrainment in FA concrete.

With an increased need for high volume fly ash concrete the need for real knowledge about AEA and SP interaction in FA concrete is growing. Yet worth mentioning that in the near future the availability of "pure" FA may be reduced due to combusting coal together with waste products (rubber etc.), which might complicate the task of making concrete which fulfills the demands for XF4.

The scope of this work was to investigate air void content and structure from laboratory Fly Ash concrete mixes where both the type of AEA and the dosage sequence of AEA- and a co-polymer SP were varied. If effective, it would be a practical and simple way of remedying the problem.

2. MATERIALS AND MIXES

Two main series of concrete mixes (d_{max}>6mm) were made to investigate the effect of admixture combinations and dosage sequence on air-void parameters:

- ***M-series*** [7] where compositions were constant while comparing the effect of dosage sequence
- ***O-series*** [18] where two different binder types were investigated and where much more emphasis was put on controlling workability and total air content by varying AEA dosage, which is more related to practice.

2.1 Constituent materials

Table 1 – Material parameters

Material	Density [kg/m^3]	Carbon [%]	Loss on ignition [%]	Blaine [m^2/kg]
M-series				
Norcem Standard cement (CEM I 42,5R)	3 150		2,35	396
Norcem Fly Ash	2 300	1,74	2,27	334
Limestone filler	2 730		37,66	362
O-series				
Norcem Anlegg cement (CEM I 52,5N)	3 140	0,42	2,33	360
Norcem Anlegg FA cement[1] (CEM II/A-V – 42,5N)	3 020	0,79	2,74	384
Norcem Fly Ash, LN3-17	2 310	3,01	3,16	334
Silica Fume 940D	2 200			

[1] Norcem Anlegg FA cement contains 14,1% fly ash as a replacement by mass
[2] Carbon content in fly ash was measured by ELTRA (combustion and infrared detection)

Table 2 – Aggregate grain-size distribution

Aggregate	Cumulative [%] passing for sieve opening [mm]														
	11,2	8	5,6	4	2	1	0,5	0,25	0,125	0,063	0,032	0,016	0,008	0,004	0,002	0,001
Sand 0-8	100	98,9	89,7	79,8	62,1	44,6	28,8	16,1	7,2	2,7						
Limestone filler									100	87,2	67,1	46,7	30,1	18,7	9,9	3,3

Table 2 shows the aggregate size distribution. It was the standard Norwegian gneiss-granitic 0-8 mm sand supplied by NorStone Årdal.

Admixtures

Anionic air-entraining agents:

- AEA4 (***M-series***) – ready to use olefin sulfonate, synthetic tenside,
- AEA5 – a concentrate based on synthetic tensides and tall oil derivatives (natural):
    - AEA5 fresh (***M-*** and ***O-series***) – AEA5 blended 1:9 with water shortly before mixing;
    - AEA5 pre-blended (***O-series***) – aged AEA5 up to 2 months after blending the concentrate of it with water 1:9.

Note: AEA4 and AEA5 of the same batch were used in the Foam Index study [6], and, therefore, the coding for the admixtures was kept unchanged for traceability.

Superplasticizer (SP) for both series – ether-based polycarboxylate from the same batch, solid content – 30±1.5%.

SP and AEA5 are from the same producer, and accordingly "compatible".

2.2 Mixes

Table 3 shows the ***M-series – "constant AEA dosage-variable workability"***, where three different mix compositions were dependent on the volume fraction of filler-modified paste (= matrix = all liquid, admixture, binder and mineral filler with particle size < 125 microns) and used air-entraining agent: 330 and 400 liters of matrix with AEA5 and 400 liters of matrix with AEA4.

Key requirements to ***M-series*** mixes:

- w/b – 0,46 (400L matrix), 0,57-0,63 (330L matrix)
- FA/(FA+C) – 0,30
- Limetone – appr. 24kg/m^3
- Slump cone (Mortar cone) - 100±10mm (only for 400L matrix mixes)
- Constant dosage of AEA.

Table 4 shows the ***O-series – "constant workability – variable AEA dosage",*** where two different binder types (see Table 4) were investigated and emphasis was put on controlling workability (100 +/-10mm) and total air content, which is more related to practice.

In both series, the idea was to find the most reliable dosage sequence of AEA and SP in terms of air void system and reproducibility.

Key requirements to ***O-series*** mixes:

- w/b – 0,40 (400L matrix)
- FA/(FA+C) – 0,35
- Slump cone (Mortar cone) - 100±10mm

- Air content – 6-8% (for d_{max} 8mm), corresponding to about 4-6% for concrete with d_{max} >16mm.

Table 3 – Mix design (corrected for measured density and fresh air) for ***M-series***

Mix	AEA	ID	Mass of constituent materials [kg/m^3]						
			Cement	Fly ash	0-8mm	Filler	Water	AEA	SP
330	−	0	289,3	124,0	1575,8	20,7	213,6		
	AEA 5	AEA	262,3	112,4	1647,6	18,7	194,2	2,6	
		AEA-SP	253,8	108,8	1617,7	18,1	193,3	2,6	0,7
		SP-AEA	251,7	107,9	1604,4	18,0	191,7	2,5	0,7
		AEA+SP	251,8	107,9	1605,2	18,0	191,8	2,5	0,7
400	−	0	341,1	146,2	1585,1	25,6	199,4		
	AEA 5	AEA	339,6	145,6	1564,6	25,5	195,3	3,4	
		AEA-SP	347,4	148,9	1594,1	26,1	197,8	3,5	2,2
		SP-AEA	335,1	143,6	1537,7	25,2	190,8	3,3	2,1
		AEA+SP	321,6	137,8	1476,0	24,2	183,1	3,2	2,1
	AEA 4	AEA	337,6	144,7	1555,3	25,4	194,1	3,4	0,0
		AEA-SP	344,4	147,6	1580,7	25,9	196,1	3,4	2,2
		SP-AEA	328,4	140,8	1507,2	24,7	187,0	3,3	2,1
		AEA+SP	302,1	129,5	1386,6	22,7	172,0	3,0	1,9

*Table 4 – Mix design (corrected for measured density and fresh air) for **O-series***

ID	Mass of constituent materials [kg/m^3]									SP/b,%	AEA/b,%
	CEM I[1]	CEM II[2]	SF	FA	Sand 0-8	Free water	Abs. water	SP	AEA		
AEA-SP		363,9	14,6	108,6	1560,2	194,9	4,6	2,9	3,7	0,60	0,76
SP-AEA		363,7	14,6	108,6	1560,2	194,8	4,6	3,8	3,9	0,77	0,79
AEA-SP		364,0	14,6	108,6	1560,2	194,9	4,6	2,4	9,7	0,50	2,00
SP-AEA		363,7	14,6	108,6	1560,2	194,8	4,6	3,8	3,4	0,77	0,70
SP-AEA		363,7	14,6	108,6	1560,2	194,8	4,6	3,8	3,7	0,77	0,76
SP-AEA		363,8	14,6	108,6	1560,2	194,8	4,6	3,4	3,9	0,69	0,80
SP-AEA	485,5		15,0	0	1560,2	200,2	4,6	3,5	4,1	0,71	0,82
SP-AEA	485,6		15,0	0	1572,4	200,2	4,6	3,5	4,2	0,71	0,84
SP-AEA	485,6		15,0	0	1572,4	200,2	4,6	3,5	4,2	0,71	0,84
SP-AEA	485,5		15,0	0	1560,2	200,2	4,6	3,5	3,9	0,70	0,78
SP-AEA	485,5		15,0	0	1560,2	200,2	4,6	3,5	3,5	0,70	0,70
SP-AEA	485,5		15,0	0	1560,2	200,2	4,6	3,5	4,5	0,70	0,91
SP-AEA	485,2		15,0	0	1560,2	200,1	4,6	4,5	3,7	0,91	0,73
SP-AEA	485,4		15,0	0	1560,2	200,1	4,6	3,9	4,9	0,78	0,99
SP-AEA		364,2	14,6	108,7	1545,5	195,0	4,6	3,4	4,8	0,70	0,99
SP-AEA		363,7	14,6	108,5	1560,2	194,8	4,6	3,9	5,4	0,80	1,11
SP+AEA		375,2	15,0	112,0	1545,5	201,0	4,6	2,5	8,0	0,49	1,60
SP+AEA		375,2	15,0	112,0	1545,5	201,0	4,6	2,5	8,0	0,49	1,60
SP+AEA		375,1	15,0	112,0	1545,5	200,9	4,6	3,1	3,5	0,62	0,70
SP-AEA		375,2	15,0	112,0	1545,5	201,0	4,6	2,5	3,9	0,49	0,78
AEA-SP		375,2	15,0	112,0	1545,5	201,0	4,6	2,5	10,1	0,49	2,01
AEA-SP		375,2	15,0	112,0	1545,5	201,0	4,6	2,5	10,1	0,49	2,01
AEA-SP		375,1	15,0	112,0	1545,5	200,9	4,6	3,1	7,6	0,62	1,52
AEA-SP		375,0	15,0	111,9	1545,5	200,5	4,6	3,5	4,5	0,70	0,90
AEA-SP		375,1	15,0	112,0	1545,5	200,9	4,6	3,1	6,0	0,62	1,19
SP-AEA		376,0	15,0	110,3	1560,2	200,5	4,6	3,9	3,5	0,78	0,70
SP+AEA	495,2		15,4	0	1560,2	204,1	4,6	3,6	3,8	0,70	0,74
AEA-SP	494,8		15,4	0	1560,2	204,1	4,6	3,8	3,9	0,74	0,76
AEA-SP	495,2		15,4	0	1560,2	204,1	4,6	3,6	3,9	0,70	0,76
AEA-SP		363,8	14,6	108,6	1560,2	194,8	4,6	2,4	9,7	0,50	2,00
SP+AEA		364,0	14,6	108,6	1560,2	194,8	4,6	2,4	7,8	0,50	1,60
SP		363,7	14,6	108,6	1560,2	194,8	4,6	3,9	0	0,80	0
SP		363,7	14,6	108,6	1560,2	194,8	4,6	3,9	0	0,80	0

[1] Norcem Anlegg cement; [2] Norcem Anlegg FA cement

Note: the shaded cells highlight mixes with "AEA5 fresh", while for unshaded cells "AEA5 pre-blended" according to the notation for admixtures given in 2.1.

3. METHODS

Table 5 presents admixture combinations and mixing sequences chosen based on the experience with Foam Index (FI) testing [6]. The FI testing is done in the following order: (1) add AEA with precision pipettes into a container with pre-shaken (10 Hz, 60 seconds) mix of binder and water (w/b 2,5), (2) close the lid and shake the container for 15 seconds (10 Hz), (3) remove the lid and observe the foam for 45 seconds, recording the time of stable foam. The procedure is described in detail in [6].

Table 6 gives an overview of used methods and equipment during the testing. The prolonged mixing time of at least 2 minutes after the addition of AEA was chosen to assure full activation of surfactant [19] and reduced variability caused by fly ash [20]. We changed the mixing equipment from Hobart to Sandby mixer because of the unavailability of the first equipment and a need to increase the batch size for additional tests. It should be mentioned that despite similar mixture proportions for ***M-*** and ***O-series***, changing the mixer type could affect the performance of the admixtures. This could largely affect the size of the air bubbles [2], hence the stability of air content and the air-void structure.

Tables 7 and 8 give a summary of fresh concrete properties for ***M-*** and ***O-series***, respectively. Complete tables for fresh concrete properties for each mix in ***M-*** and ***O-series*** are in Tables A and B respectively, see Attachment.

Table 5 – Admixture combinations and mixing sequences

Series	Admixture	Mixing sequence
M-series	0	1 min dry materials, 3 min water
	AEA	1 min dry materials, 3 min water+AEA
	AEA - SP	1 min dry materials, 2 min water+AEA, 1 min SP
	SP - AEA	1 min dry materials, 1 min ½ water+SP, 2 min ½ water+AEA
	SP + AEA	1 min dry materials, 3 min water+AEA+SP
O-series	SP	1 min dry materials, 1 min water, 5 min SP, 2 min rest, 1 min mixing
	AEA - SP	1 min dry materials, 1 min water, 3 min AEA, 2 min SP, 2 min rest, 1 min mixing
	SP - AEA	1 min dry materials, 1 min water, 2 min SP, 3 min AEA, 2 min rest, 1 min mixing
	SP + AEA	1 min dry materials, 1 min water, 5 min SP and AEA, 2 min rest, 1 min mixing

Table 6 – Equipment and test methods

Series	Mixing		Properties of concrete		
	Batch size [l]	Equipment	Air content, air-void system, fresh state	Workability	Porosity, air-void system, hardened state
M-series	4	5L Hobart mortar mixer	Density method[1], Pressure method[2]	Mortar slump cone[3] 120x80x40mm^3	Image analysis, PF-method
O-series	5 or 6	10L Sandby SU10 Paddle mixer	Density method, Pressure method		

[1] According to ASTM C138/C138M - 17a by comparing unit weight with theoretical density
[2] Pressure device for mortars (1L) was used
[3] Same procedure as for the standard slump test EN 12350-2, but the mini-cone is filled in 2 layers, each is tamped with 25 strokes [7,18].

The Image analysis on hardened specimens 160 x 40 x 40 mm^3 was performed in accordance with [21] and ASTM C457 on two well-hardened specimens for each series. The specimens were cut normal to a casting surface, ground using SiC grinding papers of 320, 500, 1200 grit to a light-reflective surface and the air-voids with sharp edges. Then the ground surface was painted black with a marker Edding 850 3 times, and the air voids were filled with the $BaSO_4$ powder (particles 1-4μm) by finger-tapping and pressing. The excessive powder was firstly dragged off by a straightedged plastic ruler, and secondly by a slightly moist finger. Further, cracks and blemishes on the aggregates that got filled by the barium sulfate powder and, therefore, could cause erroneous air void characteristics, were painted black under the microscope. Prepared samples were placed on transparent foil, scanned by Epson Perfection V600 Photo at 2400ppi and analyzed using the Matlab script, developed by Fonseca [21].

The consistency, air content and density measurements for both series were performed between 10 and 15 minutes after water was added to the mix.

'*Table 7 – Properties of fresh concrete. **M-series***

Matrix volume [l]	Paste volume [l]	w/b	Type of AEA[1]	AEA, [% (c+FA)]	SP, [% (c+FA)]	Slump [mm]
330	359	0.57	-	0	0	30
330	319 - 326	0,60 – 0,63	AEA5	0,7	0 – 0,20	20 - 60
400	371	0,46	-	0	0	20
400	346 - 373	0,46	AEA5	0,7	0 – 0,45	30 - 100
400	325[2] - 370	0,46	AEA4	0,7	0 – 0,45	25 - 105

[1] See 2.1. [2] 325L for SP+AEA, while other admixture combinations ranged from 353L to 370L.

*Table 8 – Properties of fresh concrete. **O-series***

Matrix volume [l]	Paste volume [l]	w/b (w/c)	Mixing volume, L	Type of AEA	AEA, [% b]	SP, [% b]	Slump [mm]
CEM I							
400	363	0.40	5,0 – 6,1	AEA5 fresh	0,70 – 0,91	0,70 - 0,71	82 - 109
400	363-370	(0.41)		AEA 5 pre-blended	0,73 – 0,99	0,70 – 0,91	85 - 110
CEM II							
400	370	0.40	5,0 – 6,1	AEA5 fresh	0,50 - 0,70	0,70 – 2,0	82 - 105
400	370-381	(0.62)		AEA 5 pre-blended	0,49 – 0,80	0,70 – 2,0	85 - 107

4. RESULTS & DISCUSSION

4.1 Fresh air void content

Due to many factors affecting air entrainment, we have in the following made an effort to look at air entrainment effect of (1) workability, (2) AEA dosage and (3) AEA-SP dosage sequence. The latter is the main point of this study and we have therefore paid special attention to this in terms of analyzing air-void parameters of hardened concrete as the effect of AEA-SP dosage.

Figure 1 shows similar relationships between density-based and pressure-meter-based air-void content for the ***O***- and ***M-series***, though better correlated for the ***M-series***. This is presumably due to that in the ***O-series*** both dosage and sequence varied (in the ***M-series*** dosage was constant while the sequence of addition varied), accompanied by variations in batch size and use of less efficient mixing equipment for ***O-series***.

Figure 1 does not show a 1:1 relationship and some apparent negative values are displayed for the density method, presumably due to several factors. One could be that the constituent materials do not exhibit the same particle densities in the fresh mix as in the particle density measurements. Another reason is undoubtedly the very different principles with Boyle-Mariotte's law behind the pressure meter and different effect on air voids of different sizes due to their different compressibility. The smaller the void the larger the pressure needed to compress, but the more likely to dissolve the air void into the water. The two measurements were made on the same fresh concrete sample but the pressure meter measurement could, of course, have a systematic error for various reasons (equipment, calibration, operator dependent, etc). Still, from the ***M-series,*** it appears that the density method for a given set of part-material data and rather a simple lab equipment (container, balance) is capable of giving a very good correlation to the standard pressure meter.

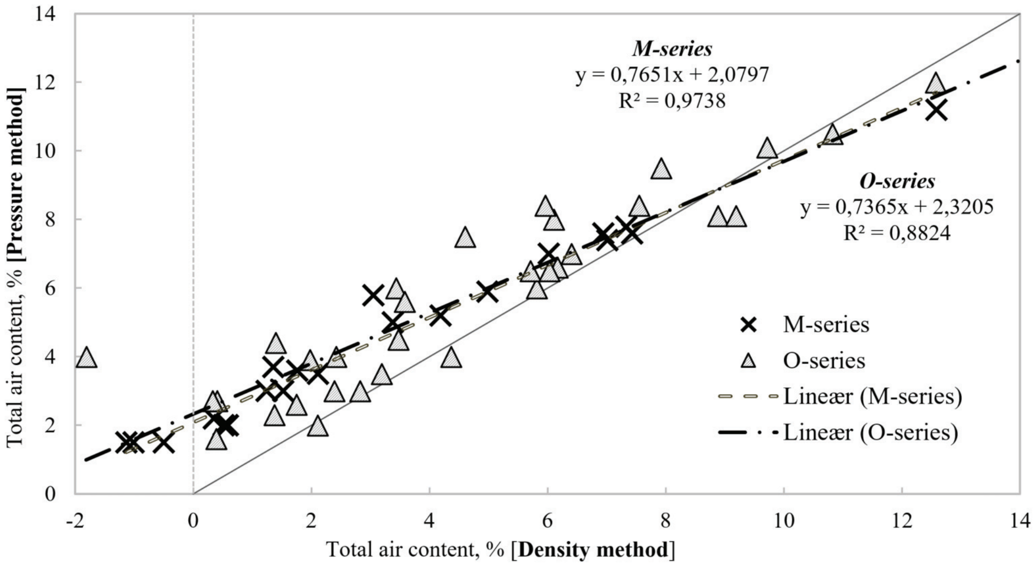

*Figure 1 – Correlation between density and pressure method for obtaining a value of the total air content in fresh concrete for **M-series** and **O-series.***

4.2 Effect of AEA-SP sequence on fresh air void content

Figure 2 shows the effect of AEA-SP dosage sequence on air entrainment in the ***M-series*** compared to reference mixes without any admixture and with only AEA. From the bar graph, it is clearly seen that the same dosage of AEA results in widely different air entrainment in fresh concrete depending on the sequence of dosage. Of the 3 sequences with both AEA and SP we see that 4-AEA+SP simultaneously always gives the highest air void content, 2- AEA before SP always give lowest, and 3-SP before AEA gives an intermediate fresh air void content. The references without admixture and the references with only AEA give low air content within each group of matrix volumes. Also, note that within each group of matrix volume the slump was almost constant: 60, 90-100 and 90-105 mm for 330L AEA5, 400L AEA5 and 400L AEA4, respectively.

The workability also affected air entrainment, as seen by comparing with the reference mixes without AEA: #0 without any admixture (20 – 40 mm slump) and #1 with only AEA (20-30 mm slump). Possibly, there is some sort of reciprocal effect between air content and slump. For all the mixes, comparing a sequence #1 without SP to other with SP, we could observe a general increment in values of the total air content with increased workability, except for matrix 400L and sequence #2 – AEA-SP (SP added after AEA).

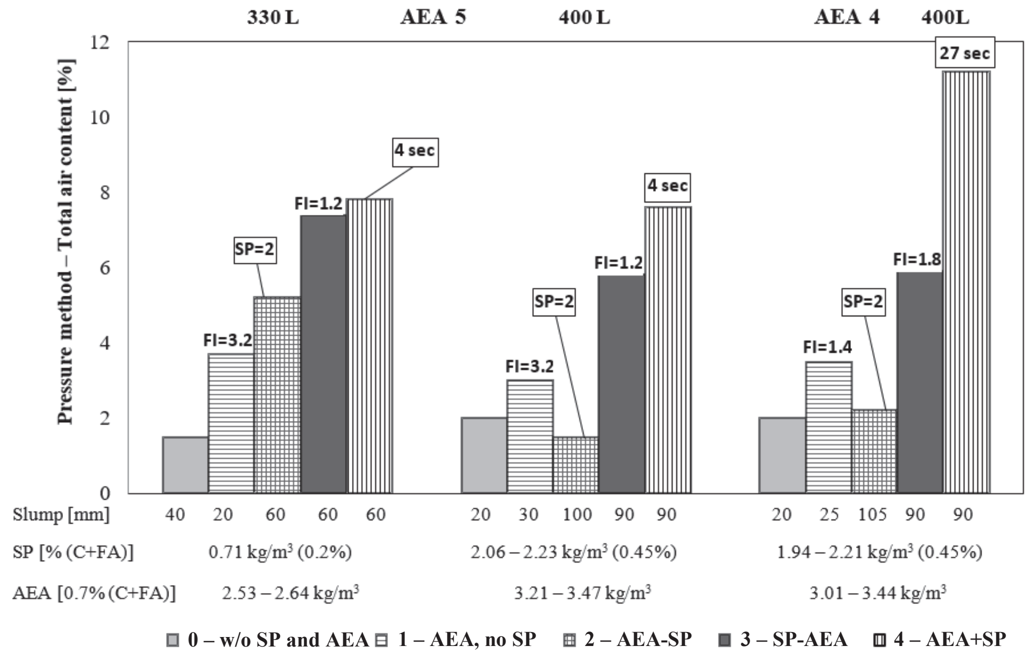

Figure 2 – Effect of AEA-SP dosage sequence on air entrainment. M-series: Comparison of pressure meter results with FI measurements, Jacobsen et al. [6] for different matrix volumes and AEAs.

<u>Note</u>: In Figure 2, FI (Foam Index) means a dosage of AEA in ml per gram of binder needed to obtain a stable foam for 45 seconds after shaking a water/binder suspension with AEA in a closed container. The framed text above the columns means that a stable foam was not obtained for the mixes. SP=2 means that after adding AEA and obtaining a foam it took 2 droplets of 20 µl SP each to kill the foam. Time in seconds is a lifetime of the foam on the surface.

Increase in matrix volume (or content of fines) for AEA5 generally led to a reduction of the total volume of air bubbles, and the drop is almost threefold for series #2 – AEA - SP. It means that the addition of SP after AEA to obtain flowable consistency in a rather refined system (400L of matrix contra 330L) causes coalescence and loss of stability for air bubbles (see discussion for Figure 3), and, hence, unwanted air detrainment [2, 9]. It is worth mentioning that for 330L matrix, the addition of SP (leading to 60mm in slump) led to an increase in air content for all the series with SP, meaning that it may be either an increase in workability to 100mm or refinement of the system that caused air detrainment for the sequence #2 for 400L matrix.

When other parameters are kept constant, the highest amount of air voids is guaranteed by adding AEA and SP simultaneously (series #4 – AEA+SP), Eickschen [10] and Puthipad [9] also mention this effect. When added together with SP, the pure synthetic surfactant AEA4 shows much higher air entrainment compared to the mixture of natural and synthetic tensides of AEA5, while the difference is small for other dosage sequences.

The results of the foam index measurements on slurries do not fully reflect the properties of the mixes, because this indicative test does not predict the development of the air-void system from the fresh to the hardened state (see corresponding Figure 3 displaying results of air void analysis of hardened concrete). Furthermore, the very high air content for series 4 – AEA+SP does not correspond to the "foam-killing" effect (instability of air) observed in [6].

Steinhoff [11] also confirmed difficulties in the application of FI test to verification of mutual performance of AEA and SP, and there was no more good correlation between the BET surface area of FA in concrete and FI, as other authors report [2, 16, 20, 22].

4.3 Effect of AEA-SP sequence on hardened air void content

Figure 3 shows the air entrainment in hardened concrete for two of the three matrix-volume series in Figure 2. Figure 3 confirms a clear effect of dosage sequence also in hardened concrete, especially for 6 mixes with 100 ±10 mm slump at constant SP and AEA dosages. The tendency from left to right for the total air content is the same as for the fresh concrete measurements (Figure 2).

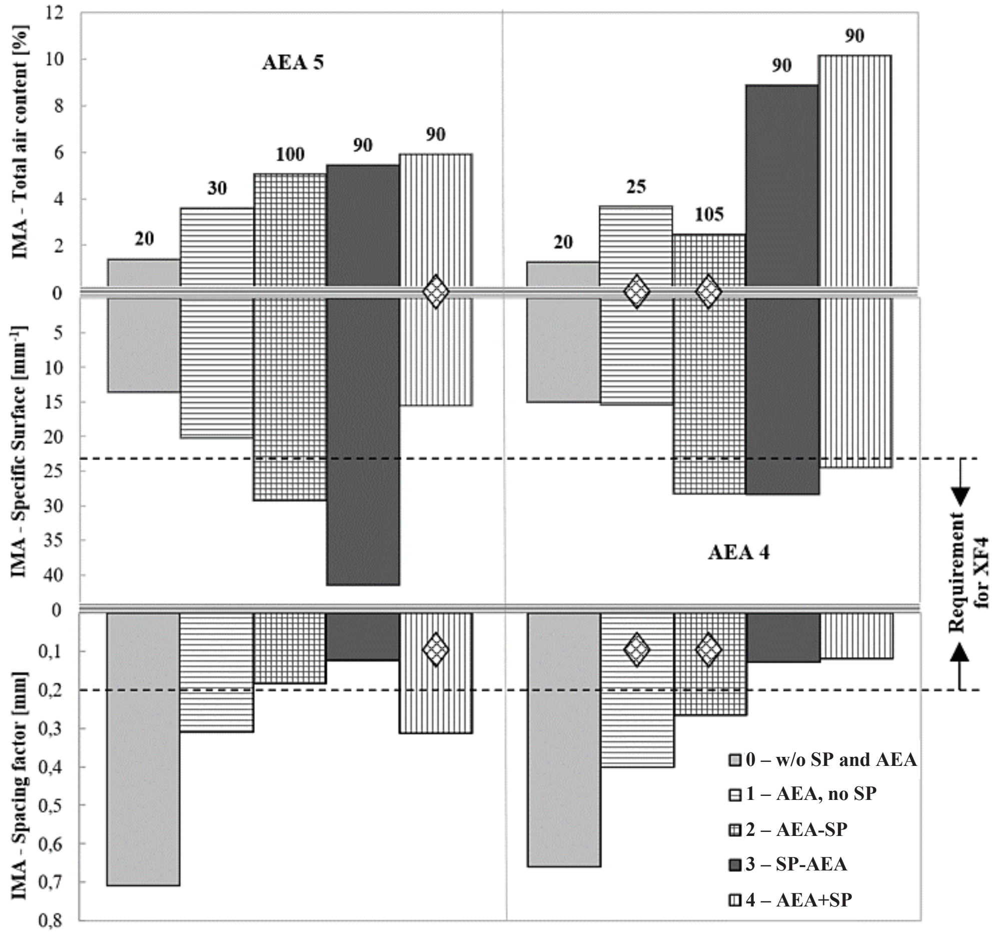

Figure 3 – M-series: Air-void analysis for mixes in the 400L series with the two AEAs

(the numbers over the bars for total air content show values for workability on mortar slump cone. Shaded rhombuses – done by a different operator).

Note: Retention of air content in the fresh state over time (up to 60 min) was reported and summarized by Pedersen [3]. Only three long-chain hydrocarbon-based and epoxy sulfate AEAs showed an increase up to 40% after simulation of transportation for 45 min. In the case of the series #2 AEA – SP and AEA5, there is more than a double increase in total air content from fresh (Figure 2) to hardened state. This we could either assign to erroneous surface preparation for the air-void analysis or a time-dependent increase in the fresh state without re-agitation due to the coalescence of small voids with higher pressure inside into larger voids with lower pressure. This follows from Laplace giving the pressure difference over the air-liquid interface is $\Delta p = - 2\sigma/r$ with σ = surface tension water-air, r =void radius.

The effect of AEA in a stiff concrete is minor (sequence #1 – AEA, no SP) due to resistance against bubble growth from the stiff paste and a limited amount of sites for the AEA to adsorb on, because of the absence of a rather efficient dispersive agent. All mixes with SP give a better air void system than in stiff mixes.

We could assign the variation in air content for sequence #2 – AEA-SP to a difference in the influence of de-foaming agent within SP on the efficacy of AEA. From a limited number of mixes, the influence is stronger for synthetic AEA4, and it may be due to some compatibility of AEA5 and SP (the same producer).

Also, despite low air content (especially in a fresh state, Figure 2) for #2, the air-void specific surface remains high for both AE agents, hence, the reduction of air content for AEA4 can be associated with instability of the coarse air voids over 300 μm when SP is added after AEA. It also implies that having requirements for total air content solely is not the right approach, even though some country standards and organizations have it so [14].

The bars in Figure 3 for a sequence #3 – SP-AEA show that adding AEA in a flowable concrete led to an increase in coarse air voids for synthetic AEA and gave a drastic increase, compared with other sequences, in fine air bubbles (smaller than 300μm) for a semi-synthetic AEA5.

Eickschen [10] described the interaction between AEA and SP when SP comes first – the air void system is formed in a softer concrete, which results in a coarser system, explaining it by competitive adsorption of admixtures on cement particles, resulting in unstable air content. It does not seem to be valid for FA concretes. It was only Pathipad [8] who suggested adding SP before AEA to obtain the most refined air void system, even though he reported suitability of that mixing procedure only for OPC concretes.

The computed spacing factor values from the measured specific surface using Fonseca's method [21], see the solid dark-grey bars in Figure 3, stay well within the required limits (listed in Norwegian national Annex to EN 206 for the most severe frost exposure class XF4). This implies that adding SP first in FA concrete can likely improve the air-void system, comparing to adding SP after AEA (i.e. sequence #2).

As for sequence #4, with simultaneously added AEA and SP, the air void parameters for the concretes primarily depend on the efficacy of the AEA to compete for the sorption sites.

Reproducibility should be taken into consideration because according to the literature [12, 9] the combination #4 is least predictable. Speaking of all the series, mixes with AEA5 were reproduced at least two times, whereas with AEA4 – they were produced only once.

4.4 Effect of AEA-SP sequence vs effect of AEA-dosage

Figure 4 shows AEA dosage vs fresh air void content for all mixes of this study:
O-series: varying AEA-dosage with two different types of AEA: **1**: shaded legends – AEA5 pre-blended- natural (tall oil derivatives)-synthetic mix, diluted 1:9 and stored in lab up to 2 months before use. **2**: open legends – AEA5 fresh - natural-synthetic mix diluted 1:9 and used freshly blended, i.e within 1 hour.

M-series: the vertical bar at constant (0,7 %) AEA dosage (indicating the range for AEA4 Synthetic olefin sulfonate and AEA5 fresh natural-synthetic mix (same as ***O-series***), diluted 1:9 shortly prior to mixing.

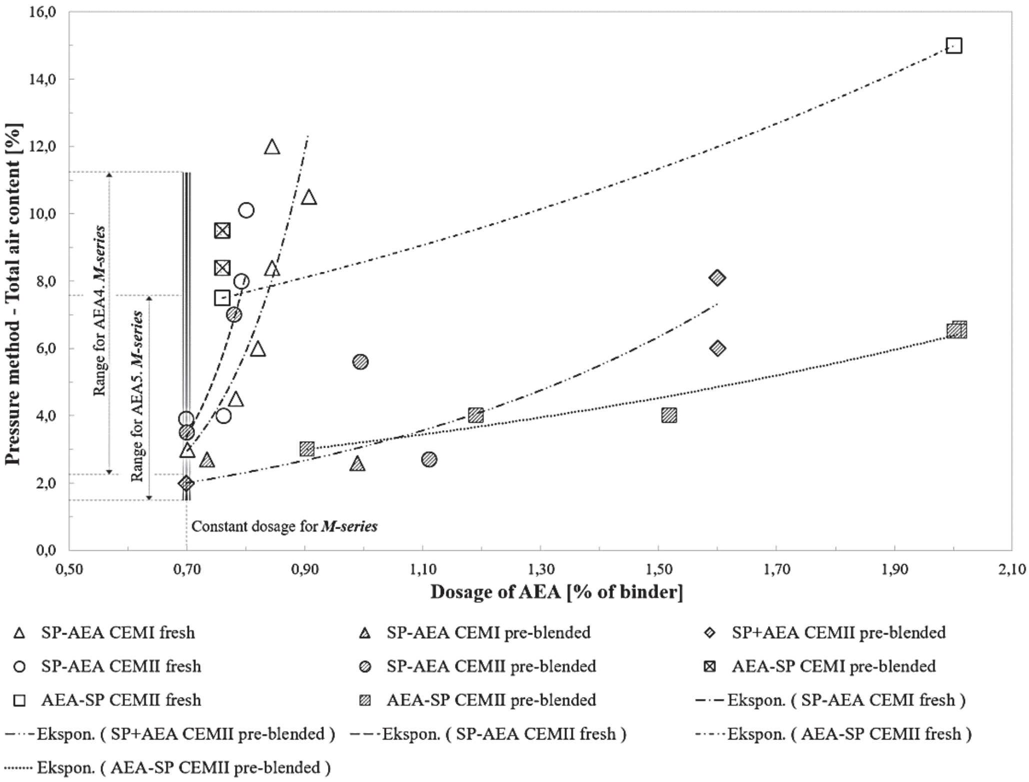

Figure 4 – Influence of AEA-SP dosage sequence on binder type, AEA type and dosage, and total air content.

The target of 6-8% air content in ***O-series*** at ≈ constant workability (see more on workability further below) caused the dosage of AEA for successful mixes to vary from 0,76 to 2,0 % of binder.

In Figure 4, we see that for the pre-blended AEA and the sequence AEA-SP (the hatched square legends), considering low variability of the curve for FA concrete, the dosage of AEA needed to reach circa 9% (the crossed square legend – OPC reference) of air should be quadrupled when FA is present. That agrees well with Zhang [23], who found that AEA dosage for obtaining air content as in OPC concrete should be 2-6 times higher for FA concrete. Nonetheless, for fresh AEA and the sequence SP-AEA (open triangle and open circle legends), we could read off the increased demand in AEA only up to 15% at most for the mix with 35% FA/b.

Zhang [23] also found that batch-to-batch variability of air content in fresh FA was lower than for OPC concrete. However, when comparing SP-AEA sequence for CEMI and CEMII for fresh AEA, variability is lower for CEMI.

There is, in fact, a large number of variables in addition to the three types of AEA: variable AEA dosage, variable dosage sequence, variable quality and volume fraction of filler-modified cement paste, variable binder type (CEMI – OPC and CEMII Fly Ash+OPC) and variable workability. However, compared to all these variables Figure 4 shows that the dosage sequence has a very large effect on air void content, particularly when considering that workability for most of these mixes was constant (100±10mm): By comparing AEA5 fresh for the ***M-*** and ***O-series*** we see that the magnitude of the dosage sequence effect (M-series, constant dosage, variable sequence) is approximately half (1.8 – 7.6 % air) of the magnitude of the variable dosage series (***O-series***, variable dosage, variable sequence) which varies 2 – 15 % air.

When looking at all three AEA types (AEA5 fresh, AEA5 pre-blend, AEA4) Figure 4 shows that the variation in air-void content is similar in the ***M-*** and ***O-series***. Hence, the dosage sequence has a very large effect on air entrainment. Within the ***O-series*** there is, however, a clear effect of the type of AEA since pre-blended (aged) AEA5 results in lower air entrainment compared to freshly blended AEA5. This is in accordance with Dodson [24] and Spörel [20], who noticed that the properties of AEAs change with age (especially synthetic) when conducting Foam index tests and who advised using solutions (AEAs) that are just a few days old. In connection to this, it is worth mentioning that admixture producers for economical reasons sell concentrated air-entraining agents to concrete producers to be diluted at the facility, but it has not been reported how the reduced performance of AEA with ageing is compensated and controlled.

Within the ***M-series,*** the type of AEA seems to have less effect than within the ***O-series*** except for the simultaneous addition AEA4-SP which gives much higher air void content than simultaneous AEA5-SP.

Looking at the hatched triangles (SP-AEA CEMI pre-blended) and circles (SP-AEA CEMII pre-blended) in Figure 4 one can see the series with the highest scatter. In fact, the hatched circles with dosage of AEA 0,7% (very first successful mix, i.e AEA was aged from a few hours to a few days) and 0,8% of binder represent concretes cast about 3 weeks apart, and the results fit well the exponential curve for the same order of addition of the admixtures with the fresh AEA (blank circles). Two other hatched circles and the hatched triangles were cast the same day, but 6 weeks later than the two abovementioned mixes. Here it is clear that the performance of the AEA had become unpredictable.

From the example above, it could be that within a certain period of time the pre-blended AEA performs as well as freshly blended. But if we look at the hatched and blank squares for AEA-SP sequence with pre-blended and fresh AEA5 in FA concrete respectively, there is a drop of the effectiveness of the AEA of about 2,5 times, and it is despite the fact that the AEA solution was also aged in about 3 weeks, as in the previous example.

We assume that the reason for the different behavior of the mixes is that adsorption susceptibility of the active ingredient in AEA to the carbon in FA is higher when the AEA is added in stiff concrete, i.e before SP. It may be that with ageing AEA loses the active ingredient easier to the carbon in FA, comparing to the freshly mixed AEA. This conclusion can presumably be valid for simultaneous addition of AEA and SP (the hatched rhombuses in Figure 4, age of AEA – ca 3 weeks).

4.5 Fresh air void content, AEA and workability

Figure 5 shows slump vs total air content with shaded legends for the ***M-series*** and open legends and "X" for the ***O-series***. Again, we see the somewhat higher variation in air content for the ***O-series*** (variable AEA dosage) than for the ***M-series*** (constant AEA dosage – variable sequence).

Exponential trendlines (added to facilitate readability of the figure) for the same dosage sequences are drawn in one style, and we can see that the effects of dosage sequences in the ***O-*** and ***M-series*** can be somewhat related despite different mixing equipment, quality of AEA (fresh or pre-blended), batch size used and, even, constituents (limestone filler in ***M-series*** was replaced with 4% Si/b in ***O-series***).

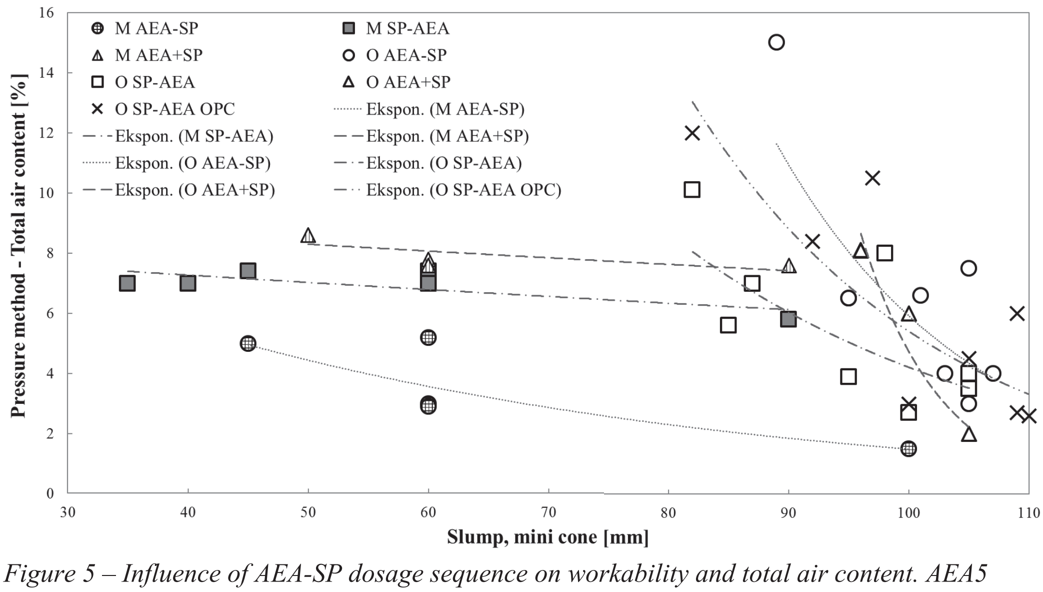

Figure 5 – Influence of AEA-SP dosage sequence on workability and total air content. AEA5

As mentioned, there could be a reciprocal effect between air voids and workability. The too low viscosity of the paste could allow air voids to rise and disappear whereas increased paste volume due to air voids increases slump.

Dodson [24] noted that air content increases by an increasing slump from 75 to 150 mm, but above 150 mm the air content drops because of the reduced viscosity in the paste insufficient to withstand buoyance forces by large air bubbles. This is in line with the "reciprocal effects" including the right-hand side of Figure 5 where there is a tendency of low air void content at high slump values, i.e. air voids escaping more easily in a fluid mix. Sequence AEA-SP for both ***M-*** and ***O-series*** gave the highest decrement in air content with an increased slump, which could indicate susceptibility to air detrainment when SP enters the system after AEA.

On top of this, there is the effect of surfactants on increasing the yield stress and hence the slump which counteracts the effect of the air voids [25]. The effect of air entrainment on workability, therefore, is hard to predict.

5. CONCLUSIONS

- Traditional thinking of admixture dosage sequence in OPC concretes cannot be applied for FA concretes. The most favorable admixture combination for air entrainment in FA concrete seems to be when SP is added before AEA.
- The results from Foam Index measurements [6] do not fully reflect the properties of fresh and, especially, hardened concrete. Hence the Foam Index test seems unsuitable for a combination of admixtures.
- To fulfill requirements to effective air void spacing while maintaining as low air void content as possible within the requirements for exposure class XF4 seems possible. However, it requires specific procedures based on trial mixing including requirements to the sequence of AEA-SP dosage.
- Performance of a selected AEA plays an important role, which could possibly allow ignoring differences in AEA-SP dosage sequence when the needed dosage is found and reproducibility achieved during a pre-qualification phase.
- Performance of selected SP was not affected by AEA-SP dosage sequence as the amount of SP was kept constant for ***M-series***. It means that the selected polycarboxylate may not lose its active ingredient to the carbon in FA. Yet the polycarboxylate SP, when added first, seems to shield AEA from being adsorbed and becoming less efficient.
- The workability affects air entrainment with some sort of reciprocal effect between air content and slump. However, we cannot confirm that the results described for OPC on Abrams slump cone [2, 24, 12] fit the obtained relationship for fly ash concrete on mortar-cone.

6. FUTURE WORKS

Comparison of air void structures in hardened concrete for varying only matrix volume and dosage sequence of the admixtures could be of use to pick out the most favorable combination for fly ash concrete produced in laboratory conditions.

Studies on air void stability for different dosage sequences with AVA (Air Void Analyser), using a similar approach as Pathipad [9] and Spörel [20], will be an important supplement to the present paper.

In addition, more systematic research on the variability of the fresh AEA demand and the total air content for concrete with and without fly ash and various AEA-SP orders of addition is required.

We cannot draw a conclusion about the effect of the AEA-SP order of addition on the demand for AEA (see Figure 4) because of a lack of data and unconfirmed effectiveness of pre-blended AEA for different mixes. Therefore, we think that more systematic research on the variability of the fresh AEA demand and the total air content for fly ash concrete (and without fly ash) and various AEA-SP orders of addition is required.

Further investigations with a rather systematic approach aiming at obtaining a reproducible air-entrained fly ash concrete with d_{max} increased to at least 16mm is required.

6. EXAMPLE OF APPLICATION

Based on the positive response (in terms of air entrainment) of FA-mix on the addition of SP first and subsequent addition of AEA, the main concrete mixes (d_{max} 16mm) for the Ph.D. project "Production and documentation for frost durable concrete" were successfully produced.

About 60%-80% of SP was added together with water to obtain consistency of about 170-180 mm for standard Abrams slump cone. AEA5 (ready to use diluted by the producer) was added 1 minute after SP (ether-based polycarboxylate, solid content – 23±1.5%) followed by 2 min rest, then dosing more SP to obtain slump of 200 ± 10mm and remixing for about 1 minute. Concrete volume – 57 liters. 35% FA/b, 4% SF/b.

Table 9 – The practical application of research results in the Ph.D. project

Concrete mix	Dosage of admixtures per mass of binder, [%]		Slump, [mm]	Density method		AVA measurements						Air content in hardened concrete, PF-test, [%]
						Sampling after 20-25 min			Sampling after 60-70 min			
	AEA	SP		Fresh density, [kg/m^3]	Air content, [%]	Micro air [1]	Spacing factor, [mm] [2]	Specific surface, [mm^{-1}] [2]	Micro air [1]	Spacing factor, [mm]	Specific surface, [mm^{-1}]	
0,40w/b, 35%FA	0,43	0,85	200	2319	5,6	2,4	0,24	25,3	2,2	0,25	24,2	5,0
0,45w/b, 35%FA	0,49	0,72	200	2299	5,8	2,1	0,24	26,7	2,6	0,23	25,4	5,9
0,293w/b, 35%FA	0,60	1,46	220	2346	5,9	3,4	0,15	35,8	3,2	0,17	33,0	5,2
0,45w/b, 0%FA	0,20	0,80	190	2327	5,1	3,7	0,20	24,4	2,6	0,25	23,3	5,4

[1] Chord length <0,35mm

[2] Requirements for the air-void spacing factor – max 0,25mm, specific surface – min 24 mm^{-1}, micro air – 1,8%.

Table 9 shows that it is possible to produce a robust air-entrained concrete with a high volume of FA, workable and stable. It took just one 30L-trial mix for each FA-concrete to obtain a material of the required parameters. OPC concrete mix required three trial mixes and two additional 57l-mixes with the same mixing procedure, time-dependent AVA results show that the air-void system is not persistent. The last can only confirm Eickschen's theory [10] about the instability of air bubbles when AEA is added into soft (somewhat flowable) concrete.

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APPENDIX A. FRESH CONCRETE PROPERTIES

Table A – Properties of fresh concrete. Full range of the mixes. ***M-series***

Sequence	Paste volume [l]	w/b	Admixture dosage [% b]		Air content [%]		Slump cone [mm]	Fresh density, [kg/m3]
			AEA	SP	Pressure method	Density method		
AEA5. 330l matrix volume								
0 – No AEA, no SP	359	0,57	0	0	1,5	-1,1	30	2223
	359	0,57	0	0	1,5	-1,0	40	2221
1 – AEA	325	0,59	0,7	0	3,6	1,8	20	2227
	326	0,59	0,7	0	3,7	1,4	20	2238
2 – AEA-SP	321	0,62	0,7	0,2	5,2	4,2	60	2195
	324	0,60	0,7	0,2	5,0	3,4	45	2221
3 – SP-AEA	319	0,62	0,7	0,2	7,4	7,0	60	2177
	321	0,61	0,7	0,2	7,0	6,0	35	2199
4 – AEA+SP	319	0,62	0,7	0,2	7,8	7,3	60	2178
	318	0,61	0,7	0,2	7,6	7,4	60	2178
AEA5. 400l matrix volume								
0 – No AEA, no SP	371	0,46	0	0	2	0,5	20	2299
	371	0,46	0	0	2	0,6	20	2297
1 – AEA	367	0,46	0,7	0	3	1,2	25	2281
	366	0,46	0,7	0	3	1,5	30	2274
2 – AEA-SP	367	0,46	0,7	0,2	3	1,3	60	2279
	368	0,46	0,7	0,2	2,9	1,2	60	2281
	373	0,46	0,7	0,45	1,5	-0,5	100	2320
3 – SP-AEA	348	0,46	0,7	0,2	7	6,6	40	2157
	347	0,46	0,7	0,2	7,4	6,8	45	2152
	348	0,46	0,7	0,3	7	6,4	60	2161
	360	0,46	0,7	0,45	5,8	3,1	90	2238
4 – AEA+SP	341	0,46	0,7	0,2	8,6	8,4	50	2115
	340	0,46	0,7	0,2	8,6	8,5	50	2116
	346	0,46	0,7	0,45	7,6	6,9	90	2148
AEA4. 400l matrix volume								
1 – AEA	364	0,46	0,7	0	3,5	2,1	25	2261
2 – AEA-SP	370	0,46	0,7	0,45	2,2	0,3	105	2300
3 – SP-AEA	353	0,46	0,7	0,45	5,9	5,0	90	2193
4 – AEA+SP	325	0,46	0,7	0,45	11,2	12,6	90	2018

Table B – Properties of fresh concrete. Full range of the mixes. ***O-series***

Sequence	Mixing volume, [l]	Paste volume [l]	Admixture dosage [% b]		Air content [%]		Slump cone [mm]	Fresh density, [kg/m3]
			AEA	SP	Pressure method	Density method		
Cem I:								
SP-AEA	6,1	363	0,82	0,71	6,0	3,4	109	2302
SP-AEA	6,1	363	0,84	0,71	12,0	12,6	82	2086
SP-AEA	6,1	363	0,84	0,71	8,4	7,5	92	2205
SP-AEA	5	363	0,78	0,70	4,5	3,5	105	2301
SP-AEA	5	363	0,70	0,70	3,0	2,4	100	2327
SP-AEA	5	363	0,91	0,70	10,5	10,8	97	2126
SP-AEA	5	363	0,73	0,91	2,7	0,4	109	2374
SP-AEA	5	363	0,99	0,78	2,6	1,8	110	2343
SP+AEA	5	370	0,74	0,70		19,7	85	1913
AEA-SP	5	370	0,76	0,74	8,4	6,0	107	2239
AEA-SP	5	370	0,76	0,70	9,5	7,9	95	2192
Cem II:								
AEA-SP	6,1	370	0,76	0,60	7,5	4,6	105	2239
SP-AEA	5	370	0,79	0,77	8,0	6,1	98	2203
AEA-SP	6,1	370	2,00	0,50	15,0	17,8	89	1930
SP-AEA	5	370	0,70	0,77	3,9	2,0	95	2300
SP-AEA	5	370	0,76	0,77	4,0	2,4	105	2290
SP-AEA	5	370	0,80	0,69	10,1	9,7	82	2118
SP-AEA	6,1	370	0,99	0,70	5,6	3,6	85	2260
SP-AEA	6,1	370	1,11	0,80	2,7	0,3	100	2338
SP+AEA	5	381	1,60	0,49	8,1	8,9	96	2131
SP+AEA	5	381	1,60	0,49	6,0	5,8	100	2203
SP+AEA	5	381	0,70	0,62	2,0	2,1	105	2289
SP-AEA	5	381	0,78	0,49	7,0	6,4	87	2189
AEA-SP	5	381	2,01	0,49	6,6	6,2	101	2195
AEA-SP	5	381	2,01	0,49	6,5	5,7	95	2205
AEA-SP	5	381	1,52	0,62	4,0	4,4	107	2237
AEA-SP	5	381	0,90	0,70	3,0	2,8	105	2273
AEA-SP	5	381	1,19	0,62	4,0	-1,8	103	2381
SP-AEA	5	381	0,70	0,78	3,5	3,2	105	2266
AEA-SP	5	370	2,00	0,50	6,5	6,0	95	2205
SP+AEA	5	370	1,60	0,50	8,1	9,2	96	2131
Only SP	5	370	0	0,80	2,3	1,4	100	2314
Only SP	5	370	0	0,80	1,6	0,4	105	2337

Note: the shaded cells highlight mixes with "AEA5 fresh", while for unshaded cells "AEA5 pre-blended" according to the notation for admixtures given in 2.1.

Manuscript 1

Frost testing of HVFA concrete.
Part 1. Surface and internal damage

This manuscript is awaiting publication and it not included in NTNU Open

Manuscript 2

Frost testing of HVFA concrete.
Part 2. Strength, hydration and liquid transport

This manuscript is awaiting publication and is not included in NTNU Open

Part IV
Supplementary papers

Paper S-I

Effect of AEA-SP dosage sequence on air content and air void structure in fresh and hardened fly ash mortar

Shpak, A., Turowski, M., Vimo, O.P., Jacobsen, S.
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Effect of AEA-SP dosage sequence on air content and air void structure in fresh and hardened fly ash mortar

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ABSTRACT

Laboratory measurements show that varying the dosage sequence of air-entraining agent and co-polymer in the mix (SP added before, after or together with AEA) greatly affects air entrainment in fresh and hardened fly ash mortar. Image analysis shows a good correlation between total air content and the air void spacing factor, though with a somewhat lower specific surface when SP is added together with AEA. Foaming measurements on the same materials and dosage sequences were therefore found less useful for studying the effect of admixture combinations. More work, including AVA measurements, is in progress to learn how to combine AEA, SP and fly ash in concrete.

Key words: Admixtures, Fly ash, Air entrainment, Testing.

1. INTRODUCTION

1.1 General

Production of frost durable fly ash (FA) concrete with a stable and protective air void system has often proven to be difficult. The problem has been ascribed to the variable carbon content in the fly ash causing variations in the required dosage of air entraining agent (AEA). Trial mixing to ensure quality output is therefore unavoidable even for batch-to-batch variations in fly ash.

The problem can hypothetically be resolved by reducing the number of sorption sites on carbon before the AEA encounters them. We think superplasticizer (SP) could block access of AEA to some carbon in one of the AEA-SP combinations. Previous measurements have shown large effects on foaming in cement-fly ash water slurries of various SP/AEA combinations and dosage sequences [1]. The foam study indicated that a combination with SP drastically affects the adsorption kinetics. The same materials from the foam study [1] were also used to study the effect of addition of the admixtures on air entrainment [2].

The sequence of the addition of SP and AEA in concrete has been debated among practitioners for a long time, but the authors know of no experimental studies of SP-AEA dosage sequence in fly ash concretes in the literature. For regular OPC concrete, some authors [4], with reference to [7], suggest adding AEA after blending SP in the mix to give a stable air-void spacing factor; others [3] say that SP should be added after AEA, providing time for AEA to precipitate. No *standards*, *committees,* or *guidelines* specify the AEA-SP interaction. *In the industry*, SP-AEA dosage sequence practice varies due to the limitations of the concrete plant, economic reasons, or the producer's or client's established practice. The concrete producers reviewed recommend that AEA is added either before or simultaneously with SP in the concrete mixes containing either pre-blended or separately added FA. The need for real knowledge about AEA and SP interaction in FA concrete is growing with the increased need for high volume fly ash concrete. *The scope of this work* was to investigate air void content and structure from laboratory mortar mixes where both the type of AEA and the dosage sequence of AEA- and a co-polymer SP were varied.

2 EXPERIMENTS

2.1 Mixing sequences and materials

Table 1 shows the 5 (five) admixture combinations and mixing sequences chosen based on experience with Foam Index testing:

Table 1 – Admixture combinations and mixing sequences.

Series ID	Admixture	Mixing sequence
	No admixture	1 min dry materials, 3 min water
1	Only AEA	1 min dry materials, 3 min water+AEA
2	AEA then SP	1 min dry materials, 2 min water+AEA, 1 min SP
3	SP then AEA	1 min dry materials, 1 min ½ water+SP, 2 min ½ water+AEA
4	SP with AEA	1 min dry materials, 3 min water+AEA+SP

2.2 litre mortar mixes were prepared in a Hobart table mixer. The volume fractions of filler-modified paste (all liquid, admixture, binder and mineral filler with particle size < 125 microns) were 330 and 400 litres/m^3 mortar, corresponding to normal and rich concrete mixes. The w/(c+FA) ratio = 0.46 for the 400 series and 0.57-0.63 for the 330 series [2]. FA/(c+FA) = 0.35 for all mixes. Two different anionic AEAs were used: AEA5 was based on synthetic tensides and tall oil derivatives, whereas AEA4 was an olefin sulfonate. The SP was an acrylic polymer. The cement used was a CEM I with a Blaine of 396 m^2/kg, additional low lime fly ash with 1.74 % carbon and a Blaine of 334 m^2/kg, and a limestone filler with a Blaine of 362 m^2/kg was used. All powders were supplied by Norcem-Heidelberg in Brevik, Norway. For details about admixtures and powders, see [1]. The aggregate was NS3099 standard Norwegian granitic 0-8 mm sand supplied by Norsk Stein Årdal.

2.2 Fresh and hardened mortar measurements

In the fresh mortar, the following measurements were made for each series: slump (40/80/120 slump cone on plexiglass plate) after 5 minutes, density after 8 minutes, pressure meter (1 L for mortars) after 9 minutes, and casting of 3 specimens 40 x 40 x 160 mm^3. All the mixes for AEA5 were reproduced at least twice, whilst mixes with AEA4 were made once.

In the hardened mortar, we measured the total air content from the demoulding density and using the PF-method, and air void parameters using image analysis (IMA) [6]. The samples were polished, inked, air voids filled with 2-4 μm $BaSO_4$ powder, scanned, and analysed after 90-120 days of storing in water.

3 RESULTS AND DISCUSSION

3.1 Fresh mortar measurements

Table 2 shows the 5 (five) different mortar mixes and their workability values.

Figure 1 shows the effect of AEA-SP dosage sequence on the total air content in the fresh mortar, when the dosage of AEA [% of binder] is held constant, and the corresponding Foam Index (FI) values from [1] with the same binders and admixtures. For the mixing sequences 2-4, the workability was kept constant at 60±10mm for the 330 series and 100±10mm for the 400 series respectively. FI measurements in squares above the bars show either the number of drops of SP to stop foaming or the time in seconds of stable foam (with a requirement at 45 sec).

Table 2 – Properties of the fresh mortar.

Mix	Paste volume, L [2]	w/b	Type of AEA*	AEA, [% (c+FA)]	SP, [% (c+FA)]	Slump [mm]
330	359	0.57	-	0	0	30
330	319–326	0.60–0.63	AEA5	0.7	0-0.20	20-60
400	371	0,46	-	0	0	20
400	346–373	0.46	AEA5	0.7	0-0.45	30-100
400	325**–370	0.46	AEA4	0.7	0-0.45	25-105

* AEA4 – Olefin sulfonate, AEA 5 – based on synthetic tensides and tall oil derivatives.

** 325L for series 4 (see Table 1), while series 1–3 range from 353L to 370L.

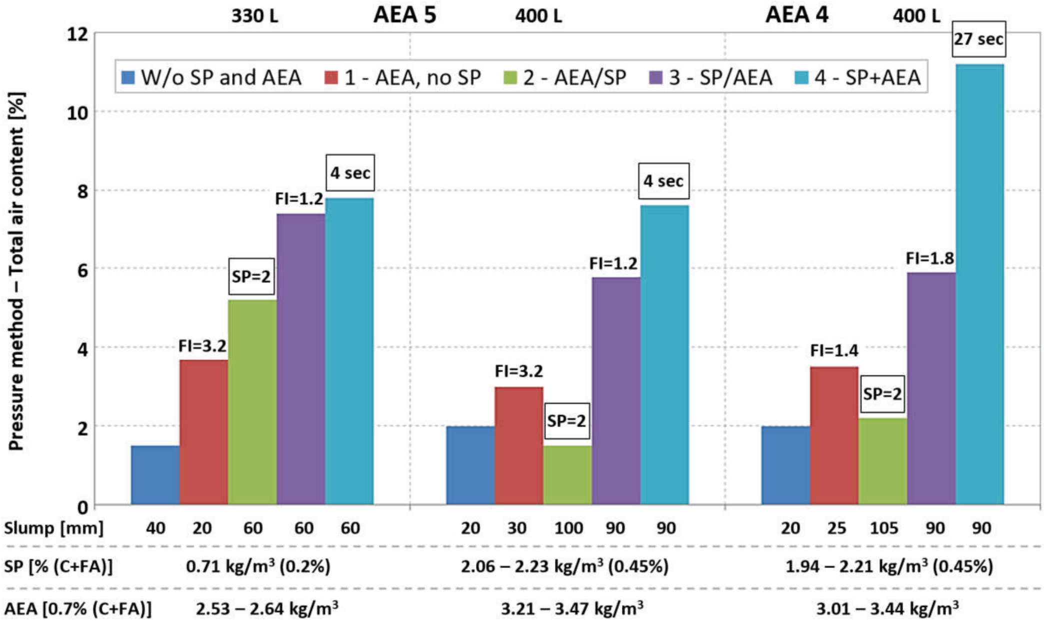

Figure 1 – Comparison of pressure meter results with FI measurements, Jacobsen et al. [1] for different matrix volumes and AEAs.

The results in Table 2 and Figure 1 show that both AEAs give big differences in air entrainment depending on the AEA-SP dosage sequence. The workability also affected air entrainment with some sort of reciprocal effect between air content and slump; most pronounced for AEA 5 when added first, see Figure1. The other series show negligible effect of workability. The highest amount of air voids is guaranteed by adding AEA and SP simultaneously (series 4), Eickschen E. and Müller C. [3] also mention this effect. When added together with SP, the pure synthetic surfactant AEA 4 shows a great contrast to the mixture of natural and synthetic components in AEA 5, but the difference is insignificant for other dosage sequences.

The results from foam index measurements do not fully reflect the properties of the mortar mixes, because this indicative test does not predict the development of the air-void system from the fresh to the hardened state. Furthermore, the very high air content when AEA and SP are added together does not correspond to the "foam killing" observed in [1]. Figure 2 shows that air content increased for series 2 (AEA before SP) meeting the requirements for the air void system, but in the fresh state (Figure 1) this combination was less promising.

3.2 Hardened mortar measurements

Mortar samples 160 x 40 x 40 mm were cast and split in two for hardened air-void analysis (IMA).

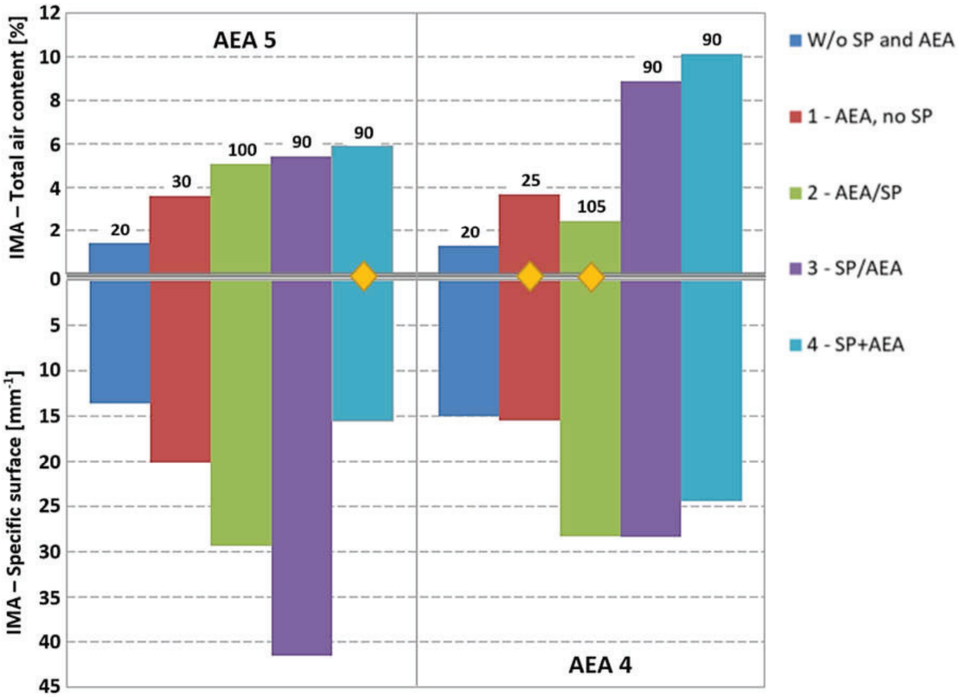

Figure 2 – IMA. Total air content and specific surface for mixes in the 400 L series with the two AEAs (the numbers over the bars show values for workability on 40/80/120 slump cone. Yellow rhombuses – done by a different operator)

Air void formation depends on several factors. Those that could possibly vary for mixtures with the two different AEAs are: the ratio of mixing time to activation time, and the susceptibility to coalescence and dissolution.

Further studies of fresh mortar with AVA are being carried out to study the impact of dosage sequence on air-void characteristics, including the ageing effect and the dosage for an acceptable air content and the resulting air void system.

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Paper S-II

Requirements and recommendations to frost durable concrete – an overview

Shpak, A., Fossaa, K.T., Jacobsen, S.

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Requirements and recommendations to frost durable concrete – an overview

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ABSTRACT

Requirements and recommendations for frost durable concrete from standards and specifications from Europe, North-America and Asia, various international organizations and construction projects are reviewed, compared and discussed. This is done based on exposure or “load” (wetness/saturation/situation, de-icers, frost, etc.), material or “resistance” (air voids, w/b, binder type, strength etc.), execution (pumping, casting, finishing, curing etc), and tests (air voids, porosity, strength, various frost tests). Finally, some practical examples of the specification together with examples of need of stringency and some occurring peculiarities in testing are given. Also the large variation in how frost durability is perceived in different parties of the decision-, planning-, execution- and commissioning process around the world are discussed and illustrated.

The full report can be downloaded from:
https://ntnuopen.ntnu.no/ntnu-xmlui/handle/11250/2598133

Keywords: concrete, constituent, proportions, execution, frost, standard, testing, performance

1. INTRODUCTION AND SCOPE

Frost deterioration of concrete is seen as progressive scaling, spalling and crumbling of particles from a surface or/and cracking of the volume of the concrete. The cracking can eventually be visible at the surface, or there can be a combination of scaling and cracking. Frost damage can happen with the simultaneous presence of low temperatures and a wet surface, and the surface damage is amplified in the presence of de-icers such as salts during exposure to frost.

With some experience into the topic, it is easy to imagine that agreeing on how to make frost durable concrete structures based on the huge amount of research results available in the literature would be both a vast and likely impossible task. The many experimental results, theories, models and test methods presented over the years have given a large number of data that are difficult to unify for practical purposes. Another approach would be to review the standards, requirements, and recommendations published internationally. Design and building based on a review of the latter three types of documents are, therefore, an easier task than to review experimental results, theories, and models. Design and building are based on loads on and resistance of the structure in question. For frost exposure, this is less clearly defined than for most types of mechanical loads on a concrete structure. Frost durability of concrete is often described as its ability to withstand repeated freeze-thaw cycles throughout a defined life of a structural element while exposed to periodical wetting and drying, often in combination with deicers (road salt, sea water, urea, etc.) It is the country standards, norms and regulations for concrete that stipulate:

- the exposure (wetness/saturation, chlorides, frost, etc.) to base the design on,
- the material requirements (air void requirements, w/b-ratio, binder composition, strength, etc.) to select for that exposure,
- the production techniques and rules (placing arrangements, finishing, curing) to apply for making the concrete frost resistant and
- the test methods (air voids, frost tests, porosity, strength, etc.) for the final product to confirm compliance.

The approach to frost durable concrete varies much from country to country. Numerous committees and unions with their sets of recommendations in addition to national standards have worked with this topic. Selection of the reviewed documents in this paper is limited to standards and recommendations from the USA (ACI, AASHTO, ASTM), Canada (CSA, BNQ), Norway (NS-EN), Sweden (SS, SIS), Danmark (DS), Germany (DIN, ZTV, BAW), Russia (SP, GOST, SNiP) and China (GB/T). The scope is to give an organized and systematic overview of the documents relevant to frost exposure requirements and recommendations including examples for application.

2. DOCUMENTS, EXPOSURE, MATERIALS PROPERTIES, EXECUTION AND TESTS

Based on the available documents reviewed [1] it seemed most convenient to use the following division given in table 1:

Table 1 - List of the main tables in [1]

	Table 2	Overview of the documents included in the review
Load	Table 3a	Classification for freeze-thaw exposure conditions. LOAD
	Table 3b	Summary of exposure classes from the reviewed standards and specifications
Resistance	Table 4	Material requirements. RESISTANCE
Execution	Table 5	Production and execution of concrete works. Requirements and recommendations
Tests	Table 6a	Tests for frost durability – material characterization
	Table 6b	Tests for frost durability – freeze-thaw tests
	Table 7	Overview of requirements for frost durable concrete

2.1 Documents

Table 2 in [1] is a rather large 1-page table, yet limited to only 53 different documents from Europe, North-America and Asia divided into exposure classes, material requirements, production- and execution-standards and recommendations, and freeze-thaw tests. By going into all sorts of details this could, of course, have been a much higher number of documents since such standards, specifications, etc. link to other technical documents. However, the cited 53 documents include the central national standards, requirements from professional organizations such as ACI and large construction owners such as road- and bridge authorities and oil companies. We, therefore, think Table 2 in [1] represents a broad overview and is very useful to the parties of the decision-, planning-, execution- and commissioning process.

2.2 Load (=exposure) and Resistance (=material)

Tables 3a, 3b and 4 in [1] make up a main body of the work. They show the big differences in how frost exposure and material parameters central to obtain durability against frost are seen among different parties in the building process worldwide. Some of the differences, particularly for exposure, pertain to real differences in exposure. A sort of consensus seems to be division onto wetness and to what extent deicers are present. Other differences could be linked to local perceptions and practices for how to obtain frost resistant materials. In general water/binder ratio, strength and air entrainment are basic parameters that there is more agreement on whereas how to use supplementary cementitious materials seems to be more different, difficult or not treated.

2.3 Execution – tests on fresh concrete

Table 5 in [1] is an effort to give an overview of another vast but more practical topic. Our purpose with this has been to help the reader to focus on the main operations of concrete works: mixing, transportation/delivery of fresh concrete, placing and finishing, surface protection and curing. Inevitably, this also includes some tests, eg. fresh concrete sampling and measurements at mixing and delivery/site/after casting and finishing operations are done. The outcome depends on execution, so the question of how to characterize this is obviously difficult, and we have tried to show how for example sampling of air void measurements vary, and the possible methods. Also, some fresh density- and workability tests are listed.

2.4 Tests

Table 6a in [1] is a rather "small" 1-full-page table giving an overview of indirect tests: These include hardened air void content, Protective Pore ratio (PF), i.e. the air void volume as a fraction of total porosity and air void spacing and total fresh air void content. For each country the table lists the relevant performance- (= freeze/thaw-) tests and acceptance criteria for those relative few countries that have such links. Hence, this table shows the testing tools available for requirements and recommendations based on laboratory testing.

Table 6b in [1] is much larger (3 pages) giving an overview of the main frost test methods worldwide. Each of the 3 pages is split in: Samples, Freeze-thaw cycles, Test set-up and Expression of test results. This makes it easier to get an idea about the great difference in how these tests work and how differently they express frost damage. So, in addition to the big difference between frost damage when expressed as surface damage and (internal) cracking, there are large variations in how to characterize these two forms of damage. Scaling can be rated visually, expressed as dried particles lost from the surface or as overall mass loss or -change of remaining specimen. Internal cracking is rated by relative dynamic modulus measured in various ways (resonance frequency, ultrasonic pulse velocity), length change measured in various ways or loss of strength. In addition, we believe there are large differences in how scaling and cracking are provoked in the different tests as well as to what extent the two forms of damage can contribute to each other, for example amplify each other.

3. EXAMPLES OF RECOMMENDATIONS AND REQUIREMENTS

3.1 Frost exposure in Europe - Norway

In Table 2 below an excerpt of the first part of Table 7 in [1] is shown generally for Europe and specifically for Norway of how exposure description and material proportions can be combined to give a specification. Table 7 in [1] is a full 5-page table giving a complete overview of requirements and recommendations worldwide with the information organized for each country into exposure class, material requirement, laboratory tests, and execution.

Table 2 – Excerpt from Table 7 in [1]

Country / Organization, Project	Exposure class, area, decisive parameter		Material requirements										
			Max w/c (effective w/c)	Min cement (binder) content, kg/m³	Max (Min) SF content, %	Max FA content (Max Fa/C-ratio), % or kg/m³	Min air content in fresh / *hardened*** concrete (for aggregate D_{max}, mm), %	Max electric conductivity, Coulombs	Frost resistance, cycles (frost res. class or min. durability factor,%)	Quality of macro-porosity in hardened concrete: Min specific surface, mm²/mm³	Quality of macro-porosity in hardened concrete: Max spacing factor (single results) L, mm	Min concrete comp. strength (strength for cement), MPa or class	Water impermeability class (Durability class)
General Europe EN 206:2013	XF1		0,55	(300)*	11	(33)						C30/37	
	XF2		0,55	(300)*	11	(33)	4					C25/30	
	XF3		0,50	(320)*	11	(33)	4					C30/37	
	XF4		0,45	(340)*	11	(33)	4					C30/37	
Norway NS-EN 206:2013 +NA:2014	XF1		0,60	(250)*		(35)							M60
	XF1		0,45	(300)*		(35)							M45
	XF1		0,40	(330)*	(6)	(35)							M40
	XF1	XF2 XF3	0,45	(300)*		(35)	4						MF45
	XF1	XF4	0,40	(330)*	(6)	(35)	4						MF40

Now, to be stringent about the example in Table 2 a few points could be mentioned. Normally, for XF4 to make a concrete that will also pass the severe European EN-TS 12390-9 performance test with 3 % NaCl on sawn surfaces, additional requirements could be needed. More specifically this would be to require air void spacing factor less than approximately 0.2 mm in the finished cast specimen. There could also be cases where concrete mixes pass the severe test without air entrainment. Furthermore, certain SCMs react very differently to carbonation than OPC and other SCMs, simultaneous internal cracking during scaling testing would accelerate scaling and so on. The EN-TS 12390-9 test is known for being very severe, so in practically all cases where concrete passes this test, it will also be durable under similar severe field conditions. Now similar peculiarities can be seen for other countries and other tests, such as for example what is the relevance that scaling occurs sometimes in the ASTM C666 Procedure A test, even when there is no internal damage?

3.2 Frost durability as perceived in different countries

If one should list down the requirements for concrete in a certain exposure from material requirements and testing to execution, quality assurance and handover, the results in different countries will vary in parameters, numbers, and level of detailing, see Figure 1.

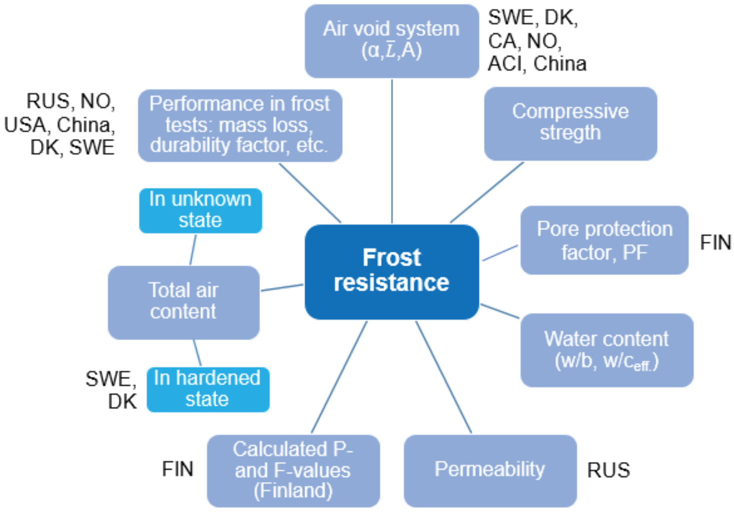

Figure 1 - The meaning of frost resistance in different countries

Figure 1 shows, concerning material requirements, how variable the definition of frost-resistant concrete can be, depending on which country one is going to design and build the structure in.

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Paper S-III

Frost testing of HP/HVFA concrete for severe offshore conditions

Shpak, A., Jacobsen, S.

Concrete in arctic conditions, Proceedings from a Nordic workshop no.16
NTNU, Trondheim, 2019

Frost testing of HP/HVFA concrete for severe offshore conditions

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ABSTRACT
In the rim of DaCS (Durable advanced Concrete Solutions) project, financed by Norwegian research council and the industry partners, one of the work packages is associated with production and documentation of frost durable low-carbon concrete suitable, in addition, for offshore and arctic exposures conditions. A number of freeze-thaw experiments with high-volume fly ash concrete were carried out. This paper presents some preliminary results made to proceed in the understanding of : 1.how internal and superficial frost damage occur in ASTM C666 procedure A, rapid freeze-thaw testing in fresh water (by far the most common way of frost testing concrete), 2. how this is related to water uptake during curing and during subsequent freeze-thaw, and 3. how the air entrainment contributes to frost resistance in this test. The results show that the water uptake during curing, presumably due to self-desiccation and air void filling, is much lower than the accelerated uptake due to the wet freeze/thaw. Furthermore, air voids in FA-concrete seem to be filled less than inwet curing. A surprisingly high scaling was observed for FA-concrete in these fresh water tests even without any internal damage and cracking and scaling were accelerated as both occurred.

Key words: Frost resistance, Fly ash, Air voids, Freshwater, Scaling, Cracking

1. INTRODUCTION

Concrete has proven to be the only material used in large offshore structures in subarctic exposure that could withstand severe freeze-thaw and ice loads and maintain longevity, and recently the developers of White Rose oil field on Canadian Grand Banks opted for concrete for a 120m tall semi-submersible gravity-based structure [http://westwhiteroseproject.ca/]. Increasing demand for low-carbon binders leads to an increasing need for technology on how to produce frost-resistant low-carbon concrete for these kinds of severe conditions. Fly ash concrete was found capable to withstand severe freeze-thaw cycles with moderate Fly ash dosage and when the concrete is properly air-entrained and cured [1]. However, for high fly-ash replacement and little curing frost durability can be a problem [2].
Rapid freeze-thaw testing in fresh water ASTM C666 procedure A [3] has been observed to cause surface scaling (no deicing salt) and internal damage. The last we can relate to the accelerated water uptake during freeze-thaw [4], which was found to continue during cycling

and to correlate to internal cracking [5]. The specimens of ASTM C666 are cured in lime water continuously until the start of freeze-thaw and, hence there is an initial water uptake that is caused by self-desiccation and possibly some air-void filling. This paper presents new findings on the water uptake due to self-dessication and freeze/thaw and its relation to frost damage.

2. EXPERIMENTS AND MATERIALS

All concrete mixes were exposed to ASTM C666 Proc A testing with simultaneous measurements of RDM (Relative Dynamic E-Modulus), scaling and weight increase due to water absorption. The compensation of loss of evaporable water in the scaled mass was done by multiplying dried scaled mass with the evaporable water content of concrete measured by the PF-testing [6] and corrected for the increased paste fraction at the surface.

Table 1 shows some key mix details, air-void system, water uptake in curing and compressive strengths. The mix code refers to w/b-ratio, FA/b-ratio and the air-entrainment, i.e. 0.40-35 A means w/b=0.40, FA/b = 0.35 and air-entrained, while 0.40-35 0 is a code for non-air-entrained mix. All mixes contain 4% of silica fume (SF) of binder, the slump variation was 190 - 220 mm. The w/b = 0.293 mixes were made to represent w/c = 0.45 for zero pozzolan hydration, i.e. opposed to a reference mix 0.45-0 A.

As we see, most entrained air-void systems were very effective in terms of both air-void spacing [4] and Pore Protection Factors [6]. The absorption during curing is lower for the fly ash concrete compared to the previous measurements on OPC and OPC+SF concrete [7]. The absorption during curing, similar strength and the air content of 0.293-35 A and 0.45-0 A indicate that Fly Ash (FA) did not contribute to hydration the first 14-28 days (Analyses of hydration with other methods are underway). Furthermore, the levels of absorption in Fly Ash concrete are low compared to previous experiences with this simple method on OPC and OPC+SF specimens.

Non-air-entrained mixes were produced to see whether frost-durable low-w/b FA-concrete could be made without all the difficulties associated with air-entrainment. There is evidence of concrete without air being frost durable, however, it had low w/b, at least 10% SF and no FA [8].

Table 1. Properties of fresh and hardened concrete

Mix	Paste volume [L]	Air content, fresh[1] [%]	Air content, hardened[2] [%]	Spacing factor[2] [mm]	PF [%]	Absorption during curing [vol% paste] ASTM cylinder				Compressive strength [MPa]		
						ASTM cylinder	Cubes[3] 100 mm					
						14d[4]	28d	91d	1y	28d	91d	1y
0.40-35 A	265	5,6	4,7	0,24	30,2		6,8	7,4	8,5	59,3	71,6	81,2
0.40-35 0	263	1,2	2,8	0,79	16,0		6,1	7,2	7,8	73,2	90,2	101,9
0.45-35 A	266	5,8	4,1	0,18	32,5		5,9	7,0		50,4	63,3	
0.45-35 0	263	1,3	2,0	0,68	17,0		6,5	7,0		67	81,9	
0.293-35 A	266	5,9	5,9	0,20	36,1	5,3	5,6	6,2		81,5	93	
0.293-35 0	262	2,0	1,8	0,63	17,6	5,1	6,0	6,8		98,8	114,9	
0.45-0 A	270	5,1	6,2	0,30	31,7	6,2	6,9	8,3		77,2		

[1] Density method
[2] Acc. to Fonseca et al. and ASTM C457, scanning - 3200ppi
[3] Measured on cubes 100 x 100 x 100 mm^3
[4] Beginning of ASTM C666 testing, measured on cores 300 mm D~95-100mm

3. RESULTS AND DISCUSSION

Figure 1 shows internal damage and surface scaling during freeze-thaw cycling. From the results, it is clear that a proper air-void system with L ≤ 0.24 mm protects the fly ash concrete whereas the OPC mix 0.45-0 A with L = 0.30 mm suffers internal damage.
We could clearly see that the drop in RDM is accompanied by a sudden increase in the scaling rate. From that case, for "bad concrete", internal cracking and scaling could possibly be interrelated. For stronger concretes with low water-to-binder ratio (0,293-35) or well-cured fly ash concretes (0.40-35 water-cured for 1 year) the cracking and scaling results do not complement one another. Looking at the dashed lines for non-air-entrained concretes, we can see that the resistance to freeze-thaw is improved with increased compressive strength of concrete (also valid for the air-entrained concretes), which is obtained either by reducing w/b-ratio (0.293-35 0) or by prolonging water-curing time (0.40-35 0 1y). However, none of the mixes without AEA had even passed 150 cycles with RDM being above 80%.
The relatively high scaling is surprising since the test is done in fresh water and the scaling levels are quite high after 300 cycles for the worst specimens.
Despite high PF-value for 0.45-0 A, concrete is not frost resistant.

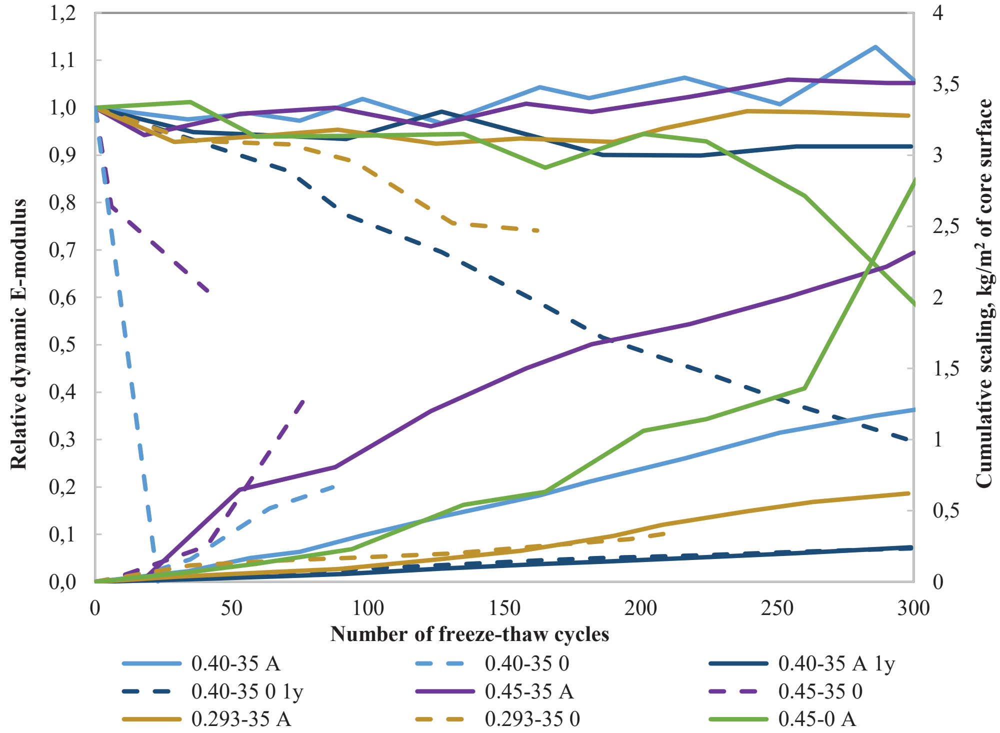

Figure 1. Relative dynamic E-modulus and cumulative surface scaling in freshwater freeze-thaw test ASTM C666 procedure A. Average values for 2 cores

Figure 2 shows the cumulative water uptake during the freeze-thaw test, compared to absorption during curing in limewater. The uptake by freeze/thaw is much larger than that due to self-desiccation. Water uptake for a reference concrete without FA resonates with the drop in RDM shown above. On the contrary, the non-air-entrained mix 0.293-35 0 failed at 164 cycles (pulse was not sent through), but it did not affect water uptake and scaling curves for measurements for another 40-50 cycles. Mixes with FA of the same w/(C+SF)-ratio as in reference concrete show lower water uptake and lower absorption during curing, probably due to known lower chemical shrinkage of the FA concretes. It is interesting to note that there is a negligible difference in uptake values between non- and air-entrained mix (as in the surface scaling), meaning that, unlike SF, FA does not cause increased air void filling in water curing.

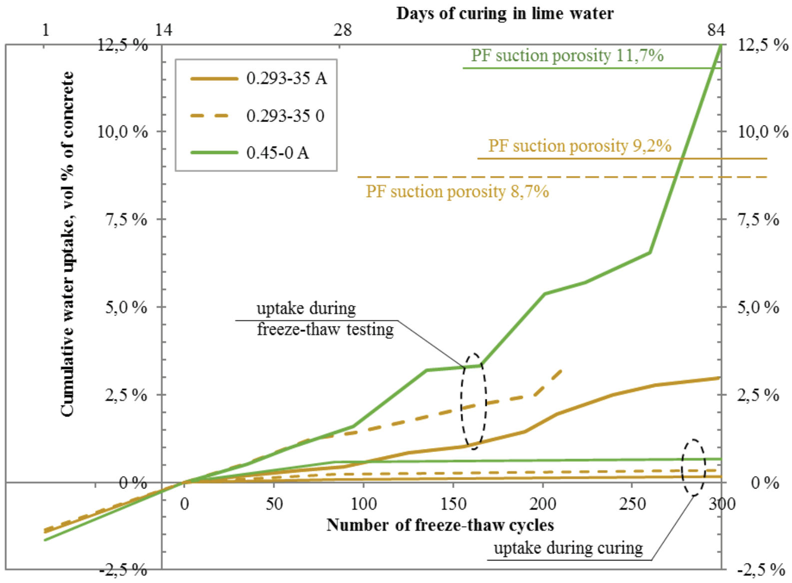

Figure 2. Cumulative water uptake during freeze-thaw testing in ASTM C666 procedure A and water absorption during curing (for selected mixes). Average values for 2 cores.

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Paper S-IV

Frost testing of HP/HVFA concrete for severe offshore conditions

Shpak, A., Fossaa, K.T., Jacobsen, S.

Durable Concrete for Infrastructure under Severe Conditions,
Proceedings of Lorcenis conference
Ghent, 2019

Frost testing of HP/HVFA concrete for severe offshore conditions

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Abstract
In the Durable Advanced Concrete Solution (DACS) project lead by Kvaerner and supported by a group of Norwegian concrete industries and The Norwegian Research Council the production and documentation of frost durable concrete for arctic and subarctic marine conditions is studied. The area encompasses frost exposure conditions and national standards relevant for the Arctic region. A series of HP/HVFA concretes were therefore subjected to various severe freeze-thaw tests of degradation due to internal cracking and surface scaling. Two of the most used methods, ASTM C666, procedure A for rapid freeze-thaw in water and CEN/TS 12390 for surface scaling in presence of 3 % NaCl solution, were used and extended to investigate how cracking, scaling and saturation progress at standard (-20 C) and very low (-50 C) temperatures in such severe conditions. This paper presents some preliminary results made to proceed in the understanding of how internal frost damage and surface scaling occur in rapid freeze-thaw testing in water (by far the most common way of frost testing concrete), how this relates to water uptake during curing and during subsequent freeze-thaw and the effect of air entrainment in this kind of frost testing.

Keywords
Frost resistance, Fly ash, Air voids, Arctic Exposure, Freshwater scaling

1. Introduction

Concrete has proven to be the only material used in large offshore structures in subarctic exposure that could withstand severe freeze-thaw and ice loads and maintain longevity, f.ex. Hibernia (CA), Sakhalin-I (RU), Hebron (CA) offshore gravity base structures, Confederation Bridge (CA), Øresund bridge (SWE/DK). Concrete was again chosen for a 120m tall semi-submersible foundation of White Rose platform that is being built in NF, CA [http://westwhiteroseproject.ca/]. With the increasing demand for low-carbon binders, there is also an increasing need for technology on how to produce frost-resistant low-carbon concrete for these kinds of severe conditions. Fly ash concrete has to a certain extent been found capable to withstand severe freeze-thaw cycles: with moderate Fly ash dosage, when properly air-entrained and cured [1], but for high fly-ash replacement and little curing frost durability can be a problem [2].
Rapid freeze-thaw testing in water has been observed to cause surface scaling from concrete (even in the absence of deicing salt) and the internal damage has been found to relate to the accelerated uptake of water during freeze-thaw [3]. Furthermore, the accelerated water uptake in ASTM C666 procedure A testing [4] has been found to continue during cycling and to correlate to internal cracking [5]. The specimens of ASTM C666 are cured in water continuously until the start of freeze-thaw and, in this period, there is an initial (isothermal) water uptake that is caused by self-desiccation and possibly some air-void filling. This paper presents some new findings in this field.

2. Experiments and materials

All concrete mixes were exposed to ASTM C666 Proc A testing with simultaneous measurements of RDM (Relative Dynamic E-Modulus), scaling and weight increase due to water absorption. The compensation of loss of evaporable water in the scaled mass was done by multiplying dried scaled mass with the evaporable water content of concrete measured by the PF-testing and corrected for the paste fraction at the surface.

Table 1 shows a few key mix design results, air-void system, water uptake in curing and compressive strengths. The mix code refers to w/b-ratio, FA/b-ratio and the air-entrainment, i.e. 0.40-35 A means w/b=0.40, FA/b = 0.35 and air-entrained (0.40-35 0 is a code for non-air-entrained mix). All mixes contain 4% of SF in a binder, and they were produced with the slump variation 190 - 220 mm. The w/b = 0.293 mixes were made to represent w/c = 0.45 for zero pozzolan hydration, i.e. as a “rival” to 0.45-0 A. As we see, most entrained air-void systems were very effective in terms of both air-void spacing [4] and Pore Protection Factors [6]. The absorption during curing is low for the fly ash concrete compared to previous measurements on OPC and OPC+SF concrete [7]. The absorption during curing, similar strength and the air content of 0.293-35 A and 0.45-0 A indicate that Fly Ash (FA) did not contribute to hydration the first 14-28 days. Analyses of hydration with other methods are underway. Furthermore, the levels of absorption in Fly Ash concrete are low compared to previous experiences with this simple method on OPC specimens. It should also be mentioned that air voids do not increase the absorption as marked in FA- as in OPC- a silica fume concretes.

Table 1. Properties of fresh and hardened concrete

Mix	Paste volume [L]	Air content, fresh[1] [%]	Air content, hardened[2] [%]	Spacing factor[2] [mm]	PF [%]	Absorption during curing [vol% paste] ASTM cylinder	Cubes[3] 100 mm			Compressive strength [MPa]		
						14d[4]	28d	91d	1y	28d	91d	1y
0.40-35 A	265	5,6	4,7	0,24	30,2		6,8	7,4	8,5	59,3	71,6	81,2
0.40-35 0	263	1,2	2,8	0,79	16,0		6,1	7,2	7,8	73,2	90,2	101,9
0.45-35 A	266	5,8	4,1	0,18	32,5		5,9	7,0		50,4	63,3	
0.45-35 0	263	1,3	2,0	0,68	17,0		6,5	7,0		67	81,9	
0.293-35 A	266	5,9	5,9	0,20	36,1	5,3	5,6	6,2		81,5	93	
0.293-35 0	262	2,0	1,8	0,63	17,6	5,1	6,0	6,8		98,8	114,9	
0.45-0 A	270	5,1	6,2	0,30	31,7	6,2	6,9	8,3		77,2		

[1] Density method

[2] Acc. to Fonseca et al. and ASTM C457, scanning - 3200ppi

[3] Measured on cubes 100 x 100 x 100 mm^3

[4] Beginning of ASTM C666 testing, measured on cores 300 mm D~95-100mm

3. Results and discussion

Figure 1 shows internal damage and surface scaling during freeze-thaw cycling. From the results, it is clear that a proper air-void system with L ≤ 0.24 mm protects the fly ash concrete whereas the OPC mix 0.45-0 A with L = 0.30 mm suffers internal damage. However, we could also clearly see that the drop in RDM is accompanied by a sudden increase in the scaling rate. From that case, for “bad concrete”, internal cracking and scaling could possibly be interrelated. For stronger concretes with low water-to-binder ratio (0,293-35) or well-cured fly ash concretes (0.40-35 water-cured for 1 year) the cracking and scaling results do not complement one another.

Looking at the dashed lines for non-air-entrained concretes, we can see that the resistance to freeze-thaw is improved with increased compressive strength of concrete (also valid for the air-entrained concretes), which is obtained either by reducing w/b-ratio (0.293-35 0) or by prolonging water-curing time (0.40-35 0 1y). However, none of the mixes without AEA had even passed 150 cycles with RDM being above 80%.

The relatively high scaling is surprising since the test is done in fresh water and the scaling levels are quite high after 300 cycles for the worst specimens.

Despite high PF-value for 0.45-0 A, concrete is not frost resistant.

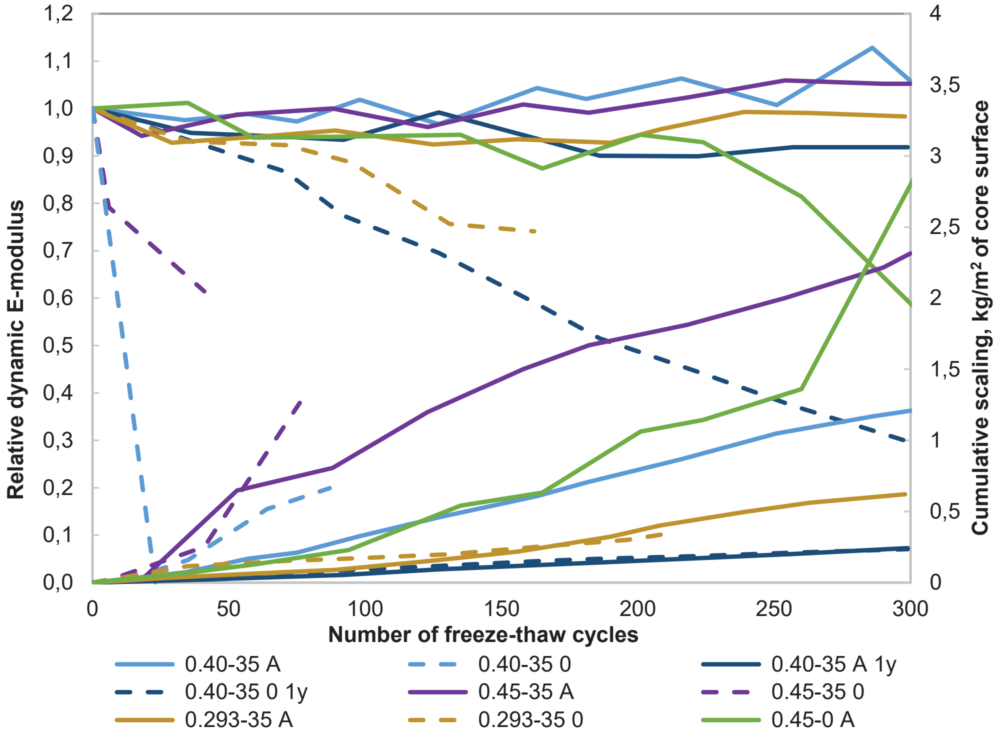

Figure 1. Relative dynamic E-modulus and cumulative surface scaling in freshwater freeze-thaw test ASTM C666 procedure A. Average values for 2 cores

Figure 2 shows water uptake during curing in water (negative numbers) and water uptake during rapid freezing and thawing (positive numbers). All absorption values have been calculated as the volume fraction of concrete in the corrected mix recipes, i.e. proportions corrected for measured density and air content of fresh concrete, (in addition to the mentioned correction for scaling). It was assumed in the calculations that the volume of the cylinders under freeze-thaw remains unchanged which introduces only a small error in the vol-% uptake [7]. The absorption values during freeze-thaw are highest for the cracked concretes, presumably due to that cracks are filled as the damage evolves in the non-air entrained specimens. The prolonged water curing for 1 year reduces water uptake during freeze-thaw. The most surprising feature of the absorption during freeze-thaw is that for two of the surviving concrete mixes (045-35A and 040-35 A) the absorption is very high compared to the absorption during water curing: in the order of 9,5 and 5 % of concrete volume after 300 cycles, whereas in the foregoing 13 days of water curing the uptake is only in the order of 2 %. How can this be? Also interesting to note that low water uptake for 0.40-35 0 1y and 0.293-35 0, similar to air-entrained companions, does not guarantee resistance to internal damage (see Figure 1). We are now investigating furter these features of HV FA concrete.

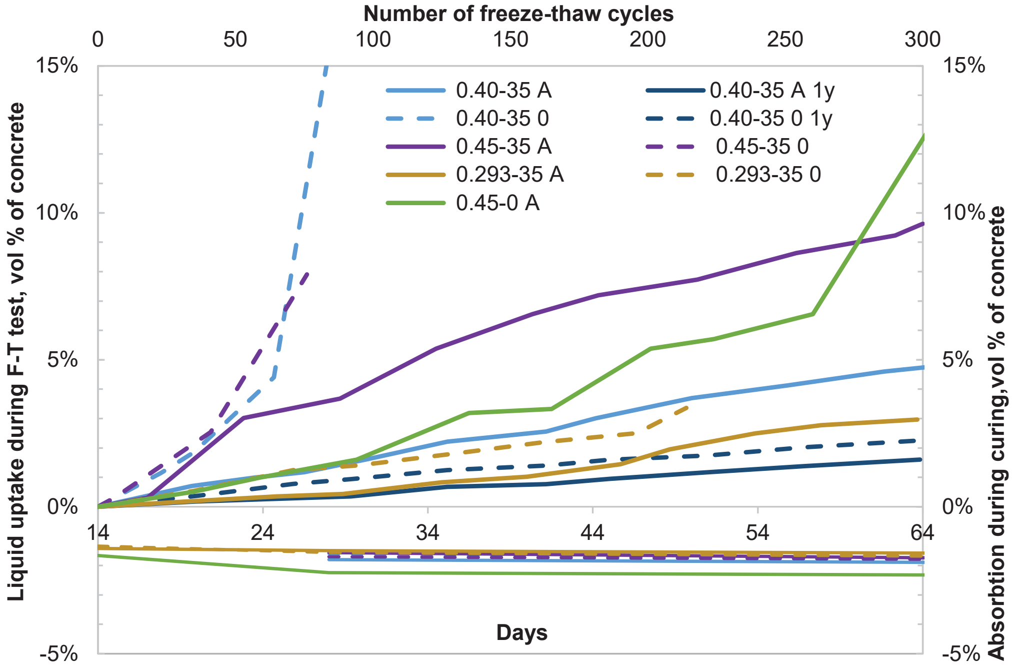

Figure 2. Cumulative liquid uptake in rapid freeze-thaw ASTM C666 procedure A and water absorption during curing. Average values for 2 cores.

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Paper S-V

Cracking in High Volume Fly Ash Concrete specimens during the European salt-frost slab test: dilatometry measurements and consequence for surface scaling

Shpak, A., Jacobsen, S.

Design and construction of sustainable concrete structures: causes, calculation and consequences of cracks
Proceedings from a Nordic workshop no.17, Oslo, 2019

Cracking in High Volume Fly Ash Concrete specimens during the European salt-frost slab test: dilatometry measurements and consequence for surface scaling

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ABSTRACT

The European salt-frost slab test CEN/TS 12390-9 measures the resistance of concrete to surface scaling under the combined attack of frost and deicer salt. Frost damage can also be measured as internal cracking. For that length change measurements with invar steel dilatometers equipped with LVDTs in a low – temperature chamber were taken during repeated freeze-thaw cycles in the slab test. Some preliminary results are shown on the effect of environment (minimum temperature, liquid uptake during cycling) and material (air voids, fly ash, water-binder ratio) on cracking expressed as residual length change and its consequence for surface scaling damage.

Key words: Frost testning, salt scaling, internal cracking, fly ash

1 INTRODUCTION

Concrete exposed to freezing and thawing can be tested in accordance with very different freeze-thaw testing procedures used in different parts of the world. Alongside with harsh freeze-thaw test in freshwater ASTM C666 procedure A, which is often used for offshore and bridge concretes to identify internal cracking susceptibility, concrete can also be tested for resistance to surface damage in the presence of deicing salt [1]. The latter test was modified by using in-house-developed unique leakage-proof preparation system adapted for simultaneous measurements of internal cracking and surface damage to investigate the consequence of cracking for surface damage in concrete with high amount of fly ash.

2 METHODS

Table 1 shows some relevant properties of hardened concrete. The mix code is translated as follows: 0.45-35 **A** means w/b=0.45, FA/b = 0.35 and air-entrained (0.45-35 **0** is a code for non-air-entrained mix). All mixes contain 4% of SF in a binder. The w/b = 0.293 mixes were made to represent w/c = 0.45 for zero pozzolan hydration, i.e. in comparison with non-FA 0.45-0 A. All the mixes were exposed to standard salt-frost sclaing slab test. Mixes 0.293-35 were in addition exposed to standard slab salt-frost testing [1] and “Arctic” test (36h long “Arctic” cycles - +20C°…-52C° with heating and cooling velocities from the slab test) after 4 months of storage in lime water.

Table 1. Properties of hardened concrete

Mix	Air content, hardened [%]	Air void spacing factor [mm]	PF [%]	Comp. strength [MPa] 28d	Comp. strength [MPa] 91d
0.293-35 A	5,9	0,20	36,1	81,5	93
0.293-35 0	1,8	0,63	17,6	98,8	114,9
0.45-35 A	4,1	0,18	32,5	50,4	63,3
0.45-35 0	2,0	0,68	17,0	67	81,9
0.45-0 A	6,2	0,30	31,7	77,2	

3 RESULTS

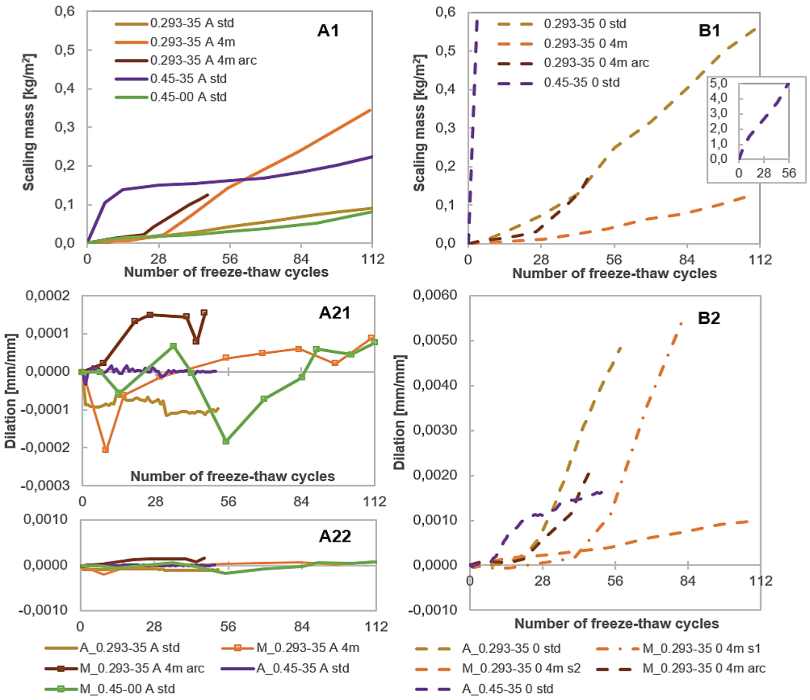

Figure 1 – Surface damage, length change, and water uptake in salt-frost scaling test:
***A** and **B** divide the concretes to air-entrained and non-air entrained; A1,B1 – surface scaling; A21,A22,B2 – dilation, where A22 shows A21 in the same scale as B2.*
**Note: In the legend for A21, A22, B2 there M and A letters in front of the mix code mean that the measurements were taken Manually and Automatically (continuously). Manual measurements of a length change of the specimens with LVDT, comparing to a length of the invar reference rod. The fluctuations of length change on A21, A22 can be related to a difference in temperature of the specimens between the measurements.*

To assess possible consequences of cracking for surface scaling damage we looked at the curvature of the accumulated scaling as a function of number of cycles in salt-frost testing. The acceleration is taking place if the ratio between mass at 56 and 28 (as per standard [1]) or at 112 and 56 cycles is more than 2. Grand average acceleration factor (GAAC) of all tests shows that non-air entrained concrete (GAAC 3,3) has larger tendency to accelerated scaling than air-entrained concrete (GAAC 2,2). Figure 1 shows scaling and internal cracking for a selection of the tested concretes.
Figure B2 shows that the non-air samples have a tendency to internal damage whereas Figure A21 and A22 show low / no internal damage for air-entrained concrete as expected. There is a tendency that concretes with the lowest w/b-ratio and a reference concrete (with "poor" air void system) show expansion to a level near strain capacity of concrete. B2 also shows that initiation of internal damage is delayed as effect of lowered w/b-ratio and prolonged curing time.
Comparing internal damage (B2) and scaling (B1) it seems that very high and accelerated scaling correlates to dilation (=internal cracking) for all non-air entrained samples. For air-entrained samples Figure A1 shows that the 2 concretes with accelerated scaling (or delayed onset of scaling?) also have larger length change but the scaling level is low and scatter of length change is possibly high. In addition to scaling and internal cracking measurements, we are looking into liquid uptake in the salt-frost testing and freshwater rapid freeze-thaw testing known as ASTM C666 procedure A for rather broader variation of concretes and curing ages.

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Paper S-VI

Salt frost scaling testing HVFA concrete to -52C: internal cracking measured with dilatometry

Shpak, A., Brun, M., Fossaa, K.T., Jacobsen, S.

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Sandefjord, 2020

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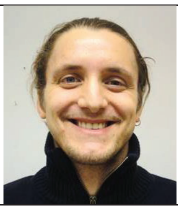	Andrei Shpak Ph.D. candidate Norwegian University of Science and Technology, Department of Structural Engineering Richard Birkelands vei 1a, NO-7034 Trondheim, Norway e-mail: andrei.shpak@ntnu.no
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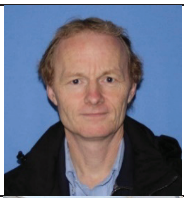	Kjell Tore Fossaa Ph.D. Kvaerner Concrete Solutions Kværner ASA Oksenøyveien 10, 1366 Lysaker, Norway e-mail: kjell.tore.fossa@kvaerner.com
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ABSTRACT

The salt-frost slab test CEN/TS 12390-9 measures surface scaling with 3% NaCl but states that any internal cracking shall be noted. Here length change measurements were made horizontally at the center of the slabs with invar steel dilatometers with LVDTs in a low – temperature chamber. Arctic freeze-thaw cycles (+20°C/-52°C) were used on well-cured HVFA concrete with w/b= 0.40 with and without air entrainment. The scaling was 0.60 and 1.28 kg/m^2 after 70 and 29 cycles on air entrained and non-air entrained specimens respectively. The dilatometry showed how the well-spaced air voids protected with no deleterious expansion whereas a huge cumulative residual expansion in the order of 1 % after 29 cycles was seen on the concrete without air voids. The huge accumulated dilation was in the order of 100 times the fracture tensile strain after 29 cycles. During individual cycles dilation larger than fracture tensile strain happened from -40 °C to -52 °C already at the first cycle and with similar features throughout the cycling resulting in a rather linear evolution of the accumulated residual strain. At later cycles large primary freezing dilation around -5°C was also observed. The total dilation within one cycle was much larger than the residual dilation indicating partly crack closing during thawing. A reciprocal effect between scaling and cracking occurred as acceleration of the scaling of the non-air entrained specimen.

Keywords: concrete, fly ash, low temperature, frost resistance, standard, testing, performance

1. INTRODUCTION AND SCOPE

The increasing focus on environmental friendly binders such as composite cements with fly ash has led to some questions about the frost durability of these, partly due to their slow hydration and some difficulties with air entrainment. One area of relevance is for concrete used in arctic conditions with very low temperatures, and to our knowledge few/no studies of frost durability of fly ash cement at very low temperatures exist.

In general frost deterioration of concrete is seen as progressive scaling, spalling and crumbling of particles from a surface or/and cracking of the volume of the concrete. The cracking can eventually be visible at the surface, or there can be a combination of scaling and cracking. Scaling damage can happen with the simultaneous presence of low temperatures and a wet surface, and the surface damage is amplified in the presence of de-icing agents during exposure to frost.

The scope of this paper is to give a limited presentation of some preliminary results of surface and internal damage measurements on well-cured specimens tested to very low, arctic temperatures from two of the mixes included in a more comprehensive PhD program [1].

2. MATERIALS AND METHODS

Two concretes with w/b = 0.40, 4 % silica fume and 33 % fly ash by weight of binder, one without and one with air entrainment, were selected for the tests presented here. Standard 15 cm laboratory cast cube specimens that had been water cured for 10 months were used. Air void systems (total air, specific surface and Powers spacing factor) were measured in fresh concrete with the AVA (air void analyser) immediately after mixing, and in the air-entrained concrete also measured delayed after 1 hour at rest. Air void systems were also measured on polished sections of hardened concrete by Image analysis (IMA). The specimens for salt frost testing were 5 cm thick slabs cut from the 15 cm cubes prepared according to the scheme of CEN/TS 12390-9 after the prolonged water curing. The scheme includes sawing and storage at 65 % RH and 20 °C while preparing the leakproof butyl dam on roughened and specially primed lateral surfaces, pre-wetting the test surface with pure water and finally freeze/thaw exposure with a 3 mm layer of 3 % NaCl. The slabs were also equipped with embedded epoxy-glued invar studs for dilatometry measurements parallel to the wet top surface during the preparation procedure. The frost cycle had the same cooling and heating rates and proportional duration of all parts of the standard CEN/TS 12390-9 cycle except for going to -52 °C and hence lasting 36 hours. An advanced Vötsch low temperature chamber was used for freeze/thaw exposure. Invar steel frames made in accordance with ASTM C671 and equipped with HBM low-temperature LVDTs used for continuous dilatometry during freeze/thaw. The dilatometry data were corrected for blank runs with invar steel rod in the dilatometer and concrete strain then calculated based on effective concrete length L. Figure 1 shows a slab with 8 mm diameter invar studs and set-up with dilatometer around an insulated slab.

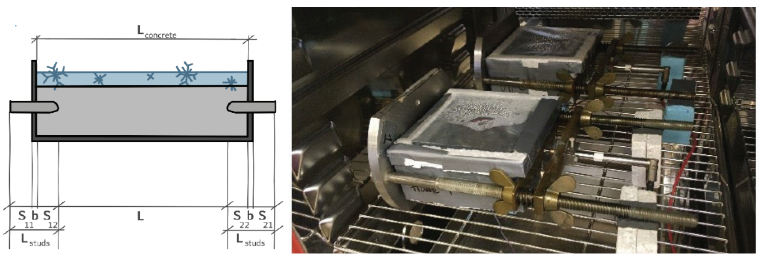

***Figure 1**. Set-up of slab test for combined salt scaling and dilatometry in low temperature chamber*

3. RESULTS AND DISCUSSSION OF FINDINGS - SUMMARY

Table 1 shows air void system of the two sets of concrete specimens with and without air entrainment. The results show a clear effect of the air void system and the measurements made with AVA and IMA detect air void parameters of similar magnitude for the two concretes. The delayed AVA measurement indicates that the entrained air void systems are very stable in the fresh concrete.

***Table 1**. Air voids in 040-33 concrets with and without air*

	Air			Non-air	
	IMA	AVA	AVA delayed	IMA	AVA
A, %	4,66	3,30	3,20	2,58	1,00
α, mm^{-1}	20,62	25,30	24,20	9,19	11,50
$\bar{L}$, mm	0,242	0,239	0,252	0,742	0,900

Figure 2 shows scaling of 0.60 and 1.28 kg/m^2 after 70 and 29 cycles of air entrained and non-air entrained specimens respectively with a clear effect of the entrained air void systems. In addition to higher scaling, the more convex curvature to the x-axis of the non-air entrained mix indicates a stronger acceleration of surface scaling damage for this concrete compared to the air entrained concrete until the experiment was terminated after 29 cycles. The air entrained concrete was tested to 60 cycles and showed a nearly linear evolution of the damage.

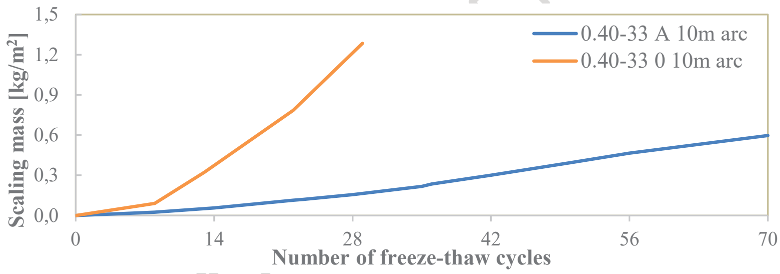

***Figure 2**. Cumulative scaling with 3 % NaCl for the two concretes tested to -52°C*

Figure 3 lefthand side shows dilatometry curves of the non-air entrained concrete. It is seen how the low-temperature freezing has a detrimental effect on the expansion of the non-air entrained concrete by freezing water at around -40 °C and further down to -52 °C.

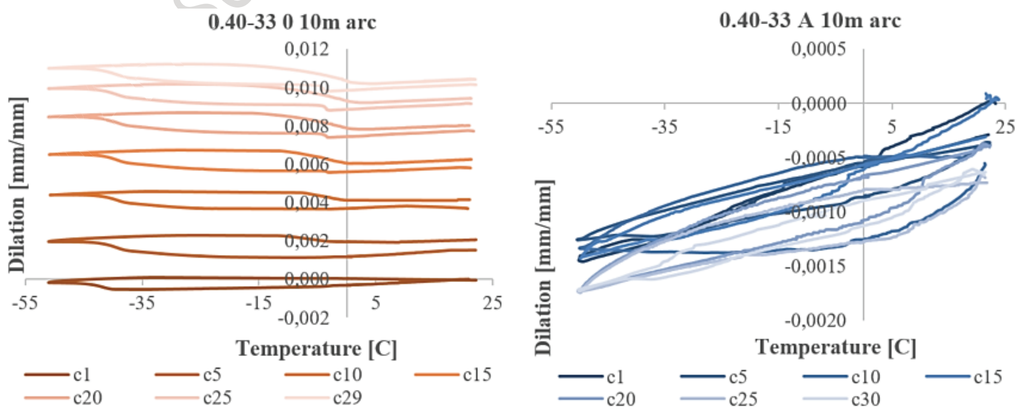

***Figure 3**. Dilatometry during freeze/thaw cycling for the two concretes tested to -52°C*

The resulting freezing dilation from this water that is non-freezable at ordinary test temperatures to -20 °C corresponds to the freezing peaks normally seen at -40°C of low temperature calorimetry curves run on saturated samples of cement paste and concrete. Such experiences from earlier studies with low temperature calorimetry to -40 °C and further down therefore show a clear ice formation around -40 °C, even when there is no or very little primary freezing just below 0°C.

The resulting huge accumulated dilation after 29 cycles in Figure 3 is in the order of 100 times the fracture tensile strain of concrete. Also during individual cycles dilation larger than fracture tensile strain were seen from -40 °C to -52 °C starting already at the first cycle and with similar features throughout the testing resulting in a rather linear evolution of the accumulated residual strain. At later cycles also large primary freezing dilation around -5°C was seen and the dilation within a complete cycle was in the order of 1000 microstrain whereas the residual strain of a complete cycle was less than half of the total loop strain indicating partly crack closing during thawing. Hence the salt scaling tests to very low temperatures show a marked difference in the porewater freezability compared to in the standard test to -20°C and its mechanical effect on the concrete is detrimental. Note that the vertical displacement between the different complete individual cycle dilation loops are to scale so there is an approximate linear progress of residual expansion as function of number of cycles.

Figure 3 righthand side shows dilatometry on the air entrained concrete and it is seen how the well-spaced air voids worked to protect with no deleterious expansion (note the different scale of the strain axis). There is even possibly a tendency of permanent contraction that so far has not been confirmed with manual length change measurements.

Looking at Figure 2 and 3 simultaneously the resistance to internal damage seems in a way to be more improved by air entrainment than does the resistance to surface damage with 3 % NaCl under the actual conditions of this test. Furthermore, possible impact of cracking on surface scaling can be observed with noticeably larger acceleration of scaling of the non-air entrained specimen during the 29 cycles of exposure to the "arctic" cycle.

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