NORDIC EXPOSURE SITES

INPUT TO REVISION OF EN206-1



WORKSHOP PROCEEDING

FROM A

NORDIC MINISEMINAR

HIRTSHALS – DENMARK

12 - 14. NOVEMBER 2008



PREFACE

This publication contains 18 papers presented at a Workshop (Nordic Mini Seminar) concerning collection of data from Nordic Exposure Sites, with the aim to submit information to the working groups concerned with the revision of the European concrete standard, EN 206-1. Many of the participants are also active in several standardisation committees, both nationally and within CEN. Hence the information and knowledge gained during this workshop can have a direct impact on the revision, particularly regarding the issue of "Equivalent Performance Concepts".

The workshop was organised by Aalborg University, Department of Civil Engineering.

In order to stimulate discussions between the participants, the Workshop was arranged as a threeday residential course, located in Hirtshals in the Northern part of Denmark, November 12 - 14, 2008.

Nordic Mini Seminars are workshops arranged solely for researchers from the Nordic Countries in order to strengthen the inter-Nordic co-operation. A few foreign specialists can however be invited. To further stimulate discussions, only participants actively contributing are invited. 72 such Mini Seminars have been held since 1975.

19 researchers from Finland, Norway, Sweden, Denmark, United Kingdom, The Netherlands and Switzerland participated in the workshop.

The present publication is Number 8 in a special series of Workshop-Proceedings of the Nordic Concrete Research.

Nordic Concrete Research (NCR) is a bi-annual publication of The Nordic Concrete Federation, presenting research and practical experience in the field of concrete technology, both from structural and material perspective. Every third year one of the publications is devoted to abstracts from the Nordic Concrete Research Meeting. (Nordic Concrete Research – Research Projects 20XX).

Papers published in NCR are normally thoroughly reviewed by three reviewers. The papers in the present proceeding have however not been reviewed in this way. Instead the authors revised their papers after the workshop, based on comments and information obtained there.

Aalborg, December 2008

Eigil V. Sørensen, Chairman

Dirch H. Bager, Editor

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INTRODUCTION

Even though there are many similarities between the Nordic countries some testing and calculating procedures are not identical. In order to be able to compare data presented in the different papers, the following information is included in all papers

- Cement notation according to EN 197-1, at least once in the paper. A list of cement notations according to EN 197-1 is given in table 2
- The content of mineral admixtures are calculated both as a percentage of the cement, and as a percentage of the amount of binder.
- The equivalent w/c-ratio is calculated according to the following equation:

$$eqv.\frac{w}{c} = \frac{w}{(c+0.4*FA+2*MS+0.6*Slag)}$$

• Compressive strengths are measured in different ways in the Nordic countries. Some equations for recalculating from the actual measured 28 days compressive strength to 28 days compressive strength of a <u>water-stored 150 mm cube</u> is given below.

Calculation of Compressive Strength

Within the Nordic countries, the 28 days compressive strength of concrete are measured on different types of specimens and after different types of curing. Comparison of strength levels is therefore not straightforward.

For this reason some calculation rules are set up.

Table 1 from /1/ gives the relationship between compressive strengths measured on different types of concrete specimens, stored in 20 °C water until testing.

Specimen	Dimensio	ons, mm	Relative strength	Factor	
opecimen	Diameter	Height	Keiduve Suengui		
Cylinders	50	100	1,09	0,92	
	75	150	1,06	0,94	
	100	200	1,03	0,97	
	150	300	1,00	1,00	
	200	400	0,97	1,03	
	300	600	0,91	1,10	
Cubes		100	1,25	0,80	
		150	1,20	0,83	
		200	1,14	0,88	
		300	1,08	0,93	

Table 1:Relation between compressive strengths measured on cylinders and cubes.Reference is the 150/300 mm cylinder. The accuracy of the figures is $\pm 5 - 10\%$.From /1/

The compressive strengths are normally measured according to the following:

- > DK: 150/300 mm cylinders, wet stored $[f_{cyl150}]$. However, for practical reasons, 100/200 mm cylinders, wet stored $[f_{cyl100}]$ are often used.
- > FI: 150 mm cubes, wet stored $[f_{cube150}]$
- > IS: 100/200 mm cylinders, wet stored. $[f_{cyl100}]$
- NO: 100 mm cubes, wet stored $[f_{cube100}]$
- SE: 150 mm cubes, dry stored [f_{drycube150}]

In /2/ the Nordic Concrete Committee presented the following relationships between 28 days compressive strength measured on the individual types of specimens and on 150/300 mm wet stored cylinders $[f_{cyl150}]$

$$\begin{split} f_{cyl150} &= 0.54* {f_{cube150}}^{1.12} \\ f_{cyl150} &= {f_{cube100}} - 5 \quad \text{for } f_{cube100} > 20 \text{ MPa} \end{split}$$

$$f_{cy1150} = 0.75*f_{cube100} \text{ for } f_{cube100} < 20 \text{ MPa}$$

$$f_{cy1150} = 0.63*f_{drycube150}^{1.04}$$

Thus recalculation from the direct measured 28 days compressive strength values to the 28 days compressive strength of water-cured 150 mm cubes are expressed as:

DK: f_{cube150} = 1.20*0.97* f_{cy1100} = 1.16* f_{cy1100}
DK: f_{cube150} = 1.20*f_{cy1150}
FI: f_{cube150} = direct measured
IS: f_{cube150} = 1.20*0.97* f_{cy1100} = 1.16* f_{cy1100}
NO: f_{cube150} = 1.20*(f_{cube100} - 5)
SE: f_{cube150} = 1.20*0.63* f_{drycube150}^{1.04} = 0.76* f_{drycube150}^{1.04}

References:

1	Beton-Bogen. Aalborg Portland/CtO. 2 nd edition 1985 (In Danish)
2	Nordisk Betong 1 – 1981 (In Swedish)

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Cement	EN 197-1 Cement notation	Clinke	er content (%)	Other main constituents
Portland cement	CEM I		95 – 100	-
Portland-slag cement	CEM II/A-S		80 - 94	6 - 20
	CEM II/B-S		65 - 79	21 - 35
Portland-silica fume cement	CEM II/A-D		90 - 94	6 - 10
	CEM II/A-P		80 - 94	6 - 20
Portland-pozzolana cement	CEM II/B-P		65 - 79	21 - 35
	CEM II/A-Q		80 - 94	6 - 20
	CEM II/B-Q		65 - 79	21 - 35
	CEM II/A-V		80 - 94	6 – 20
Portland-fly ash cement	CEM II/B-V		65 – 79	21 - 35
	CEM II/A-W		80 - 94	6 – 20
	CEM II/B-W		65 – 79	21 - 35
Portland-burnt shale cement	CEM II/A-T		80 - 94	6 – 20
	CEM II/B-T		65 – 79	21 - 35
	CEM II/A-L		80 - 94	6 – 20
Portland-limestone cement	CEM II/A-LL		80 - 94	6 - 20
	CEM II/B-L		65 – 79	21 - 35
	CEM II/B-LL		65 – 79	21 - 35
Portland-composite cement	CEM II/A-M		80 - 94	6 - 20
	CEM II/B-M		65 - 79	21 - 35
	CEM III/A		34 - 64	36 - 65
Blastfurnace cement	CEM III/B		20 - 34	66 - 80
	CEM III/C		5 - 19	81 - 95
Pozzolanic cement	CEM IV/A		65 - 89	11 – 35
	CEM IV/B		45 - 64	36 - 55
Composite cement	CEM V/A		40 - 64	36 - 60
	CEM V/B		20 - 39	61 - 80
S Blastfurnace slag		v	Siliceous fly ash	(e.g. pfa)
D Silica fume		W	Calcareous pfa (e.g. high-lime pfa)
P Natural pozzzolana		Т	Burnt shale	
Q Natural calcined poz	zolana	L	Limestone, Carbo	on content < 2%
		LL	Limestone, Carbo	on content $< 5\%$

 Table 2: Cement types according to EN 197-1

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Equivalent durability concept



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ABSTRACT

The equivalent durability concept is a tool for providing a concrete that has an equivalent durability to one with a long established record of adequate performance in the local environment. Performance-related test methods are used for the comparison and uncertainty of measurement is taken into account. Concrete produced under the equivalent durability procedure will not be subject to maximum w/c ratio or minimum cement content requirements and therefore there is the potential to produce more sustainable concrete. The aim is to introduce this concept into the next revision of the European concrete standard, but it is recognised that this is an ambitious target.

Key words: Durability, carbonation, chlorides, freeze-thaw, performance-related test methods, standards

1. INTRODUCTION

The general approach to the specification of durability has been unchanged for over a century. The universal parameter has been a maximum w/c ratio to which is added a minimum cement content, limits on constituents and in some cases a compressive strength class or minimum air content. The equivalent durability concept is based on the assumption that any combination of materials that satisfies the current local durability provisions give an acceptable local performance. Given the range of materials that are used to make concrete it is not surprising that traditional specifications do not lead to a consistent performance. This is illustrated in Figure 1 for carbonation resistance.

Figure 1 comprises a range of cement and aggregate types that are in common use in the UK. With most of the sets of materials, a sub-set was produced without the use of an admixture, a sub-set using a water reducing admixture and a sub-set using a superplasticizer to permit the study of the effect of w/c ratio and cement content as independent variables. If the assumption that all these concretes give an adequate performance is correct, this indicates that some of these concretes are significantly over-safe, e.g. unnecessarily low water/cement ratios. The simplicity of the current approach has to be set against the cost of being over-safe and the impact of this over-design on the environment. A modest improvement in the sustainability of the most over-



safe concrete will be significant given the fact that concrete is the most widely used construction material.

Figure 1 — Example of the range of carbonation resistances achieved with concrete conforming to a maximum w/c ratio of 0.55, minimum cement content of 300 kg/m³ and compressive strength of at least 40 MPa [1]

The link between data from laboratory tests and the performance occurring in a concrete element that has a significant risk of, for example, carbonation-induced corrosion is complex, as it comprises a mixture of concrete, workmanship and exposure influences. However, workmanship and exposure influences are not critical to the equivalent durability concept, as the starting point is a reference concrete with a known track record of good performance. The assumption is that workmanship and exposure influences will be similar for the traditional (reference) concrete and the concrete designed using the equivalent durability concept.

The equivalent durability concept is a scheme for establishing conformity to EN 206-1 of concrete compositions that deviate from compositional requirements in the place of use. This concept only applies to concrete compositions that comprise constituents (natural, manufactured or recycled) covered by European technical specifications referred to in EN 206-1 or national provisions valid in the place of use.

2. **DEFINITIONS**

2.1 Candidate concrete

concrete comprising a closely defined set of constituent materials under investigation to determine the mix proportions that are likely to provide a service life equal to or greater than a reference concrete for the selected exposure class

2.2 Equivalent durability

process by which a candidate concrete is shown to have a similar or better durability to a reference concrete in a selected exposure class. The process includes testing both concretes by the same durability test linked to a particular exposure class

2.3 Reference concrete

fully prescribed concrete, including all its constituents, conforming to the provisions valid in the place of use that has a long and successful track record of use in the selected exposure class

3. OUTLINE OF THE CONCEPT

The basic premise is that if a concrete has a similar performance in a durability-related assessment procedure to one with a known history of satisfactory use, it should perform equally well in the same environment. The assessment procedure comprises initial durability testing of the reference concrete and the candidate concrete and an adjustment of the numbers to take account of uncertainty of measurement.

At the national level, for each relevant exposure class or combined classes, a reference concrete is selected that is known to perform well in the local environment. Using test methods and assessment procedure defined in the 2010 revision of EN 206-1, the producer, as an alternative to supplying a concrete conforming to the local limiting value criteria, may develop and supply a concrete that has a demonstrably equivalent durability. For such equivalent concretes, there will be no limiting values of composition for durability, as this would prevent the sustainability benefits being achieved and remove any incentive a producer may have to go this route. Requirements for structural strength and consistence will be as specified, i.e. unchanged.

It will be strongly recommended that equivalence durability is established by a body that is independent of the producer and initial testing, including sampling, is undertaken by an independent laboratory accredited for the tests. Routine production of concretes supplied on this basis should be assessed and surveyed by an approved inspection body and then certified by an approved certification body. Both the specifier and producer benefit from these recommendations for independent third party certification. They give the specifier confidence that the concrete will truly give an equivalent durability and they protect the producer as it is the third party that has determined what gives an equivalent durability. It is not permitted to make this strong recommendation a normative requirement in a voluntary European standard such as EN 206-1. It could, however, become a requirement for concrete products covered by CE-marking should the Standing Committee for Construction so decide.

In principle the concept is applicable to any exposure class except X0, but in practice it is limited to exposure classes where there are agreed test methods. There are no agreed test methods for comparing resistance to aggressive chemicals and consequently in the 2010 version of EN 206, the equivalent durability concept will be limited to the XC, XD, XF and XS exposure classes. The survey of national provisions used with EN 206-1 [2] showed that in some CEN members exposure class XC1 was identified where others have used X0. If limiting values have been specified for reinforced concrete on the basis of engineering judgement and not real durability concerns, it would be inappropriate to apply the equivalent durability concept to this exposure class.

In recognition of the fact that not all CEN members have the same level of development in their concrete producing industries, the equivalent durability concept will only be available at the national level if and when the national standards body defines a set of reference concretes. This approach means that CEN members with a sophisticated producing industry and established third party certification can apply the concept immediately the revised EN 206 is published and it allows other CEN members to delay or not introduce the concept.

4. **PROPOSED TEST METHODS**

4.1 Carbonation-induced corrosion (XC exposure classes)

Equivalent durability in the XC exposure classes will be based on a carbonation test that is in the process of being standardized in the EN 12390 series as prTS 12390-AC. It is presently at the working draft stage. This test uses an enhanced level of carbon dioxide ($4 \ \% \pm 0.5 \ \%$) at a relative humidity of 55 $\% \pm 5 \ \%$. The relative humidity of this test is lower than the TS 12390-10 test and reflects the need in a shorter test to open relatively rapidly the pore structure to the diffusion of carbon dioxide. As with the TS 12390-10 test, once the concrete has adjusted to the relative humidity in the chamber, hydration effectively stops. To try and minimise differences due to differences in the rate of strength gain of different concretes, after 28 days of water curing the specimens are exposed to laboratory air for 14 days prior to placing in the test chamber. Such a test cannot reflect the effects of long term strength gain and so the results of this test will be calibrated against the outside protected version of the TS 12390-10 test. This test will be published as a Technical Specification (TS) as the reproducibility of the test is unknown. A precision test is being organised and when the results of this test are known, this Technical Specification will be upgraded to full European standard.

From the date of casting specimens, the test takes 16 weeks to complete. This is not ideal but the Task Group standardizing this test has technical concerns about using more accelerated tests, e.g. drying a specimen and then exposing it to 100 % carbon dioxide.

This accelerated test is the reference method for comparing equivalent carbonation resistance. The numerical value of the carbonation depth obtained from this test cannot be compared directly with the minimum cover to reinforcement.

The true depth of carbonation is measured using thermogravimetric analysis, but such equipment is not widely available and in practice, in TS 12390-10 and in the accelerated test the depth of carbonation is taken as the depth of phenolphthalein neutralization. The true depth of carbonation is usually a few millimetres greater than that recorded using phenolphthalein, but provided the

carbonation depth is always measured in the same way this is not significant with respect to the equivalent durability concept.

4.2 Chloride-induced corrosion (XD and XS exposure classes)

Equivalent durability in the XD and XS exposure classes will be based on a chloride diffusion test or a rapid chloride migration test. Both tests will also be standardized under the EN 12390 series. The diffusion test (prTS 12390-CD) is at the formal vote stage as a Technical Specification, but work on drafting the rapid migration test is unlikely to start before the end of 2008. While concrete exposed to the XD and XS exposures may also be subject to the XC exposure, the requirements to resist the XD or XS exposures are more severe and so additional testing to show adequate carbonation resistance is not being proposed. Seawater is slightly aggressive to concrete and how this aspect of performance is to be addressed has still to be agreed.

With the chloride diffusion test, a specimen, either a cylinder or cube, is cast and cured in accordance with EN 12390-2, with a minimum curing period of not less than 28 days. The specimen is divided into two sub-specimens, a 'profile specimen' that is used to determine the chloride profile after exposure to unidirectional chloride ingress, and an initial chloride sub-specimen that is used to determine the initial chloride level, C_i . This initial figure is taken as the chloride level of the cast concrete.

The profile specimen is vacuum saturated with distilled or demineralised water, coated on all sides but one and then the uncoated face is exposed to a chloride exposure solution. The exposure is achieved by complete immersion, ponding the uncoated face or inverting the specimen and having the uncoated face immersed in the chloride exposure solution. The reference solution is a 3 % by mass sodium chloride (NaCl) solution, for a period of 90 days (other concentrations or solutions e.g. artificial seawater, are permitted as are different exposure periods). The procedure also permits the use of large fully immersed specimens.

After 90 days of exposure, at least 8 parallel layers of the chloride exposed surface are ground off the profile specimen. The acid-soluble chloride content of each layer and the average depth of the layer from the surface of the concrete exposed to the chloride solution are determined. The initial chloride content is determined by grinding a sample from the other sub-specimen and the acid-soluble chloride content determined.

By non-linear regression analysis by least squares curve fitting, the surface chloride content (C_s) and the non-steady state chloride diffusion coefficient (D_{nss}) are determined.

Because of the high coefficient of variation, ~ 15 % for D_{nss} for the test, it is required to test at least two specimens and report the results separately.

By keeping additional specimens exposed to the exposure solution, it is possible to test these at a later age and use the two determinations of D_{nss} to determine the ageing factor.

At the time of drafting this paper no proposal for the rapid migration test has been submitted, but the test will be based on one of the existing tests.

CEN/TC104/SC1/TG17 is debating two approaches. In the first approach, the chloride diffusion test is used to compare the concretes. The comparison will include a factor to cover uncertainty of measurement and ageing effects. As the numerical values of the ageing effect are likely to be controversial, a simpler alternative approach is being considered. In this case a rapid migration test is undertaken at three months and the comparison will only take account of uncertainty of measurement. After three months the non-steady-state chloride migration, as measured by the rapid migration test, changes little with time and so ageing effects can be ignored. Because a charge is applied to the concrete, the binding capacity is different to that achieved in the chloride diffusion test.. The relative simplicity of the test, the speed of the test and relatively low cost are factors that favour this approach. However the most important consideration is whether the candidate concrete will in the service-condition give an equal or better performance than that given by the reference concrete.

4.3 Freeze-thaw resistance

In some CEN Member Countries, a direct performance requirement is specified for the XF exposure classes or permitted as an alternative to satisfying limiting values. In such CEN Member Countries there is no need for an equivalent durability concept for the XF exposure classes. However, the TS 12390-9: *Testing hardened concrete — Part 9: Freeze-thaw resistance — Scaling* tests are very severe and in some CEN Member States the application of these tests will fail concretes that have a long history of satisfactory use. CEN/TC51(CEN/TC 104)/JWG12: *Performance-related test methods* has been asked to prepare a less severe test that is suitable for relative testing. If this requires a small modification of TS 12390-9, e.g. fewer cycles, then the next revision of European concrete standard can cover the XF exposure classes. If a new test procedure has to be developed, this is unlikely to be completed in time.

5. **REFERENCE CONCRETES**

5.1 General

A CEN Member Country is free to specify different reference concretes for each of the XC exposure classes and each of the XD and XS exposure classes. The choices of reference concretes are decisions for the national standards bodies and they should be based on concretes that have been proven to work well in the local environment.

One of the first decisions that the national standards body has to take is whether they are going to simply define the reference concretes, or define the reference concretes then undertake a testing programme and specify the performance for the reference concrete directly. If testing is undertaken, sufficient testing is needed to ensure that the specified performance is the true mean value of the reference concrete. Such an approach takes out one of the sources of uncertainty, namely the true value of the reference concrete. However if there are differences between or within laboratories, specifying and testing the reference concrete each time will minimise the effects of any bias. A possible compromise would be to specify performance directly and require laboratories undertaking this work to participate in two yearly proficiency exercises.

The reference concretes should meet all the requirements of the local provisions. In addition for each reference concrete the type of cement/addition, the cement strength class, the aggregates types and admixture type have to be specified. It is likely that there will be a recommendation to convert all these requirements for the reference concretes into fully prescribed concrete

specifications. A consistence should also be specified together with a modest tolerance on this value, e.g. $120 \text{mm} \pm 20 \text{mm}$.

As shown in Figure 2, the local specification is likely to lead to a range of different performances, all being assumed to be adequate. The criteria for accepting a candidate concrete will take into account the uncertainty of measurement and depending on the approach adopted, may take into account ageing effects.

In the situation where the ageing effects of the reference concrete and the candidate concrete are the same, if the reference concrete is selected from the best performing set of constituents, the requirements for the candidate concrete will be higher than anything currently required. In this situation no producer will use this concept as the concrete is likely to be more expensive than one supplied using the traditional limiting value approach. If the reference concrete is taken from the middle of the range, when the margin is applied, the candidate concrete is likely to be close to the top of the range and there will be no commercial and little environmental point in using the concept. Consequently the reference concrete should be selected such that it, in effect, places the candidate concrete in the mid-range of performance. The net effect of this proposal is that the requirements for the most over-safe concretes will be relaxed while those in the mid and lower end of the acceptable performance range will be unchanged.

The equivalent durability concept is based on the assumption that the local provisions are adequate even when using the set of conforming constituent materials that perform least well. If the recommendation above is followed, this will mean there will be about half of the concretes currently in use that will not meet the equivalent durability requirements. This must not be taken as an indication that these concretes are inadequate for the local environment, as experience has shown them to be adequate. It simply reflects the fact that the equivalent durability concept is being introduced in a very safe and conservative manner.



Figure 2 — Example of the range of carbonation resistances achieved with concrete conforming to a maximum w/c ratio, minimum cement content and compressive strength class specification, data from [1]

5.2 W/C ratio

It is likely to be recommended that the w/c ratio of the reference concrete is 0.02 below the locally applied maximum w/c ratio for the exposure class under consideration. For example, where a maximum w/c ratio of 0.55 is specified in the local provisions, the w/c ratio of the reference concrete should be 0.53 except where requirements for compressive strength or minimum cement content controls the mix design. By applying such a requirement, the control of production may be that normally applied and there is no need for an additional margin to cover batch to batch variability, see 6.2.

5.3 Minimum cement content

The prescribed reference concrete should have at least the minimum cement content required by the local provisions. In many cases the prescribed cement content will be higher than the minimum value as more cement will be needed to satisfy the maximum w/c ratio requirement and consistence. In addition the concrete should contain enough fine material (it does not have to be all cement) to give a concrete with a closed structure.

5.4 Cement type and strength class

It is essential to specify the cement type and its cement strength class. If an addition is to be included, the type and quantity should also be specified.

5.5 Aggregate type

Aggregate type and grading has a significant impact on performance. With porous aggregates, carbon dioxide and chloride ions can diffuse through the aggregate particles. Consequently it is essential to closely define the aggregates used in the reference concretes. Data [3, 4] showing this influence are given in Table 1.

Cement Type ²	Coarse aggregate type	Granite	Carboniferous limestone	Natural gravel	Jurassic Oolitic limestone	Dolomitic magnesium limestone Sintered pfa lightweight
Ι		12.5	13.5	15.5	17.5	17.5 27
II/A-L		15.5				
II/B-L		24				
II/A-Q (metakaolin)		17				
II/B-Q (shale ash)		23				
II/A-D		15				
II/B-P		25				
II/B-V (35 % pfa)		26				
III/A (50% ggbs)		16		23		
V/A (29% ggbs, 21%	6 pfa)	22				
	• .1	200 (2401 /	5		

Table 1 — 20 weeks accelerated carbonation data for a w/c ratio of 0.55^{1}

¹ Cement contents were in the range 300 to 340 kg/m^3 .

 2 No data were available for cements with more than 50 % of a second main constituent.

5.6 Admixtures

The majority of concrete produced in Europe contains at least one admixture. To reflect practice, it is suggested that the specification for the reference concrete includes an admixture. For the XC exposure classes a water reducing admixture would be appropriate and for the XD and XS exposure classes a superplasticizing admixture is appropriate. Where the local provisions require air entrainment for freeze-thaw resistance (XF exposure classes), the specification of the reference concrete needs to include an air-entraining admixture. Where none is required, a water reducing admixture is appropriate.

Where the reference concrete contains an admixture, the target slump should be specified together with a modest tolerance, say $120mm \pm 20mm$. Providing an indicative free water content may also be helpful to the laboratory producing the specimens.

6. **PRODUCTION CONTROL**

6.1 Initial testing

As it is unlikely that a candidate concrete can be selected that gives exactly equivalent durability, it is recommended that at least three mixes be cast and tested. One of these three mixes will be a mix that is expected to give the equivalent durability, one mix that is designed to give a better performance and the other mix that is designed to give a lower performance. The results from the three mixes are used to interpolate the mix proportions that give the equivalent durability. Where the requirement from the reference concrete is close to the limit of the performance of the candidate concrete, more than three mixes may be required as there could be a turning point in performance, i.e. no improvement in performance with reducing w/c.

Other concrete mixes made with this set of materials but with a lower w/c ratio should be regarded as achieving the required performance. Other concrete mixes made with this set of materials where the w/c ratio is higher than that of the equivalent durability mix or made with a low fines content should not be regarded as giving equivalent durability unless proven by testing. Chloride and carbon resistance depends upon there being enough fine material to give a closed structure. Research on the role of minimum cement content in providing concrete durability [4] showed that when a superplasticizing admixture was used to minimise the cement content, the mixes with low fines content tended to give a reduction in chloride/carbonation resistance. It was postulated that this was due to the lack of a closed structure. The water penetration test (EN 12390-8) may be a means of showing when a concrete has a closed structure.

For convenience it has been proposed that in EN 206:2010 the uncertainty of measurement and, if applied, the ageing effect are combined into a single factor. As the ageing factor is largely dependent upon the cement type (or cement and addition type), the factor given in EN 206:2010 may be based on the cement type used for the reference concrete and the cement type used for the candidate concrete. Exactly how this could be done is the subject of current debate within the CEN Task Group.

The literature indicates that the ageing factor is not a constant for a given cement type, but a range of values. However, the same criticism is true for most engineering properties of concrete, e.g. elastic modulus, creep and drying shrinkage. We have dealt with variability in performance

before and in principle there is no reason why a robust, but still economic, solution cannot be found for dealing with ageing effects.

The candidate concrete has to have a measured value of performance not greater than a factor given in EN 206:2010 times the performance of the reference concrete for it to be shown to have equivalent durability. It should be noted that with the carbonation test, chloride diffusion test and the freeze-thaw test, the lower the measured value the better is the performance. The permission to permit the factors given in EN 206 to be over-ridden by a nationally determined factor is being kept under review. If necessary such a permission will be included in EN 206, but the ideal solution would be to have the same values throughout Europe.

A future development may be to devise an experimental way of justifying a different ageing factor, but such a system is unlikely to be ready in time for the 2010 revision of EN 206. However, the uncertainty of measurement has also to be taken into account and this will be set by CEN and there will be no national deviations, as the values will be based on the test precision.

6.2 Routine production control

Before describing what is being proposed for the equivalent durability concept, a brief description of the current system for limiting values is given. Limiting values have been selected by the national standards body based on previous experience and perhaps additional test data for new cements. The (potentially significant) impact of aggregate type is rarely taken into account. The only requirements for production control are the use of the permitted constituent materials, checking the concrete does not exceed the maximum w/c ratio and checking that the concrete does not go below the specified minimum cement content. Put simply, once the performance of a concrete has been established in terms of its composition, the only control is on composition, not on performance. This system is widely accepted as being adequate.

The test methods needed to show equivalent durability are not suitable for the routine control of production and consequently the control of production should be the same as the existing system with the exception that in this case the concrete has been demonstrated by initial testing to give an equivalent performance.

The recommendation to national standards bodies is for the reference concrete to have a w/c ratio of 0.02 lower than the recommended maximum w/c ratio for the exposure class. According to EN 206-1: 2000, most batches are required to achieve the maximum w/c ratio and no batch may have a w/c ratio greater than the specified maximum w/c ratio plus 0.02. Provided a batch does not have a w/c ratio greater than the specified maximum w/c ratio plus 0.02, it is regarded as being of acceptable quality. The equivalent durability concept uses the same criteria. With most modern concrete plants there is an autographic recording system and conformity to the target w/c ratio and cement content can be done automatically. Is more than this needed?

In the view of the author, further control is needed. This is because the constituent materials do change over time and this may effect a change in the durability of the concrete. Such changes in constituents are usually reflected as a change of compressive strength. From our knowledge of concrete, it is safe to assume that a change that has a negative impact on average strength will also have a negative impact on durability and vice versa.

The variability in compressive strength is due to a combination of factors including changes in constituents, batching variations, plant factors, sampling and testing variations. By comparing changes in average strength, a number of these variables should either equal out or show a systematic change, e.g. the strength is less than the target strength because of an error in the weigh scales. A change of average strength is usually a reflection of a real change in one or more of the constituent materials or a change in the performance of the plant, e.g. the weigh scales go out of calibration. An important exception to this generalisation about the relationship between compressive strength and durability is where the reduction in concrete strength is due to a reduction in cement strength. A change in cement strength may or may not have an impact on durability, but the 'safe' solution is to assume that it has a detrimental effect.

For a given set of materials, durability is a function of w/c ratio, but routine control of performance and production of concrete is based on compressive strength. It is therefore necessary to relate this limit on acceptable variation in w/c ratio to compressive strength. In the normal range of concrete strength, a step of 0.01 in w/c ratio is approximately equal to 1.0 N/mm² in cube strength or 0.8 N/mm² in cylinder strength.

Controlling the target compressive strength is a universally applied and familiar procedure used in the production of concrete. In practice the producer batches the same mix proportions in the expectation of achieving the target strength. The testing of production concrete will produce a scatter of results around this target value, but there are established statistically-based systems for determining if the target strength is being achieved. A reasonable system is capable of determining when the actual strength is 0.5 standard deviations (0.5σ) below the target strength. Assuming the standard deviation is 4 N/mm² (cube), this equates to a decline in cube strength of 2 N/mm², or, if nothing has changed other than the w/c ratio, a change in w/c ratio of 0.02.

Therefore when using this concept, EN 206:2010 should include a requirement in the factory production control system that requires action to be taken when the actual mean strength is consistently 0.5σ below the target strength. During the period before a strength reduction is confirmed, there may be individual batches where the w/c ratio is less than the maximum w/c ratio, but because a 0.04 margin between the target value and that permitted for an individual batch (maximum w/c ratio plus 0.02) has been included in the reference concrete and consequently reflected in the quality of the candidate concrete, these production batches will still have an acceptable performance. Any batch with a w/c ratio more than 0.04 above the target value is declared as non-conforming. Therefore the proposed control of production requires nothing less or nothing more than the present system of control.

There is a view that a periodic check on performance would help establish confidence in the system and so there may be a requirement for a sample to be taken from the production concrete at intervals not exceeding two years and tested to confirm the performance of the production concrete.

7. CONFORMITY

As with the traditional durability requirements, conformity will be based on batching the correct mix proportions. Identity testing using the reference test methods is not appropriate because the tests would take too long to complete and with the way the system has been set up, i.e. the candidate concrete falling in the middle of the currently accepted performance range, it would

require a gross error to put the structure at risk and gross errors will be detected by the control on batching.

8. **DISCUSSION**

The equivalent durability concept is a modest first step in moving the concrete sector into specifying durability by performance. It will allow the sector to gain confidence in the specification of durability by performance and the ability of producers to meet the necessary performance. However, even to achieve this modest step requires considerable effort from a very conservative industry. One major problem is the 'fear factor' and this could result in worse case assumptions throughout the process. When they are put together the resulting concrete has a quality well above anything currently accepted and consequently the concept will not be used in practice. The procedures outlined above try to reach a balance between being conservative, robust in the world in which we operate and offering enough incentives to make it worthwhile to a producer.

However, because of the way the system is being constructed, there will be many concretes that meet current specifications, but fail to meet the performance of the reference concrete. This must not be taken as an indication that these concretes are not suitable (experience shows that they are suitable); it is only a reflection of the conservative way in which the equivalent durability concept is being introduced.

This process of evolution is being helped by the slow realisation that we need to make our product, concrete, more sustainable. The equivalent durability concept is a tool that can be used to produce concrete with the required durability but in a more sustainable way.

The drafters of the equivalent durability concept are not claiming that it will provide the information needed for service-life design. This is for the future. However, it should be noted that the procedure by which performance is established and controlled for the equivalent durability concept would be equally applicable to a direct specification of performance obtained from service-life design. The view of the author is that we should prove that we can 'walk with confidence before trying to run'.

9. CONCLUSIONS

1. CEN/TC104/SC1 is developing an equivalent durability concept with the objective of introducing this concept into the next revision of the European concrete standard.

2. The equivalent durability concept will work alongside the normal limiting value approach to the specification of concrete, which will in the foreseeable future remain the main method for satisfying durability requirements.

3. New performance-based test methods are being standardized to support the equivalent durability concept.

4. The equivalent durability concept may be used as a tool to produce more sustainable concretes.

5. The equivalent durability concept should be seen as a step in moving towards performancebased specification for durability, not the end point.

10. **REFERENCES**

10.1 Standards

EN 201-1:2000	Concrete — Part 1: Specification, performance, production and conformity
EN 206-1	Concrete — Part1: Specification, performance, production and conformity
EN 12390-2	Testing hardened concrete — Part 2: Making and curing specimens for strength tests
EN 12390-8	Testing hardened concrete — Part 8: Depth of penetration of water under pressure
TS 12390-9	Testing hardened concrete — Part 9: Freeze-thaw resistance - Scaling
TS 12390-10	Testing hardened concrete — Part 10: Determination of the relative carbonation resistance of concrete
prTS 12390-CD	Testing hardened concrete — Part CD: Determination of the chloride resistance of concrete, unidirectional diffusion
prTS 12390- AC	Testing hardened concrete — Part AC: Determination of the carbonation resistance of concrete – Accelerated carbonation method

10.2 Other references

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Use of Additions - Progress of CEN/TC104/SC1 Task Group 5



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ABSTRACT

For the next revision of EN 206-1, TG5 has accepted in 2007 the tasks to formulate proposals for modifications of clause 5.2.5 "Use of additions". The clause will give specific rules for fly ash, silica fume and ground granulated blastfurnace slag. For establishing suitability the equivalent performance of combinations concept will be introduced. The paper will give the present state-of-the-art. Many topics however will need further discussion.

Key words: additions, suitability, k-value, equivalent concrete performance concept, equivalent performance of combinations concept.

1. INTRODUCTION

CEN/TC 104/SC1 has reactivated TG 5 "Use of Additions" to reconsider the k-value-concept of EN 206-1. The aim of the TG is to produce a discussion document to provide a proposal on framework to deal with the k-value concept considering:

- variable k-values
- minimum cement content when additions are taken into account
- provision of an example in an informative annex
- use of the k-value concept with other types of cement
- equivalent performance of combinations concept.

The TG 5 members list is given in appendix A.

In this paper the present state-of-the-art is given. Many topics however will need further discussion.

2. SURVEY OF NATIONAL PROVISIONS

The starting point for TG5 were the results of the work of an ad-hoc CEN TC104/SC1 working group chaired by T. Harrison. This ad-hoc working group has prepared a comprehensive overview of the use of national requirements in conjunction with EN 206-1 [1].

With respect to the use of additions the inventory shows that the way in which these additions are used in Europe varies significantly. The findings are listed below.

- *k*-value concept in EN 206-1 is used with permitting
 - the option of varying the *k*-values for w/c ratio
 - rules with respect to minimum cement content
 - use other concepts.
 - only agreement for *k*-values for CEM I cements
- A majority of CEN Member Countries apply the rules of EN 206-1 (*k*-value, minimum cement content) for CEM I cement with fly ash or silica fume.
- For fly ash the *k*-values in combination with CEM I vary between 0,2 and 0,6.
- For silica fume CEN Member Countries apply a *k*-value of 2,0
- The k-value concept for fly ash is generally applied to cement types other than CEM I using *k*-values in the range of 0,2 and 0,5.
- Six CEN Member Countries use the *k*-value concept with silica fume and cement types other than CEM I. The application of the concept is, in the majority of the cases, restricted to CEM II (both A and B) cements.
- Six CEN Member Countries apply the *k*-value concept for ground granulated blastfurnace
- slag (ggbs). The *k*-values vary between 0,6 and 1,0.
- Generally no other concepts for the use of additions are applied in the CEN Member Countries.
- UK, Portugal and Ireland have a system for combinations of CEM I or CEM II
- with either ggbs, fly ash or limestone that uses early and 28 day strength requirements.
- Finland applies durability design rules for freeze-thaw resistance and carbonation.
- EN 206-1 does not define precisely how equivalent performance should be determined,
- although general guidance is provided in EN 206-1, annex E.
- Concepts are under preparation in a few CEN Member Countries
- Afterwards added: In the Netherlands the equivalent concrete performance concept is used for mixtures with specific cements and fly ashes.

3. APPROACH IN EN 206-1: 2000

In the present EN 206-1 the use of additions is described in clause 5.2.5.

Type II additions may be taken into account in the concrete composition with respect to the cement content and the water/cement ratio if the suitability is established.

The suitability of the k-value concept is established for fly ash and silica fume (see 5.2.5.2).

If other concepts e.g. the equivalent concrete performance concept (see 5.2.5.3), modifications of the rules of the k-value concept, higher k-values as defined in 5.2.5.2.2 and 5.2.5.2.3, other additions (including type I) or combinations of additions are to be used, their suitability shall be established.

NOTE: The establishment of the suitability may result from either:

- a European Technical Approval which refers specifically to the use of the addition in concrete conforming to EN 206-1;
- a relevant national standard or provision valid in the place of use of the concrete which refers specifically to the use of the addition in concrete conforming to EN 206-1.

4. K-VALUE CONCEPT

General

The principle of k-value calculation is explained in figure 1 [2].

In EN 206-1 is stated that the k-value concept permits type II additions to be taken into account:

- by replacing the term "water/cement ratio" (defined in 3.1.31) with "water/(cement + k * addition) ratio";
- in the minimum cement content requirement (see 5.3.2).

The actual value of k depends on the specific addition.



Figure 1 – Principle of k-value calculation [2].

k-value concept for fly ash conforming to EN 450

The maximum amount of fly ash to be taken into account for the k-value concept shall meet the requirement:

fly ash/cement \leq 0,33 by mass.

The following k-values are permitted for concrete containing cement type CEM I conforming to prEN 197-1:

CEM I 32,5	k = 0,2
CEM I 42,5 and higher	k = 0,4

k-value concept for silica fume conforming to EN 13263

The maximum amount of silica fume to be taken into account for the water/cement ratio and the cement content shall meet the requirement:

silica fume/cement $\leq 0,11$ by mass.

The following k-values are permitted to be applied for concrete containing cement type CEM I conforming to prEN 197-1:

Prescriptive k-value approach

The k-value approach is a prescriptive approach. It can be considered as a safe, lower level approach that can be used, without testing, for a variety of EN 450 fly ashes or EN 13263 silica fumes in combination with the spectrum of EN 197-1 CEM I cements.

Results presented in TG5 by R. Härdtl show that in the case of the use of fly ash the agreed k-values are clearly on the safe side [2] (see figure 2).



Figure 2 - Determination of k-values for fly ash [2].

5. EQUIVALENT CONCRETE PERFORMANCE CONCEPT

The equivalent performance in concrete permits amendments to the requirements in this standard for minimum cement content and maximum water/cement ratio when a combination of one or more specific additions and one or more specific cements are used, for which the manufacturing source and characteristics of each are clearly defined and documented.

In the Netherlands the equivalent concrete performance concept for combinations of fly ash and CEM I cement or CEM I + CEM III cement combinations is used for about 15 years now. Initially for fly ash cement combinations the procedures and requirements are laid down in the National assessment guideline BRL 1802. Recently the possibility for combinations of ground blast furnace slag, fly ash and cement were introduced with BRL 9340.

At this moment a CUR Recommendation is in development with a general concept for establishing the equivalent concrete performance [3]. The performance of the test-concrete is compared to the performance of a reference concrete which suitability is established:

Depending on the exposure class, equivalent performance of the test-concrete shall be assessed for aspects like carbonation, chloride penetration, frost-thaw resistance, seawater resistance and / or sulphate resistance. The performance of the test-concrete shall be compared with the performance of a reference concrete.

The reference concrete shall meet the requirements of EN 206-1 and the national provisions (NEN 8005) for the appropriate exposure class. This refers to the mix composition and type of cement.

The test-concrete shall contain cements and additions within the limits of EN 197-1. The binder content shall be at least the minimum cement content for the exposure class according to NEN 8005. The ratio "water/(cement+addition)" shall nor be higher than the maximum allowed "water/cement" ratio for the considered exposure class

6. EQUIVALENT PERFORMANCE OF COMBINATIONS CONCEPT

The UK uses an 'equivalent performance of combinations concept' [4]. In this concept:

- an addition added at the concrete mixer may be considered to perform in the same way, as would the same material incorporated into concrete as a constituent of cement.
- this is only permitted when a continuous programme of control testing of the specific addition with the specific cement, is carried out.

The blend of addition and cement is called a 'combination' and BS 8500 (the British Complementary Standard to EN 206-1) contains the following definition:

- Combination: "restricted range of Portland cements and additions which, having been combined in the concrete mixer, count fully towards the cement content and water/cement ratio in concrete"

A procedure for establishing the suitability of combinations is specified in BS 8500-2:2002, Annex A.

Combinations are used in concrete in the same way as cements of the same composition and strength class.

The procedure for establishing the restricted range of combinations that can count fully towards the cement content and water/cement ratio is known as the "Conformity Procedure for Combinations" and only applies for combinations of a CEM I cement of standard strength class 42,5 or greater, conforming to BS EN 197-1 with one of the following additions:

- fly ash conforming to EN 450: 2005 category A or B
- ggbs conforming to BS 6699 or EN 15167-1
- limestone fines conforming to BS 7979

Because silica fume can be used within EN 206-1 with a k-value of at least '1', it was not considered necessary to include it in the UK combinations procedure.

Establishment of suitability is based on strength testing with EN 196-1 mortar prisms, using average monthly samples of the CEM I and the addition. The combination must:

- exceed requirements for early- and 28-day strength by a statistical margin
- not exceed an upper limit on 28-day strength

The strength requirements that have to be met are based on those in EN 197-1.

In its simplest form, the procedure can be carried out for a fixed proportion of addition to cement and determines (pass/fail) whether that specific proportion meets the strength requirements. If it passes, it is a "permitted proportion". In practice, the procedure is normally carried out by the suppliers of additions who use a more complex approach in order to determine the range of "permitted proportions" (rather than determine whether one specific proportion is permitted).

The requirements are described in Annex A and Annex C to BS 8500-2. Annex A sets out the normative requirements. Annex C provides an informative example that is typical of the approach taken by suppliers of additions in order to certify one source of addition used in combination with several sources of CEM I cement. Here, relationships have to be established between the proportion of addition and strength for each of the CEM I cements and re-established every two years. Monthly testing of samples of the addition combined with samples of the CEM1 cements determines limits on the proportions of addition with each specific CEM I cement source to ensure that the conformity criteria for strength are met.

Compared with the k-factor approach, the UK Combinations Conformity Procedure is complex and requires a considerable amount of extra testing. In order to parallel the testing regimes used by the cement industry, samples of CEM and addition have to be taken regularly through the month and testing carried out on the homogenized samples. The UK procedure does however, provide confidence that the behavior of a specific addition with a specific cement, is tightly controlled and as a result consistent concrete can be produced.

A first draft for the corresponding clause in EN 206-1 has been presented in TG5:

The equivalent performance of combinations concept permits combinations of a cement and an addition to count fully towards the cement content and w/c ratio in concrete. The concept is

applicable to a specific source of addition combined with a specific source of CEM I cement conforming to EN 197-1, and determines permitted proportions for the addition relative to the cement.

The concept shall be used only for combinations of a CEM I cement of standard strength class 42,5 or greater conforming to EN 197-1 with one of the following additions:

a) fly ash conforming to EN 450-1:2005, category A or B; b) ggbs conforming to EN 15167-1;

Each month, samples shall be obtained that are representative of each lot of addition and each lot of cement that are to be evaluated for use in combination. Combinations of these addition and cement samples, in appropriate proportions, shall be tested for compressive strength in accordance with the method for testing cement specified in EN 196-1, with all references to "cement" therein, being construed as referring to the "combination" of addition plus the cement.

NOTE: Where third-party certification is required, the certification body might require the proportions selected to be justified.

7. DISCUSSION ITEMS

In TG5 the discussion was focussed on EN 206-1 clause 5.2.5 "Use of additions". The present standard gives possibilities for regulations in national application documents for the use of additions. However, it was felt that standardization on European scale would be helpful.

Ground Granulated Blastfurnace Slag (GGBS)

 Because the European Standard for ground granulated blastfurnace slag (GGBS) has been published (EN 15167) the existing clause 5.2.5. should be extended with this addition.

Details on Equivalent Concrete Performance Concept (ECPC)

- Besides the prescriptive k-value concept also the principles of the Equivalent Concrete Performance Concept (ECPC) should be given. Even if this concept is already available as informative annex E, it should be evaluated, to what extend this concept can be part of the normative text. The concept is well proven in the Netherlands.

Introduction of the Equivalent Performance of Combinations Concept (EPCC)

- Because of the experience with the Equivalent Performance of Combinations Concept (EPCC) in countries like the UK, Ireland and Portugal it should be evaluated to what extend this concept can be part of the normative text.

K-value concept for other types of cement (than CEM I)

- The clause should be formulated in a way that does not limit the types of cements and additions, which National Regulations may allow under the k-value concept. TG5 should draft suitable rules or principles for the use of additions together with CEM II cements (in a similar way as will apply for CEM I cements).

Annex E

Annex E "Guidance on the application of the equivalent performance concept of concrete properties" will probably be modified to "Guidance on the application of the Equivalent Concrete Performance Concept and the Equivalent Performance of Combinations Concept". In a future stage reference can be made to a new Annex "Equivalent Durability Concept " as under preparation by TG17.

8. **RECOMMENDATIONS FOR THE REVISION OF CLAUSE 5.2.5**

By TG5 recommendations to CEN TC 104/SC1 were formulated for the revision of clause 5.2.5 of EN 206-1.

Recommendation 1

The 'k-value Concept' (clause 5.2.5.2) and the 'Equivalent Concrete Performance Concept' (clause 5.2.5.3) should remain in EN 206-1:2010. General aspects of the 'Equivalent Performance of Combinations Concept' should be added.

Recommendation 2

EN 206-1:2010 should include reference to Ground Granulated Blastfurnace Slag to EN15167 according to the concepts referred to in recommendation 1.

Recommendation 3

EN206-1 should describe the concepts for the use of additions together with CEM II cements and TG5 should draft suitable rules or principles.

Recommendation 4

EN 206-1: 2010 should only describe the 'concepts for the use of additions' in a general way. The detailed rules for applying these concepts should be left to national application documents.

Recommendation 5

A detailed description of the existing Concepts should be prepared, to be published as a CEN report.

General remark

It should be possible to make a reference to Annex Equivalent Durability Concept as under preparation by TG17.

These recommendation which have been accepted during the TC104/SC1 meeting in Berlin (June 2008), will be the basis for the continuation of the work of TG5.

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APPENDIX A

Members of CEN TC104/SC1 TG5 "Use of Additions" (September 2008)

Nischer	Austria
Lebon	Belgium
Musikas	Belgium
Bager	Denmark
Reynard	France
Potier	France
Heinz	Germany
Seymour	Ireland
Corazza	Italy
Cornelissen (convenor)	Netherlands
Van den Berg	Netherlands
Langelaan	Netherlands
Goncalves	Portugal
Kutti	Sweden
Ljungkrantz	Sweden
Dietz	Switzerland
Sear	UK
Higgins	UK
Müller	TC51
Feuerborn (secretary)	ECOBA
Assbrock	ERMCO
Haerdtl	Cembureau


Fly ash and Co combustion



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Abstract

Production of fly ash and bottom ash is described together with information on the production and utilization of the products. Further to this the current and future standard for concrete fly ash is described. And finally the visions for the future production are envisaged. Centrally placed in all these issues ECOBA

1. GENERATION AND QUALITY OF ASHES FROM COAL COMBUSTION

In coal-fired electricity generating power plants solid minerals are produced during and after the combustion of finely ground coal in a fully controlled process. The materials under consideration are the ashes i.e. the unburnable mineral matter in the coal (bottom ash, fly ash, boiler slag, Fluid Bed Combustion-ash), and, where abatement equipment is fitted, the desulphurisation products obtained from a chemical reaction between the sulphur dioxide, which is derived from the sulphur in the coal during the combustion process, and a calcium based absorbent, in flue gas desulphurisation installations (Spray Dry Absorption product and Flue Gas Delsupurization gypsum).

Most of the by-products are produced in so called dry bottom furnaces, i.e. a combustion processes with furnace temperatures of 1100- 1400°C. The combustion process of in a dry bottom furnace and the generation of coal combustion products (CCPs) is shown in figure 1. In this report I will only focus on the ash products that can find immediate use for the production of concrete.

A similar process (wet-bottom furnace) is used for production of boiler slag. Within this combustion process the furnace temperature is higher ($1500 - 1700^{\circ}$ C) and the fly ash normally is fed back to the boiler where it melts again and forms boiler slag.

2.1 Bottom ash

Generation

During the combustion of finely ground coal in the boiler (see figure 1), some mineralized, partly melted particles agglomerate within the boiler and become sintered together. Owing to their weight these particles do not pass out of the combustion chamber with the flue gas, but fall to the bottom of the boiler, where they are either removed directly or quenched in a water bath influencing the particle structure. This bottom ash may be processed, if necessary, by dewatering, screening, breaking and/or grading before an interim storage (silo, pit) or loading onto truck, train or barge at the power plant's temporary store and dispatched to its intended use



450 MW_{el} Coal-Fired Power Plant 6000 Hours Full Load

Fig 1 Production of coal combustion products (CCPs) in coal-fired power plants

Samples for quality monitoring are usually taken directly from the loading equipment at the temporary storage facility. The nature and extent of quality monitoring depend on the area of application. Where the bottom ash is used as a lightweight aggregate for mortar and concrete, it typically has to comply with the requirements of European and/or national rules (application standards). In earthworks and civil engineering it often has to satisfy national regulations of the road authorities. In additional, specific requirements may be agreed between the bottom ash producer and the user.

Properties

Bottom ash consists of irregularly shaped particles with a rough surface. The main chemical components are silica, aluminium and iron oxide. The chemical composition of bottom ash is largely comparable to that of fly ash (see 2.2). Due to its porous particle structure, bottom ash combines low weight with good soil mechanics properties; however, its particle size distribution may vary considerably, as it depends on the fineness of the pulverized coal and the combustion conditions.

Typical uses for bottom ash, together with details of the quality requirements it must meet for these uses, include:

• for concrete blocks: EN 13055-1 and national regulations

• in earthworks and road construction: according to national regulations.

In particular, the properties of bottom ash are useful:

- in open placement for the construction of roads and pathways and the creation of industrial and storage areas,

- in landscaping and recultivation measures,
- in the construction of bound and non-bound load-bearing layers and bound base surface layers,
- in road sub bases and
- in the construction of noise barriers.
- as lightweight aggregate for concrete products according to EN 13055-2 including requirements for conformity evaluation.
- as a raw material for cement clinker production: site specific requirements
- as filler for cement: EN 197-1 & EN 450-1

- for brick production: national regulations
- for gardening and landscaping: national regulations

2.2 Fly Ash

Generation.

The pulverized coal is blown with air into the combustion chamber of the power plant boiler. Combustion (oxidation) of the coal components at a temperature of up to 1400°C produces mineralized particles which, after a residence time of up to several seconds, leave the firing chamber with the flue gas (see figure 1).

1. The flue gas containing the fly ash flows through the boiler passes and also, if present, the denitrification unit and economizer, and is then fed to the dust removal unit.

2. In the electrostatic precipitator which usually works on the principle of electrostatic precipitation and comprises a number of stages (cells), the fly ash is separated from the flue gas and removed.

3. Monitoring of fly ash quality, assuming it is intended for high-grade use takes place between the dust removal unit and the interim storage silos. The combustion process is controlled and material sorted depending on the monitoring findings.

4. On the basis of the results, the fly ash is stored in different silos depending on its quality (compliance or non-compliance with standards). From there it is transported to the place of use by road, rail or waterway.

Properties

Fly ash is a fine powder consisting mainly of spherical, glassy particles. A distinction is made between siliceous and calcareous fly ash. The principal components are silica, aluminium and iron compounds, and also — in calcareous fly ash — calcium oxide or calcium compounds. The composition of siliceous fly ashes corresponds to that of naturally occurring pozzolans (volcanic ashes), while calcareous fly ashes also contain hydraulically active mineral phases in addition to pozzolanic components. A special property of siliceous fly ash is its pozzolanic reactivity, i.e. its capacity to react with lime and water at ambient temperature to form strength-giving mineral phases similar to those in Portland cement. In view of its fineness and particle size distribution, and also its pozzolanic reactivity, coal fly ash is mostly used in cement-bound building materials to improve their technical properties and replace cement.

Use and requirements for use

In 2003, about 44 million tonnes of fly ash from lignite and coal combustion were produced in EU 15 (i.e. member states pre 2004). Most of the fly ash from lignite combustion (22.7 million tonnes) is used for reclamation of open cast mines, pits and quarries.

About 21 million tonnes of fly ash was used in the construction industry and in underground mining, e.g. as concrete addition, in road construction and as a raw material for cement clinker production. Fly ash was also utilized in blended cements, in concrete blocks and for infill (that means filling of voids, mine shafts and subsurface mine workings).

Typical uses for fly ash, together with details of the quality requirements it must meet for these uses, include:

• as addition to concrete according to EN 206-1. Fly ash is used as a concrete addition in various proportions depending on the individual mix design, and improves the properties of concrete, e.g. by reducing the heat of hydration, improving durability and increasing resistance to chemical attack. To some extent it replaces cement, enabling the content of the latter to be reduced in concrete accordingly. For this application fly ash has to be produced according to EN 450-1 and EN 450-2.

• in road construction according to European Standards and national regulations. In addition to its use in concrete layers, fly ash is used in bituminous surface layers, hydraulically bound mixtures and in unbound road bases. The relevant quality requirements are set out in instruction sheets and technical requirements issued by national authorities or by European or national standards (i.e. prEN 13282, EN 14227).

• for cement production '

Fly ash is used as a raw material component (clay substitute) in cement clinker production or as a main constituent in the production of Portland fly ash cement or Portland composite cement. For the use as raw material component site specific requirements of the cement producer has to be met, for the production of blended cement the requirements in EN 197-1 & 450-1.

• for concrete blocks: national regulations

• for infill, that means filling of voids, mine shafts and subsurface mine workings according to national regulations of the mining authorities

• for production of bricks (leaning of fatty clay): national regulations

• in earthworks and landscaping. In earthworks and landscaping the mechanical properties of fly ash are used in setting up and improvement of road foundations (embankments), the construction of noise barriers, and for recultivation and soil improvement.

• for the production of mortar, floor screed and plasters and mining mortars/civil engineering products: national standards and requirements

In line with the energy demand curve and the seasonal working load of coal-fired power stations, fly ash is largely produced during the colder months of the year when business in the building industry is slack. Silos with a capacity of up to 60,000 tonnes have therefore been built at some power plants to provide dry temporary storage facilities for fly ash prior to its use as a concrete addition. In some cases, certified fly ash in particular is temporarily stockpiled in a moistened state during the winter months, before being re-dried in separate facilities in the summer months for subsequent use in the building materials industry.

The combustion process is fully controlled to meet stringent emission control parameters as well as to meet the requirements resulting from European standards for conformity evaluation of the products. Figure 2 shows the responsibilities of the producer for e.g. fly ash for concrete according to the European standard EN 450-2.



Figure 2. The production of certified fly ash according to EN 450

The complete combustion process has to be described in a works quality manual and the process is monitored by a Notified Certification Body (third party control). A similar system for conformity evaluation is required by the European standard for lightweight aggregate.

3. PRODUCTION AND UTILIZATION OF ASH PRODUCTS.

3.1 Production of ash products in Europe

Figure 3 shows the development of CCP production in EU 15 members states from 1993 to 2006. The total amount decreased from 57 million tonnes in 1993 to 55 million tonnes in 1999 and rose again to 64 million tonnes in 2005 due to higher production of electricity and heat by coal combustion. In 2006, the amount of CCP produced in European (EU 15) power plants totalled 61 million tonnes, about 3 million tonnes less in the EU 15 member states compared to 2005. This reduction was caused by smaller production by coal combustion in some countries due to higher production by hydro power or the installation of de-NOx and de-SOx measures. In 2006, all combustion residues amount up to 80 % and the FGD residues up to 19 % by mass.



Figure 3: Development of the CCP production in Europe (EU 15) from 1993 to 2006

The development of the production of fly ash form hard coal and lignite combustion in drybottom boilers is shown in figure 2. Although in 2006 a smaller production of mostly hard coal fly ash for the EU 15 members states is observed it has to be noted that this figure do not reflect the situation in the single EU member states. In some countries the production was at same level or even higher than the year before.



Figure 4: Development of the production of fly ash from hard coal and lignite in EU 15 from 1993 to 2006

In the next years an increase of CCP is expected due to retrofitting of existing coal fired power plants with FGD based on requirements of the Large Combustion Plants Directive (LCPD), the construction of new coal fired power plants in some countries as well as the increased use of imported coal with higher ash content.

3.2 Utilization of ash products.

The rates of utilisation, temporary stockpile and disposal for the single CCP are given in figure 5.



Figure 5: Utilisation, temporary stockpile and disposal of coal ash in Europe (EU 15) in 2006

4. UTILIZATION OF THE CCP'S

4.1 Utilisation of fly ash

Fly ash obtained by electrostatic or mechanical precipitation of dust like particles from the flue gas represents the greatest proportion of the total CCP production. Depending on type of coal and type of boiler siliceous, silica-calcareous or calcareous fly ashes with pozzolanic and/or latent hydraulic properties are produced throughout Europe. The utilisation of fly ash across European countries is different and is mainly based on national experience and tradition.

In 2006, about 21 million tonnes of fly ash were utilised in the construction industry and for production purposes in underground mining. Most of the fly ash produced in 2006 was used as concrete addition, in road construction and as raw material for cement clinker production. Fly ash was also utilised in blended cements, in concrete blocks and for infill (that means filling of voids, mine shafts and subsurface mine workings) (see figure 6a).

4.2 Utilisation of bottom ash

Bottom ash is a granular material removed from the bottom of dry bottom furnaces operated at furnace temperatures of 1000 to 1200°C. Bottom ash is much coarser than fly ash. About 3.1 million tonnes of bottom ash were used in the construction industry. Out of this 41 % was used as fine aggregate in concrete blocks and in concrete and 46 % in road construction (see figure 6b).





5. REVISION OF EUROPEAN STANDARDS

5.1 Importance of different standards

The impact of European standards which are relevant to CCP utilisation is dealt with by ECOBA (European Coal Combustion Products Association). Over the last years, the main work items were related to the implementation of the European standards on fly ash for concrete EN 450-1 "Fly Ash for Concrete, the test procedure for setting time and soluble phosphate in EN 450-1 and actually the revision of EN 450. In addition, the national provisions for the use of fly ash according EN 206, regulations for concrete in contact with drinking water were compiled. To consider the specific aspects for calcareous ash in European standards a working group was founded. The working group is compiling the existing knowledge in European countries regarding the composition and properties of calcareous fly ash as well as existing standards and realized construction projects.

ECOBA is active in the development of European standards and represents the producers of CCPs on a number of CEN committees (examples include CEN TC 51 "Cement and Building Lime", CEN TC 104 "Concrete", CEN TC 154 "Aggregates", CEN TC 227 "Road materials", CEN TC 241 "Gypsum and Gypsum based Products" and CEN TC 351 "Construction Products: Assessment of release of dangerous substances").

5.1 Revision of EN 450-1 and EN 450-2

The application as concrete addition constitutes the highest added value for fly ash. By this, the European Standard EN 450 "Fly Ash for Concrete" is particularly important for the marketing of fly ash. The standard was first published in 1994 and the revised standards EN 450-1 und EN 450-2 entered force on January 1, 2007. EN 450-1 deals with definitions, specifications and conformity criteria for siliceous fly ash, which is produced by burning of pulverized coal, with or without co-combustion materials, and collected in a dry state, or which is processed by e.g. classification, selection, sieving, drying, blending, grinding or carbon reduction or by a

combination of these processes. This is because in some countries fly ash has been processed according to national regulations for years or, in some cases, decades. EN 450-2 deals with the conformity evaluation of fly ash for concrete in power plants in processing plants. Most important is the documentation of procedures for the production control in a works quality manual.

In EN 450-1 requirements regarding homogeneity, soundness and effectiveness are stipulated. The chemical requirements refer to e.g. the loss on ignition, sulphur-, chlorine- and free-limecontent. If fly ash is produced with co-combustion the content of reactive SiO₂, the total oxide content of SiO₂, Al₂O₃, Fe₂O₃, the alkali-, MgO- and phosphate-content have to be tested. The physical parameters stipulate requirements on the fineness, variation of fineness and density, on soundness and activity index. In addition to these requirements, fly ash from co-combustion has to meet the requirement of initial setting time, fly ash of category S those of the water demand. Within the ongoing revision of the standards all parameters are subject to critical review. Proposed changes will be documented in a respective background report.

The work on a new EN 450-1 and EN450-2 is proceeding with full speed. The expectation is that a revised standard will come into force in 2010. In the present draft of the standard the most important changes is:

- potential for increased co combustion of biomass fuels including animal husbandry waste
- definition of biomass fuels with reference to European standard.
- streamlining of chemical analysis and updating on some limit values
- there are still issues that needs reflections such statistical evaluation of compliance etc

6. PRESENT LEGAL ISSUES

The impact of environmental regulations on management and utilisation of CCP is also investigated by ECOBA. Some issues dealt with over the last years were the "Thematic Strategy on Prevention and Recycling of Waste", the "Amendment of the Waste Framework Directive", the "Commission Guidelines on By-Products", "Environmental regulations for the application of CCP in concrete for road construction, in contact with soil/ground and in contact with drinking water", REACH - "Registration, Evaluation and Authorisation of Chemicals", the "Assessment of Release of Dangerous Substances (CEN TC 351) and the "Harmonised Standards Code" (HSC) of the World Customs Organisation (WCO). In close so-operation with EURELECTRIC the committee prepared comments and status reports within the ongoing revision of the Waste Directive.

6.1 Revision of the Waste Directive

According to the European Waste Framework Directive from 1991 waste is defined as follows:

"Waste shall mean any substance or object in the categories set out in Annex I, which the holder discards or intends or is required to discard".

The categories in Annex I as mentioned above include: "Q 8 residues of industrial processes (e.g. slags, still bottoms, etc.)" and "Q 9 residues from pollution abatement processes (e.g. scrubber sludges, bag house dusts, spent filters etc.)".

Due to this Directive, CCP have legally to be considered as waste. Since the early 90ties of the last century discussions took place on the question if in certain cases a by-product from industrial processes is covered by the definition, i.e. has to be considered as waste or if waste properties could cease at a specific stage of the managing process. A typical example is FGD gypsum, which is on one hand a residue of a pollution abatement process and on the other hand was produced from scrubber sludge by an oxidation, cleaning and drying procedure aiming at a material, which meets technical specifications of the users (gypsum and cement industry). After several years of discussion it is now generally accepted by the authorities that FGD gypsum has ceased the waste properties after the processing in the power plant.

The case is not clear for fly ash as it is argued that no processing takes place in the power plant and that the recovery operation is the final use of the material. That would mean that the material is to be handled (collected, transported, stored) as a waste. A concrete producer would use a waste to produce concrete, i.e. a ready mixed concrete plant becomes a waste handling plant. Even if the restrictions or the additional paper work required by the authorities are not too heavy it is the image problem of the concrete, which might become an additional obstacle for the concrete producer to use fly ash.

In the course of the current process of the revision of the Waste Directive the discussion on the legal definition of by-products and end of waste criteria was restarted at the European institutions. Whereas the Commission preferred not to include a definition in the Directive and to give some guidance to the industry by guidelines, Parliament and Council were in favour of a definition for by-products, based on the jurisprudence of the European Court of Justice, in the Directive. After long discussion between the Commission on one hand and Council and Parliament on the other hand in the sitting of the parliament the introduction of a definition of by-product and of "end-of-waste" was agreed in the Waste Directive.

In article 6 of the Directive "end-of-waste" is defined by certain criteria to be defined by the Commission. The criteria shall *include limit values for pollutants where necessary and shall take into account any possible adverse environmental effects of the substance or object.*

The Commission ordered the Institute for Prospective Technological Studies (IPTS) and DG Joint Research Centre (JRC) to develop a general methodology for determining end-of-waste criteria. The methodology will be evaluated for aggregates, compost and metal scraps. For these materials pilot studies were be prepared and discussed at stakeholder workshops. The methodology is assessment based as they have to consider the wide range of waste materials. For aggregates the criteria will be based on leaching limit values. The final report will soon be available.

It has to be mentioned that in many European countries or even regions some CCP have already been accepted as by-products by the authorities. Furthermore, it has to be noted that CCP which are not subject to waste legislation are then subject to the REACH regulation.

6.2 **REACH Regulation**

On 1st June 2007, the REACH-Regulation (Registration, Authorisation, Evaluation and Restriction of Chemicals) entered into force. The overriding goal of the regulation is to improve the protection of human health and the environment from the risks of chemicals while enhancing the competitiveness of the EU chemicals industry. By this, all chemicals manufactured in or

imported into the EU have to be registered at the European Chemicals Agency (ECHA). The registration requires information on the properties and the potential risks of the substances.

REACH is not specifically made for CCP. But as CCP are mainly utilised in the building material industry, in civil engineering and in road construction they are placed on the market and for many applications they are subject to REACH.

Each producer or importer of coal combustion products (CCP) placed on the market as construction materials have to pre-register and to register their substances. The pre-registration requires information on the substance identity, the tonnages and the name and address of the producer. The registration requires i.e. comprehensive information about toxicology and ecotoxicology of the substances.

In Europe, non registered substances can not be placed on the market after 1st June 2008 any more! For CCP, since they are already registered in the European Inventory of the Existing Commercial Chemical Substances (EINECS) the deadline for registration is extended to 30 November 2010. This is only true if the producer pre-register in the period of 1st June to 30 November 2008!

6.3 Environmental compatibility of the use of CCP

There are many environmental benefits connected with the use of CCP as saving of natural resources, saving of energy, saving of emissions of pollutants to the air, saving of CO_2 emissions and saving of disposal space. Nevertheless, the environmental impact of the use of CCP has to be considered in any application. Fly ash and bottom ash as any natural minerals contain a certain amount of trace element compounds. The concentrations of some of the trace elements may be higher in fly ash than in natural minerals or products used for a certain application. In order to avoid any negative impact on the environment or on human health, regulations have been developed for the different uses of industrial by-products at a national level in the European Member States.

In November 2005, CEN established a new Technical Committee (CEN/TC 351) for "Construction products: Assessment of release of dangerous substances". The TC shall develop horizontal standardised assessment methods for harmonised approaches relating to the release (and/or the content when this is the only practicable or legally required solution) of regulated dangerous substances under the Construction Products Directive (CPD) taking into account the intended conditions of the use of the product. It addresses emission to indoor air, and release to soil, surface water and ground water. With the assessment methods information may be given for the CE marking of construction products on the release of dangerous substances in the use phase.

7. SUMMARY

In Europe (EU 15) about 61 million tonnes of Coal Combustion Products (CCP) were produced in 2006. The annual production in EU 27 is estimated to amount to about 100 million tonnes. The CCP include combustion residues such as boiler slag, bottom ash and fly ash from different types of boilers as well as desulphurisation products like spray dry absorption product and FGD gypsum.

CCP are mainly utilised in the building materials industry, in civil engineering, in road construction, for construction work in underground coal mining as well as for recultivation and restoration purposes in open cast mining. They are used as a replacement of natural resources. Their utilisation helps to save natural resources and to reduce the energy demand and greenhouse gas emissions to the atmosphere caused by mining and generation of products which are replaced by CCP.

The use of CCP has several environmental and technical benefits. It has developed by the years and is mostly based on requirements of standards or other specifications which are subject to regular revision by CEN or national authorities. At present, the European standards EN 450-1 and EN 450-2 are under revision. Within the ongoing revision of the standards all parameters are subject to critical review. Proposed changes will be documented in a respective background report. Furthermore, the European standard for hydraulic road binders with basic definition also for FBC ash as a main constituent will be published in an updated version with three parts.

The utilisation is becoming more and more restricted by environmental regulations. A European Technical Committee is working on horizontal standardised assessment methods for the release of dangerous substances from construction materials. In addition, the legal definition of CCP as waste causes hurdles, which are unnecessarily impeding the utilisation markets, which have been developed in the last decades. With the revision of the Waste Directive a definition of by-products will be introduced for materials which are from the beginning not a waste. Materials may also leave the waste regime after a recovery operation and meeting of waste stream specific end-of-waste criteria. For coal ash as aggregates these will be based on leaching limits.

Materials not being waste are subject to REACH and have to be registered before being placed on the market. For CCP, a special regulation can be used since they are already registered in the European Inventory of the Existing Commercial Chemical Substances (EINECS). By this, the deadline for registration is extended to 30 November 2010. This is only true if the producer preregister in the period of 1st June to 30 November 2008!

Ageing Factor Concept – Chloride Intrusion



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ABSTRACT :

The paper gives the background for the most used models for predicting chloride ingress into concrete with a focus on the everchanging material resistance which is modeled by a time-dependent diffusion coefficient. The time-dependant "ageing factor" α depends on a number of variables. The paper gives an overview of ageing factors reported by the author and from other sources. A method to derive information on the ageing factor from old structures where no previous measurements are available is described.

Knowledge on this aging effect of the particular concrete composition is vital to get the by CEN proposed "Equivalent Durability Concept" operable.

Keywords: Chloride ingress, concrete, ageing factor, "Equivalent Durability Concept"

1. THE CHALLENGE

When designing a new structure, or assessing remaining service life of an existing, the engineer need the help of a crystal bowl.

Based on information available at the time of the judgement, she/he has to forecast the future performance of the structure in its actual exposure conditions.

This has been regarded as a challenging profession all the time since the age of the biblical prophets.

Full-scale in-field experience for reinforced structures is only available on objects built after 1867 - 78 when Joseph Monier applied for his patents. However, the cements, steel and the rest of the construction technique of that time are not too relevant to that used today. Actually, ribbed bars were only widely adopted in the middle of the last century. Its yield strength has gradually increased from some 250 N/mm² till today's 500 N/mm². The cements and use of additions are still rapidly changing.

We therefore can't base our judgements only on previous documented performance, but we are forced to extrapolate short and medium-term experience into excepted in-field performance after 50, 100 or more years.

Such an extrapolation must be assisted by some kind of reasoning or modelling.

The younger the specimens or structure are, the more the performance tests are accelerated to derive information from the material, the more doubtful is the representativeness of the data, and the greater is the influence of the data and model uncertainties [1] [2].



Figure 1 – The significance of the prediction of service life will increase with time of the assessment and the quality of the analytical model [2]

2. SERVICE LIFE PREDICTION

The benchmark for today's experts in this field must be the first ever built reinforced bridge at Chazelet by Monier in 1875. This is actually still in use and is more or less in an acceptable condition!



Figure 2 – Bridge at the Castle of Chazelet. Designed and built by J. Monier in 1875 with reinforced concrete

In Norway, like in major parts of the world, we experienced major difficulties with bridges, marine structures and other chloride exposed structures in the 1980s. The problems were in particular on structures built according to our standards from 1972. In these documents the experts had, based on positive assessments of existing structures [3], relaxed the requirements for cover and w/c-ratios considerably. The response was that we in the 1986 revisions of the standards reintroduced low w/c-ratios (in the order of 0.45) and increased the cover to the reinforcement (typically > 50 mm in the splash zone). These were immediate actions based on the "stomach feeling" of the members in the code committee.

Some of us realized that these measures should be better anchored. We therefore started to search for a model enabling us to predict chloride ingress based on short and medium-term observations.

3 MODELS FOR CHLORIDE INGRESS

3.1. Fick's 2nd law of diffusion

In those days Fick's 2nd law of diffusion was used as proposed by Mario Collepardi in the 1970s [4].

$$C(x,t) = (C_s - (C_s - C_i)) \cdot \left[\operatorname{erf} \frac{x}{2 \cdot \sqrt{D \cdot t}} \right]$$
(1)

- C(x,t): content of chlorides in the concrete at a depth x (structure surface: x = 0 m) and at time t [wt.-%/c]
- C_s : Chloride concentration at the concrete surface [wt.-%/c]
- C_i: initial chloride content of the concrete [wt.-%/c]
- x: depth with a corresponding content of chlorides C(x,t) [mm]
- D: coefficient of chloride diffusion through concrete [mm²/years] at time t
- t: time [years] of exposure
- erf: error function

However, we soon experienced the same as other communities world-wide that the results of such modelling resulted in obvious too conservative results. In Selmer (today Skanska Norge as), Magne Maage, Jan-Erik Carlsen and myself started systematic collection of in-field data as well as undertaking parameter studies under laboratory conditions to improve the accuracy of Collepardi's model.

In the beginning of the 1990s we observed that the diffusion coefficient to Fick's 2^{nd} law decreased as a straight line when plotted in a log-log scale (D versus time of exposure). The inclination was in the order of -0.6 to -0.7 [5][6][7].

Together with Erwin Poulsen from Denmark and Øystein Vennesland, we later published this finding [8] in the mathematical expression of:

$$D_{app}(t) = D_{app}(t_0) \left(\frac{t_0}{t}\right)^{\alpha}$$
(2)

- D_{app} (t₀): is the apparent diffusion coefficient measured after a reference period of exposure of t₀
 (At that time we used thee term "effective". "Achieved" diffusion coefficient has also sometimes been used in stead of "apparent")
- α: is the ageing-factor giving the decrease over time of the apparent diffusion factor due to continued reactions of the binder and, in this respect, beneficial effect of the sea-water due to ion exchange with subsequent pore-blocking in the surface layer etc.

The "apparent" diffusion coefficient is replacing the D in the Fick's 2^{nd} law (eq. 1), and thus represents a number giving the same result on the predicted chloride profile after a period of exposure, t, as an incremental calculation based on the actual decreasing instantaneous D would have given.

Independent, and at the same time, Phil Bamforth from UK presented a similar expression [9] [10].

Later we have also learned that Takewaku and Mastumoto of Japan introduced a (very modest) time-dependency (equivalent to $\alpha = 0.1$) for the diffusion coefficient in a paper published in 1988 [11].

In the late 1990s the so-called DuraCrete-model was launched based on the same "core" as the Selmer and Bamforth-model [12]. However it includes a sub-model making it possible to base the predictions on results from the Rapid Chloride Migration (RCM) test (NT Build 492). The result from this accelerated test is then transformed to the actual diffusion coefficient used in the calculation by a sub-model taking the in-field temperature into account. It does also include a sub-model for how to handle the depth of a possible convection zone (concrete surface layer, up to which the process of chloride penetration differs significantly from Fick's second law of diffusion).

Today another variant of the Fick's 2nd law is also in use. In this not only the diffusion coefficient is time-dependant, but also the surface chloride concentration, Cs.

While nobody question that there is a build-up period of some years for the Cs, this variant claims that this increase is normally significant over the whole service life and that the simplified approach of assuming a constant value introduces major flaws in the predictions. Models in this category are the Hetek-model and the Mejlbro-Poulsen Model [13] [14].

According to the Hetek model, the build-up of the surface chloride concentration obeys the equation:

$$C_{s}(t) = C_{i} + S \cdot \left[\left(t - t_{ex} \right) \cdot D_{aex} \cdot \left[\frac{t_{ex}}{t} \right]^{\alpha} \right]^{p}$$
(3)

This equation allows the user to choose parameters representing all variations from a time-independent Cs to an ever-increasing number.



Figure 3 – An example of the time-dependency of Cs(t) depending on the chosen exponent" p" according to equation 3 (By Lars-Olof Nilsson [15])

Other variants of models, more or less based on Fick's 2nd law, are "Life-365", "LEO", and "LERM" and "JSCE".

To my knowledge the Japanese model developed by Japanese Society of Civil Engineers (JSCE) and revised in 1999, is the only one "in modern time" that still assumes that the diffusion coefficient is constant over time.

3.2 Models not based on Fick's 2nd law

There are a number of other proposals presented during the last decade. Examples are those of :

- ClinConc model by Tang and Nilsson, Chalmers
- Li and Page based on the Nernst-Planck equation.
- STADIUM model by SIMCO and Laval University, Canada also based on Nernst-Planck
- MsDiff by LMDC in Toulouse, France also based on Nernst-Planck

These models are not included in the further parameter-discussions in this paper.

3.3 Detailed presentation of the above mentioned models

The references and detailed description and discussions on the above mentioned models might be found in the report "ChlorTest – report W4 – Modelling of chloride ingress" by Lars-Olof Nilsson, Lund Technical University – LTH, from 2006 [15].

Our own "Selmer" model is described in full in the EuroLightCon report [16]

4 MATERIAL AND ENVIRONMENTAL PARAMETERS

4.1 Ageing effect on chloride ingress

The great number of small and major variations in the above referred models makes it difficult to directly compare results derived under different model-regimes as all in-field observations are processed by a best curve-fit to the actual equation.

The same raw-data (measured chloride profile) might therefore end up with different material parameters.

4.2 Time-dependant diffusion coefficient

Among all the models listed above, the most used due to its simplicity is Fick's 2nd law with a time-dependent apparent diffusion coefficient, i.e. the combination of equation 1 and 2

Comparison of the performance of concrete with different mix designs are here done by "only" 2 numbers, the diffusion coefficient, D_{app} (t_o), measured after a given period of exposure (reference time t_o) and the decline of the D_{app} (t) with time expressed by the exponent (ageing factor) α .

Different combinations of $D_{app}(t_o)$ and α might give different ranking of the effectiveness of the mixes at different length of exposure.

If only one list of ranking is wished, it must be the calculated D_{app} (t) at the end of the design service life, for instance $D_{app}(50 \text{ years})$ or $D_{app}(100 \text{ years})$.

4.3 Why does the D_{app} (t) decrease ?

It is obvious for all of us that the impermeability of the concrete improves as the cements hydrates and the additions react.

During the EuroLightCon project Selmer did undertake a number of tests where we stored specimens in fresh water and exposed them to sea-water after different periods of curing up to 1 year [16]. By comparing parallel specimens, we were able to distinguish the sole effect of prolonged curing in fresh water from the total effect of being exposed to sea-water. This component of the ageing factor α is in the Selmer-model named β .



Figure 4 - Effect of type of binder on β . Curing time in fresh water were 1, 7, 28 and 365 days before the specimens were exposed to sea-water. The results reflects the difference in D for companion specimens stored in sea-water and specimens stored in fresh water in periods given above + 35 days in sea-water to get a chloride profile to analyse. [16]

 β was found to be in the range of 0.1 - 0.2 depending on binder combination. This corresponds to an improvement of performance from 28 days to 1 year of 30 - 60 %. This is a little bit more than expected for other porosity-related performances as for instance compressive strength.

I do not have the background information on why Takewaku and Mastumoto proposed an exponent for the total ageing effect of 0.1 back in 1988. Probably it was a result of a consideration that an improvement similar to long-term strength gain might be reasonable also for this performance.

Since the total ageing factor α always are found to be bigger than the β , the difference in performance due to being stored in sea-water compared to fresh water, must be the, in this respect, positive influence of the mineral ions in the saline solution. This additional effect is in the Selmer-model named γ .

We then get the relation:

$$\alpha = \beta + \gamma \tag{4}$$

The importance of this relation is that we then are able to get some information of the ageing effect from an existing structure, even if we do not have any previous measurements from it.

The procedure is then to drill a core from the structure that has been salt-exposed for a period of t_1 . From the exposed surface the chloride ingress is mapped. Based on this profile the $D_{app}(t_1)$ is calculated by curve-fitting to the model.

Then we expose the virgin inner part of the core to an immersion test, for instance the NT Build 443 (5 weeks in saline solution) and derive the $D_p(35 \text{ days})$ by curve-fitting. If we ignore the effect of the continued hydration from start of exposure of the structure till we drilled the core at t_1 , we have the relation based on equation 2:

$$\frac{D_{app}(t_1)}{D_p(35 \, days)} = \left(\frac{35 \, days}{t_1}\right)^{\gamma} \tag{5}$$

From this the γ might be derived.

Knowing that the $\beta > 0$, and probably < 0.2, we also have an idea of the magnitude of the total ageing factor, α .

Figure 5 from 1993 [5] [7] demonstrates this exercise based on 7 marine structures of age up to 37 years. Except for object no 1, they are all based on CEM I cements and with w/c from 0.43 to 0.58.

To be able to compare structures with different qualities, we did normalize the plot by using the ratio $D_{app}(t_1)/D_p(35 \text{ days})$ as the scale for the 2nd axis.

The question is then why the diffusion coefficient decreases faster in a saline solution than in fresh water.

At the project "Shore Approach" on the west coast of Norway (ibid. [18]), we examined the surface layer of the concrete (CEM I and silica fume) after 8 years of exposure. The

examinations were done by SEM/EDAX (Scanning Electron Microscopy and Energy Dispersive Spectroscopy) and thin section microscopy. This revealed that Ca from the concrete had been exchanged with K and Mg from the sea-water in a thickness of about 2 mm. This resulted in a pore-blocking in this layer.



Figure 5 - γ derived from cores taken from 7 Norwegian coastal bridges of age up to 37 years. The γ were calculated based on equation 5. Except for bridge no 1, all bridges were constructed with CEM I cement without the use of any additions. Helgeland (1), Runde (2), Henningsvær (3), Gimsøy (4), Hadsel (5), Nerlandsøy (6), Vestnes (7) from [5]. Based on data from [17]

In 2002 Muhammed, Yamaji and Hamada published in ACI Materials Journal [19] a similar study on concrete specimens that had been exposed to the tidal zone in Tokyo Bay for 15 years. Their findings were exactly the same as those reported by Selmer/SINTEF from "Shore Approach", but the affected zone was deeper (5 - 15 mm). The Japanese samples comprised concrete based on OPC (CEM I) and blended cements with slag as well as with fly ash. The Japanese did find differences between the different binder types.

To introduce some confusion, results from 26 years old cores from "Shore Approach" will be presented by Magne Maage [18]. In spite of that these cores were drilled next to those after 8 years, and in spite of that they proved similar resistance to chloride ingress, SINTEF this time did not find significant ion-exchange in the surface zone.

Many discussions have taken place during the last 2 - 3 decades on if the application of Fick's 2^{nd} law is scientifically based or just a convenient equation for making curve-fitting against.

For practical application by engineers, this discussion does actually not matter too much.

Myself, I gradually tend to believe that what we observe in concrete of relevant quality is a process dominated by diffusion and that the material resistance to chloride ingress actually increases over time by the physical/chemical reasons explained above.

In some cases the measured profiles reflect a surface convection zone where the chloride transport must for periods also have been from the interior to the surface. To explain this, the concrete must have been subject to periods without external chloride loads and/or washing out by rain.

Our experience from marine structures with concrete compositions complying with modern standards is that the depth of such "disturbed" convection zones is much reduced. This is obviously due to the slow response in such impermeable materials to external variation.

For structures exposed only to de-icing salts during the winter season, the convection zone might be quite deep, and the application of modelling correspondingly problematic.

4.4 Time effect on surface chloride concentration, C_s(t)

If a model like the Mejlbro-Poulsen [13] is used to compare the relative differences in performances for different concrete compositions in structures subject to the same environment, not only the change in time for the material resistance, D_{app} (t), has to be looked into, but also the possible different build-up of the $C_s(t)$ over time. Different combinations of these two mechanisms might result in a great number of combinations.

Examples presented by some researchers claim that the ageing effect on the $C_s(t)$ might fully overrule the ageing effects on the $D_{app}(t)$ [15].

The unfortunate consequence by applying a time-dependant $C_s(t)$ is therefore that it is much more difficult for the engineer to draw general conclusions on mix designs compared to the situation with a time-constant C_s .

In Norway we have in Skanska worked quite a lot on this, particular in the EuroLightCon programme. A typical build-up phases for the $C_s(t)$ according to our experience is given in figure 6.

In parallel, we have derived much data from existing structures. Figure 7 gives the results from a number of offshore structures in the North Sea [20]. Based on these experiences, we have therefore made a pragmatic decision to regard $C_s(t)$ as constant over the whole service life.

The same pragmatic choice is also reported by Phil Bamforth [23] and by Gehlen and Schiessl in Germany [21].



Figure 6 – Build-up period of surface chloride concentration, Cs (% by weight of concrete), for some test series reported in [16].



Figure 7 – Surface chloride concentration, Cs, as measured from some 137 cores taken from concrete offshore platforms located in the Norwegian sector of the North Sea [20].

Personally, I do not have any experience with surface chloride concentrations, Cs, which does not reach a plateau after a few years.

However, I respect that other researchers have other experience, probably based on other mix compositions.



Figure 8 – Result from a Round-Robin exercise in the R&D project "ChlorTest". 16 research communities did, based on the same measured chloride profile from Träslövsläge after 40 weeks exposure predict the profiles after 50 years. The main difference is between those using a time-dependent $C_s(t)$ and those assuming a constant level. The results are reported in [15]

In some examples published, the Cs(t) at the end of the service life is extremely high. If it ends up in an order higher than those represented by figure 6 and 7, I fear that unjustified extrapolations of measurements during the first few years have biased these calculations.

For further discussions on the continued ageing effect on Cs I therefore refer to authors like Fredriksen and Geiker [13] [14].

5 WHAT IS THE AGEING FACTOR FOR VARIOUS CONCRETE COMPOSITIONS ?

For the effect of ageing on the material resistance, expressed by eq. 2, we normally use the exponent α as the parameter, or "ageing factor".

Our first experience was given in figure 5. These structures, up to 37 years old, also represent concrete based on CEM I and w/c-ratios in the range of 0.45 to 0.60. These were typical materials used for coastal bridges in Norway up to the end of the 1980s. Figure 9 gives the same data but updated in 1995 with some other observations.

Even if the laboratory results in the figure represent the total aging factor α and the results from the field only represent the γ - exponent, this curve demonstrated the validity of the "Selmer" model for a broad span of exposure periods.

The trend-curve for the field objects gives a $\gamma = 0.57$ which indicates that the α was in the order of 0.6 - 0.7 (reference to eq. 3).

In the EuroLightCon project [16] α was found to be in the order of 0.5 to 0.7 for different binder combinations with a mean of 0.6 Examples are shown in figure 10, 1 and 12.

These represent measurements up to a few years exposure and concrete compositions with various combinations of CEM I, silica fume, slag and fly ash.



Figure 9 – Figure 5 supplemented with Swedish (8) and Danish (9) reports. The results at 1, 4 and 25 weeks are from Selmer and are based 29 profiles from specimens immersed in sea-water in our laboratory [5] [22].



Figure 10 - Correlation between relative diffusion coefficients D/D_R and exposure time with D_R as the diffusion coefficient at 35 days of exposure after 28 days of curing in fresh water at 20 °C. The plot represent all together 305 profiles from LWAC [16]



Figure 11 - Effect of curing temperatures on diffusion coefficients for a LWAC-mix similar to that used in the Heidrun platform. [16]



Figure 12 - Correlation between diffusion coefficients and exposure temperatures $5 \,^{\circ}C$, $20 \,^{\circ}C$ and $35 \,^{\circ}C$ respectively for 3 different binder combinations cured in fresh water at $20 \,^{\circ}C$ for 28 days before exposure [16]

On the Heidrun structure I will later on this workshop [20] report on α in the range of 0.8 – 0.9 (CEM I and 7.5 % silica fume - LWA coarse aggregates). This was based on in-field measurements up to 9 years of exposure.



Figure 13 – Heidrun. Aging effect on diffusion coefficient (α). Laboratory data (exposure 1 to 4 months after casting) and in-field measurements after 2, 5 and 9 years [20]

A general trend is that the spreads in observations are big, particular when the measured $D_{app}(t)$ are in the order of 10^{-13} mm²/sec. For numbers in the decade of 10^{-14} mm²/sec, there are hardly any observations.

Experience including data from also other sources is collected in Concrete Society Technical report no 61: 2004 "Enhancing reinforced concrete durability" by Phil. Bamforth [23].

Figure 14, 15, 16 and 17 from this report again reflects a big scatter in α , but he suggests, if no better data are available for the particular project, the following mean values depending of binder composition:

- Ageing jucior a jor various binaer	combinations [25].	
	Ageing factor α	
"modern" PC concretes	0.264	
"old" PC concretes	0.54	
pfa concretes	0.699	
ggbs concretes	0.621	
PC + silica fume (w/b=0.4)	0.56	

Table 2 – Ageing factor α for various binder combinations [23].



Figure 14 – From Phil. Bamforth [23]. Overview of reported α -values for concrete based on OPC. In his data he found a distinct difference in ageing of "old type" CEM I and "modern type" CEM I despite of the big scatter.



Figure 15 - From Phil. Bamforth [23]. Overview of reported α -values for concrete based on OPC. and Fly Ash.



Figure 16 - From Phil. Bamforth [23]. Overview of reported α -values for concrete based on binders with granulated blastfurnace slag.



Figure 17 - From Phil. Bamforth [23]. Overview of reported α -values for concrete based on OPC with silica fume addition.

In informative annex B to fib MC SLD [21], Christoph Gehlen has indicated the following numbers for the α -exponent:

Tuble 5 – Ageing exponent a for vario	Jus Dinael	r combination	S [21]	
concrete	Ageing	exponent α	1	
	Splash, 1	tidal and subn	nerged zone	
	Beta fun	oction		
	Mean	Stand. deviation	Lower bound	Upper bound
Portland cement concrete CEM I: $0.40 \le w/c \le 0.60$	0.30	0.12	0.0	1.0
Portland fly ash cement concrete $f \ge 20$ %; k=0.50; $0.40 \le w/c \le 0.62$	0.60	0.15	0.0	1.0
Blast furnace slag cement concrete CEM III/B; $0.40 \le w/c \le 0.60$	0.45	0.20	0.0	1.0

Table 3 – Ageing exponent a for various binder combinations [21]

It can be concluded that the scatter is big and that the average numbers reported by Phil, Christoph and myself deviates.

In my opinion this scatter reflects the lack of insight in what factors govern the ageing effect expressed with the α - exponent. Grouping the data according to the 27 defined types of cements in EN-197, or by type of addition used, might probably not fully reflect the decisive parameters. This is also clear in Phil's reports on CEM I were α ranges from 0.26 to 0.54 depending on being a "modern" or "old" type of cement.

Other factors like the mineral composition of the cement and their fineness obviously also influence the ageing.

In my opinion, great care should therefore be exercised when applying an α - exponent derived from other mix compositions than that particular we are going to model.

5.2 Determining the *α*- exponent

The α -exponent is derived from equation no 2 based on 2 sets of measured D_{app} representing 2 different periods of exposure.

The calculation improves in robustness and reliability when:

- the periods of exposures represented by the observations are long
- the spread in time represented by the observations are big
- the observations represent the actual exposure conditions

As will be reported later on this workshop [20], we tried to derive the α -exponent from in-field observations from Norwegian offshore structures. With the exception of the Heidrun platform (see figure 13), all the other structures had a too great spread in observed D, and the spread in time was too short to get any basis for deriving the ageing effect.

5.3 Using laboratory testing to determine D

It is quite normal to determine $D_{app}(t_o)$ based on laboratory testing. The model however assumes that the exposure conditions are the same for both sets of $D_{app}(t)$ in equation 2.

Possible differences between the laboratory regime and the in-field exposure must then be compensated for. Accelerated test based for instance on applied current (like NT Build 492) might reveal a relevant material property, but this must be further processed to be applied in the model.

The DuraCrete model includes such compensating sub-models.

The proposed reference test method by CEN has deliberately chosen to use immersion in a solution with similar salt concentration to that of Atlantic sea-water and over a fairly long period (3 months) to make the transformation of the result to in-situ conditions as transparent as possible.

This is also in line with the tradition in our group where we always have used natural sea-water as the medium and no artificial acceleration whatsoever.

6. AGEING FACTOR IN THE "EQUIVALENT DURABILITY CONCEPT"

In the proposed "Equivalent Durability Concept" (EDC) under preparation by CENT C-104/SC1/TG17 and presented here at the workshop in Hirtshals by Tom Harrison [24], another meaning of the "ageing factor" than the α -exponent is introduced.

In brief, the method is assumed used to compare the performance of a new "candidate" mix composition with unknown properties with the known properties of a reference concrete composition already covered by the relevant national annex to EN 206-1.

Since the only performance of interest is that at the end of the design service life (say 50 or 100 years), and the only information available to the engineer when doing the assessment is that from a test on the young concrete, some kind of modelling must be done.

The proposed test procedure will be to expose the specimen to a 3 % saline solution for 3 months. The test will start after 1 month curing. Based on the mapped chloride profile at the end of the test, D_{app} (3 months) is derived according to equation 1.

Figure 18 demonstrates that concretes with different ageing (α -exponent) must achieve different results in this 3-month test to give the same performance after 50 years.



Figure 18 – To perform equal after 50 years, concrete compositions with different ageing (different α) must perform at different levels after 3 months immersion test (as proposed by CEN TC-104/SC1/TG17 as reference test to support the EDC).

The basis for the example in figure 18 and table 4 are:

- 1. Surface chloride concentration of 0.7 % Cl of concrete (constant over time)
- 2. Cover to reinforcement of 50 mm
- 3. Critical chloride content of 0.07 % (negligible risk for corrosion)
- 4. 3 months immersion in 3 % NaCl-solution and 20 °C as the reference test

- 5. An assumption that the reference test gives the same D (3 months) as the structure will demonstrate in the actual in-field exposure
- 6. 50 yrs initiation period

Based on the parameter-studies Selmer did undertake during the EuroLightCon programme [4], as well as figure 5 and 9 of this paper, I feel that assumption (5) above is not too inaccurate.

Table 4 - Tabulated factors to multiply the outcome of a 3 months immersion test to ensure equal performance after 50 years (as in the Equivalent Durability Concept).

Needed D_{app} (10 ⁻¹² m ² /s) at 50 yrs	Aging factor α	Needed D at 3 months (reference test)	Factor to compare eqv. performance after 3 months testing
0.28	0.30	1.4	0.20
0.28	0.40	2.3	0.33
0.28	0.50	4.0	0.57
0.28	0.60	7.0	1
0.28	0.70	11.5	1.64
0.28	0.80	19.0	2.71

In Norway the reference concrete mix would probably have $\alpha = 0.60$.

Candidate concretes with other α -values must then perform different in the short term test. A candidate concrete with $\alpha = 0.80$ would pass the benchmarking if the outcome (D at 3 months) of the test is only 2.7 times that of the reference, while a candidate concrete with $\alpha = 0.30$ would need 0.2 times that of the reference to pass.

The number to multiply the D (3 months) of the reference concrete, to ensure equivalent performance at the end of the design service life, is in this concept named "Ageing factor".

The EDC assumes that the national standardization body defines the reference concrete composition and that the same body provides to the user of the standard a table with "Ageing factors" depending on the binder composition in the candidate concrete.

It is then clear that the user of the standard will not be trusted, or entitled, to do the modelling himself. However, the experts in the national code committee have to do this exercise on behalf of the users.

The challenge for these experts will then be to identify the correct α -exponent for all possible binder-combinations if they are doing modelling according to the simple equation 1 and 2.

If more complex models with time dependent surface chloride concentration are deemed necessary, the code committees will face even greater challenges.

I have been asked by the CEN committee to come up with a proposal for "Ageing factors" depending on the cement types defined in EN-197.

My problem is that my experience is only with the few binder combinations used in Norway. Even for these the scatter in the reported numbers is quite big and the basis for heated discussions.

I must therefore admit that I do not have the data needed to produce such a table. I do not neither know anybody else with such a complete database.

Possible ways to overcome this lack of knowledge might be to:

- Require additional information on the candidate concrete as a basis for the assessment, for instance long-term experience from the nation where the cement type is produced.
- Require documentation of the ageing property for the candidate concrete composition. Such documentation will require much longer time than the 1 + 3 month reference test described above.
- Add safety margins to compensate lack of precision in the data. Such margins will jeopardize the "fair competition" for the new candidate concrete, but it would protect the society against underperforming products.

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In-field performance of north sea hsc/hpc offshore platforms with regard to chloride resistance

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ABSTRACT

Since 1973 a total of 34 platforms for the oil and gas industry, representing 2 650 000 m³ HSC/HPC, have been installed in the North Sea. The structures have been closely monitored by the operating companies with regard to their in-field performance. The presentation analyzes chloride profiles from some 180 cores taken from 9 structures after 2 to 20 years service. The profiles are applied to assess the remaining service life by modeling. Based on this, the remaining service life as a function of reliability for not passing a critical chloride content at the reinforcement, are directly quantified. The findings are compared with the requirements given in the present Norwegian standards for concrete works exposed to a harsh marine environment.

Keywords: Marine concrete structures, chloride ingress, service life design

1. INTRODUCTION

1.1 Oil and gas installations in the north sea

Since 1973, a total of 34 platforms for the oil and gas industry, representing 2 650 000 m^3 HSC/HPC, have been installed in the North Sea. Both normal density and lightweight aggregate concrete have been used. Many of the platforms are shell structures were high compressive



strengths of the concrete is utilized to withstand water pressures of up to 300 meters.

Gradually during the last decades, the focus has increased on the durability aspect for marine structures, in particular for chloride induced corrosion. One remedy to slow down the ingress of chlorides has been to require a low w/c-ratio, the same measure as when producing high strength concrete.

Due to these circumstances, structures exist that might witness how a low w/c-ratio concrete performs over long time and under realistic exposure.

When calibrating the service life design requirements in the Norwegian concrete standards for marine structures, we might benefit from this.

The oil companies operating these platforms have during the years performed quite a lot of assessments based on data derived from cored specimens. Due to the nature of this industry, it has been difficult to compile and publish these data.

However, this paper represents data from 9 structures were the operators generously have released these data for the benefit of the industry. These data represent some 180 chloride profiles from cored specimens after 2 to 20 years of field exposure. These data have been analyzed by the authors. One of the authors has also been directly involved in the inspections. We are therefore also in the position to evaluate the representativeness of the data, and the amount of visible defects on the installations.

The coring was intended to be done on a random basis. However, the inspectors admit that visual defects had a tendency to attract the location of inspection. The authors therefore judge the representativeness of the data to slightly underestimate the actual quality in the structures. The data are from the tidal zone and up.

For all the structures, the submerged parts of the platforms are equipped with sacrificial anodes to protect the mechanical installations, pipings and inserts from corrosion. Since there is electrical contact between these installations and the reinforcement, this steel is also protected from corrosion even if chlorides contaminate the concrete. The durability of these lower parts is therefore not questioned.

2. EXPOSURE DATA FROM THE NORTH SEA

To describe the exposure conditions for offshore concrete structures located in the North Sea, the following data applies: The salinity of the North Sea is about 35 grams per litre.

The surface sea temperature ranges between 5 and 17 $^{\circ}$ C. Maximum wave high and wind velocity used in the design of a structure is about 25 - 30 m and 45 m/s respectively.

Such a climate should be characterized as harsh.

3. CONCRETE STRUCTURES ANALYSED

3.1 The Structures

Data from the following structures are included:

Table 1: Structures from the North Sea assessed in this paper (GBS : Gravity Based Structure, TLP : Tension Leg Platform)

Year of installation	Operator	Structure	Type
1973	Philips	Ekofisk	Caisson
1975	Shell	Brent B	GBS
1977	Mobil	Statfjord A	GBS
1986	Statoil	Gullfaks A	GBS
1988	Norsk Hydro	Oseberg A	GBS
1989	Statoil	Gullfaks C	GBS
1993	Shell	Draugen	GBS
1995	Conoco	Heidrun, (floater)	TLP
1995	Norsk Hydro	Troll B (floater)	Semisubmergible

Table 1 continued: Data for the structures

Structure	Water depth,	Concrete volume,	Design strength
Structure	meter	m ³	Design strength
Ekofisk	71	80 000	C-45
Brent B	140	64 000	C-45
Statfjord A	145	87 000	C-50
Gullfaks A	135	125 000	C-60
Oseberg A	109	116 000	C-60
Gullfaks C	216	244 000	C-65
Draugen	251	85 000	C-70
Heidrun	350	63 000	LC-60
Troll B	325	43 000	C-75

Cement content for the structures varied in the range of 420 to 450 kg/m³.

All the inspected parts of the structures were cast by slipforming.

3.2 Concrete Compositions

Key data for the concrete used in the assessed structures are:

Aggregate)				
Structure	Aggreg.	Cement	Silica fume	w/c+s
Ekofisk	NDA	CEM I	-	0.45
Brent B	NDA	CEM I	-	0.38
Statfjord A	NDA	CEM I	-	0.38
Gullfaks A	NDA	CEM I	-	0.38
Oseberg A	NDA	CEM I	-	0.37
Gullfaks C	NDA	CEM I	2 %	0.38
Draugen	NDA	CEM I	2 %	0.40
Heidrun	NDA / LWA	CEM I	5 %	0.39
Troll B	NDA / LWA	CEM I	7 %	0.35

Table 2: Concrete compositions (NDA = Normal density Aggregate, LWA : Lightweight Aggregate)

4. METHODOLOGY FOR THE ASSESSMENTS



Fig. 1 Typical chloride profile in concrete exposed to sea water. From Oseberg A after 9 years exposure

The analyses have been based on the assumption that the chloride ingress obeys a modified version of Fick's 2^{nd} law for diffusion where the diffusion coefficient is time-dependent [1], [2].

$$C(x,t) = C_s - (Cs - Ci) * erf\left(\frac{x}{\sqrt{4tD}}\right)$$

Where :

C(x,t) = Chloride content at depth x at time t.

- C_s = Chloride content on the exposed surface.
- C_i = Chloride background content.
- D = Diffusion coefficient.

- t = Exposure time.
- erf = Error function.

In this equation, C_S represents the environmental load, while the chloride diffusion coefficient, D, characterise the materials ability to withstand the ingress of chlorides.

D was earlier considered as a time independent parameter. However, it has been confirmed both by laboratory testing and by observations from existing structures of up to 37 years of exposure that this resistance is improved over time [2], [3] and obeys the mathematical expression:

$$\frac{D(t)}{D_o} = \left(\frac{t_o}{t}\right)^{\alpha}$$

Where D(t) is the time dependent chloride diffusion coefficient, t is the time (length of exposure) of the concrete, and D_0 is a measured reference diffusion coefficient determined by curve fitting to the measured chloride profiles of the cores and t_0 is the length of exposure when drilling the cores.

The parameter α is the co-called aging-factor. Except for the Heidrun platform, the spread in the diffusion coefficients were too big and the length between the measurements too short to determine reliable α -values.

Based on previous experience and practice [4], an $\alpha = 0.60$ has therefore been used in the calculations.

Although the specified cover to the reinforcement varied in the range 50 - 75 mm, the actual distributions of the cover are not available to the authors. Since the main purpose of this paper is to assess the present requirements to mix compositions and cover to the reinforcement in the Norwegian concrete standards (NS 3473: 2003 and NS-EN 206-1: 2003), a minimum cover to the reinforcement for Exposure Class XS3 (tidal, splash and spray zone) of 50 mm (50 years service life), is applied. This corresponds to a nominal cover of 60 mm.

The interpretation of these numbers done by the standardization committee was that a maximum of about 10 % of the surface rebars would have less than 50 mm cover.

The limit state considered in this paper is when a critical chloride content to trigger corrosion of the reinforcement reaches the surface of the reinforcement. These threshold values depend on a number of parameters like the binder type, w/c-ratio, moisture etc. We have used the numbers published by Roger Browne [5] in 1980.

In this paper we have reported the calculated initiation periods till threshold values of 0.07, 0.18 and 0.36 % of concrete weight are reached at 50 mm depth.

Since the calibration of the applied model obviously becomes more and more uncertain at longer periods of exposure, only initiation periods of up to 120 years are reported.

Cl ⁻ in % of cement weight	Cl ⁻ in % of concrete weight (supposed 440 kg cement/m ³)	Probability for corrosion of reinforcement
> 2.0	> 0.36	Certain
1.0 - 2.0	0.18 - 0.36	Probable
0.4 - 1.0	0.07 - 0.18	Possible
< 0.4	< 0.07	Minimal

 Table 3: Threshold values for chloride concentrations to trigger corrosion [5]

The level of 0.07 % of concrete weight is by [6] interpreted as a 10 % probability of depassivation and by [7] as a 10 - 15 % probability.

For a probabilistic based service life design, the distribution of surface chloride concentrations and the diffusion coefficient must be known [6]. In our case, we had sufficient data to do this exercise directly.

We used the actual surface chloride concentration and diffusion coefficient from each core. With these 2 input parameters to the model, the remaining initiation length for each specimen was calculated. Based on this, we plotted the accumulated service life. Since a level of reliability of 90 % (i.e. 10 % do not stand the requirement) is often used for such design calculations [4], [6], we focused on the lower tail of the accumulated curve.

This approach is chosen since traditional calculations based on average values and standard deviations will be incorrect due to the fact that for a number of the structures, a few results on the lower tail are obviously caused by other mechanisms than the rest of the population. This important information will be smeared out and hidden if we regard the whole population belonging to the same statistical distribution. This is discussed further in paragraph 7.

5. RESULTS FROM EACH STRUCTURE

5.1 Ekofisk

The Ekofisk caisson from 1973 was the first concrete structure to be placed in the North Sea after oil and gas first were discovered in this area. The caisson was designed with a high strength concrete of that generation (characteristic cube strength of 45 MPa which was the higest design strength covered by the design standard). The binder used was an unblended Portland cement (CEM I) and the w/c-ratio was 0.45

Based on 10 cores taken from the structure in 1990 [8], [9], the time to reach critical chloride contents of 0.07 and 0.18 % of the concrete weight at a depth of 50 mm, are given on Figure 2.



Fig. 2 Ekofisk (external wall of tank). Time to reach critical chloride content at 50 mm. Accumulated based on 10 cores taken from the structure after 17 years

As might be noted, it is not evident that the resistance to chloride ingress for this structure stands the expectations for a 50 years service life in the Norwegian standards with sufficient margin. However, for the actual structure, a cover of 60 mm was specified. This corresponds more or less to the present requirement in NS of 50 mm minimum and 60 mm nominal cover. The original designed service life was only 30 years. The caisson is decommissioned in these days after 35 years service without having being subject to any major repairs or strengthening and has thus fulfilled its design assumption. The topside and the processing installations are removed, but the concrete structure will remain at its location. The operators have to our knowledge no plans, nor see any needs for remedial actions to let it remain at the field for an additional undefined period.

5.2 Brent B

After 19 years service, 15 cores were drilled from the structure and the chloride profiles were measured [10]. Based on the reported profiles, the authors of this paper have further analyzed the behaviour of the structure.

The accumulated initiation periods for reinforcement at depth of 50 mm were as given in figure 3.



Fig. 3 Brent B

Time to reach critical chloride content at 50 mm. Accumulated based on 15 cores taken from the structure after 19 years

As might be noticed, the bulk of the population stands the chloride-rich environment very well. However, the situation is troubled by a few cores with close to no resistance. Since all concrete is produced according to the same specification, the potential of the mix is represented by the mean initiation period, while the underperforming cores must have got their potential destroyed during the concreting. A concrete mix with a better potential from the batching plant, or an increased cover could not have compensated for this situation.

5.3 Statfjord A

9 cores were taken from the structure 16 years after the installation [8], [9]. None of the cores showed any ingress of chlorides at all. The reason for this situation was a discussion of the frost resistance of the concrete in the mid 1970s. Due to some uncertainty it was decided to coat the shafts above sea level with epoxy to lower the level of humidity in the concrete. This was done during the slipforming. Even if we today do not question the frost resistance of such a concrete quality, the epoxy has effectively kept the chlorides away from penetrating the structure. In-field inspections also demonstrate that the epoxy coating is still intact today 31 years after being applied.

5.4 Gullfaks A

A total of 16 cores with an age of 7 and 12 years were available to the authors [8], [9], [11]. The cores were taken from 5 to 18 meters above the sea level.

By applying the Fick's 2^{nd} law to calculate the initiation period for a level of 50 mm cover, all cores got a result of many hundred years, even for the most conservative threshold value. This is beyond any calibration of the model.

5.5 Oseberg A

For this structure, a total of 87 cores taken from the structure between 9 and 18 years of exposure were available [8], [9], [11]. The predicted initiation periods for a depth of 50 mm are given on figure 4.



Fig. 4 Oseberg A

Time to reach critical chloride content at 50 mm. Accumulated based on 87 cores taken from the structure up to 18 years after installation

Like for the Brent B platform, the bulk of the cores demonstrate a satisfactory performance in this environment. However, again a relative few cores destroy the overall picture by having close to none resistance towards chloride ingress. Since all the cores are based on the same specification on concrete composition, these few cores must have got their potential destroyed during the concreting.

Like for the other structures with some underperforming cores, the underlying data confirms that the left tail of the distribution is due to high (bad) diffusion coefficients and the right tail is due to cores where the surface chloride concentration (environmental load - Cs) by some reasons are very low.

5.6 Gullfaks C

6 cores taken 7 years after installation [8], [9] of the structure have been analysed. By applying the most conservative threshold value of 0.07 % of concrete and 50 mm cover, one of the cores indicated 55 years of initiation period. The rest of the cores gave results >> 100 years.

5.7 Draugen

23 cores with exposure lengths between 2 and 14 years [11] have been analysed. The calculated initiation periods were as given in figure 5.



Fig. 5 Draugen

Time to reach critical chloride content at 50 mm. Accumulated based on 23 cores taken from the structure up to 14 years after installation

The situation became the same as reported for Brent B and Oseberg A. The mean initiation period, representing a realistic potential of the mix, demonstrated a good in-field performance, while the left tail of the distribution again is dominated by a few cores with close to no resistance towards chloride ingress.

5.8 Heidrun

This floating structure is the only one where all the coarse aggregates have been of expanded clay. This lightweight aggregate concrete had a density in the range of 1950 kg/m^3 . The silica fume content was also higher than for the other structures reported earlier in this paper.

A total of 36 cores with 2 to 9 years in-field exposure were available [11]. The lowest initiation period with criteria of 0.09 % of chlorides to the weight of the concrete (adjusted to fit the low density) and 50 mm depth was 120 years. The rest of the cores were calculated to many hundred years. This is again far beyond any calibration of the model.

For this structure, also excessive laboratory test results on chloride resistance measured on specimens exposed to sea water at an age from 1 day to 2 years and with up till 2 years of exposure, have been available [3].

5.9 Troll B

This is also a floating structure. Parts of the coarse aggregate were LWA to reduce the density with some 200 kg/m³ compared to a mix composed only with normal density aggregates. 12 cores [8], [9], [11], taken from the structure after 2 and 8 years, have been analysed.

All cores gave, after calculation, initiation periods of many hundred years for the lowest threshold value and for 50 mm depths. Again this is far beyond any calibration of the model used, and the structure demonstrated a very good performance in its harsh marine environment.

6. DISCUSSION OF THE INPUT PARAMETERS TO THE MODEL

6.1 Environmental load - Cs

The environmental load is represented by the calculated surface chloride concentration, Cs. The calculation is carried out by curve fitting to the model and has to be used together with the corresponding diffusion coefficient derived from the same curve-fitting. This method has been applied in this paper. Since all Cs-values used in this paper are from cores with more than 2 years of exposure, we have assumed that these concentrations have reached a stable level.

This assumption is supported by figure 6 were all Cs-values are plotted as a function of age.



Fig. 6 Surface chloride concentration (Cs) as a function of age after installation of the platform. Field data from 8 structures.

The mean Cs value for 137 analysed cores was 0.63 % chlorides by weight of concrete with a standard deviation of 0.43 % (results from Statfjord A not included).

To further gain experience with the effect of the local exposure conditions on these structures, we have also looked into the effect of height above sea (the concrete shafts typically raise a little less than 30 meters from sea level). The results for heights above sea level are given in figure 7 and do hardly demonstrate any significant variations. The climatic conditions obviously make the saline environment equally harsh all the way to the top of the shafts.

The effect of the orientation has also been analysed. The dominating wind direction is from Southwest. Figure 8 indicates that the environmental load is somewhat higher on this side, but the effect is hardly significant.



Fig. 7 Surface chloride concentration (Cs) as a function of height above sea level. Field data from 8 structures.



Fig. 8 Surface chloride concentration (Cs) as a function of orientation. Field data from 8 structures.

6.2 Aging effects

In the applied model, the coefficient α represents the aging. This factor takes into account both the effect of continued hydration/reaction of the binder as well as the effect of the interaction between the concrete skin and the sea water. This interaction is mainly due to ion-exchange which results in blocking of the pore structure [12], [13].

The parameter α is normally a very influencing factor, especially when the model is used with data from young concrete. To reduce its influence in this study, the model is deliberately only

applied with input data from mature structures. Data from the Heidrun structure is the only one with sufficient precision and difference in age to calculate a reliable α .

On figure 9 the aging effect on the effective diffusion coefficient are given by curve-fitting. This gives $\alpha = 0.82$ when the in-field measurements are used together with laboratory results on young concrete specimens. If only the field data from 2 till 9 years of exposure are used, the α becomes 0.88



Fig. 9 Heidrun

Aging effect on diffusion coefficient (α). Laboratory data (exposure 1 to 4 months after casting) and in-field measurements after 2, 5 and 9 years

The first and third authors have earlier reported aging factors in the range of 0.70 from concrete coastal bridges with up to 37 years of service [1], [2]. However, it has to be admitted that also lower values have been reported. As stated under paragraph 4, all results in this presentation are therefore based on $\alpha = 0.60$.

To validate the robustness of this choice, parallel calculations have been performed with $\alpha = 0.50$

Not surprisingly, this last exercise had minor influence on calculated initiation times. This is partly due to the fact that extrapolations of mature situations were made and partly because the relative few "problematic" cores reported earlier demonstrated so bad performance anyway that different aging factor did not make much difference.

7. WHY DID THE INVESTIGATION REVEAL UNDERPERFORMING CORES?

The second author has been responsible for much of the in-field inspections carried out on these offshore structures.

His firm opinion is that local damage is observed when:

- The cover to the reinforcement is less than the specification (not a part of this paper)
- When the cover to the reinforcement is insufficient, compaction of the concrete cover is difficult and the quality of the concrete cover is consequently often poor. This is particular relevant for slipforming.
- In certain areas of the concrete surfaces, lifting cracks and poorly cast concrete has to some extent been observed. The reasons for these defects are mainly:
 - o Insufficient concrete cover to the reinforcement
 - Geometry changes in the concrete walls
 - Insufficient compaction of the concrete cover
 - Too low lifting velocity of parts with complex geometry, parts with extensive installations of embedments and parts with extreme amounts of reinforcement.

8. CONCLUSIONS

The in-field inspections clearly demonstrate that the structures perform well and according to their design assumptions. The number of deficiencies is limited and is not considered by the operating companies as giving problematic amount of maintenance.

The study clearly demonstrates that the durability potential of a concrete with w/c-ratios in the range of 0.40 (we are excluding the Ekofisk with w/c = 0.45) and a minimum cover of 50 mm, gives a service life in this saline environment of more than 50 and 100 years.

The study further clearly demonstrates that the critical factor has been the execution on site. If this is jeopardized, it does not matter how tight against chloride ingress the mix is designed for and how big the specified concrete cover is.

This is not a material (as regards its potential as delivered from the batching plant) nor a concrete cover problem, but the result of problems during the concreting.

To further improve the performance of such structures, the focus should therefore not only be the traditional one on mix compositions and increased cover, but on constructability and executional aspects. When the optimum design is based on advanced slipforming with varying diameters and shape, like on some of the reported structures, the accompanying uncertainty for the quality of the cover zone should be taken into account and compensated for, for instance by applying epoxy coating during the construction, as such treatment has demonstrated excellent field behaviour.

Undoubtedly, the economical benefits gained by applying these sophisticated geometrical shapes that caused the local quality problems for the cover zone, by far outnumber the added cost for maintenance during the structure's service life or better, remedial actions to detect and compensate deficiencies before the structure leaves the construction site.

The authors feel confident that the present Norwegian standard, NS 3473 and NS-EN 206-1, requiring a water-binder ratio of less than 0.40 combined with the use of pozzolans or slag, and a minimum cover of 50 mm, is adequate to ensure more than 50 years design service life with a level of reliability of more than 90 %.

We hope that the coming new European standard for execution of concrete structures, EN 13670; 2009 and its international counterpart ISO 22966: 2009 will be used as a tool to ensure that the design potential also will be realized on the construction sites.

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Frost resistance of concrete - Experience from three field exposure sites



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ABSTRACT

Concrete samples made from different cement/binder types, including secondary cementitious materials, have been exposed at three different field test sites for over ten years. All the sites are situated in Sweden: one in a highway environment, one in a marine environment and one in an environment without salt exposure. The resistance to internal and external frost damage has been regularly evaluated by measurements of change in volume and ultrasonic pulse transmission time. The results after ten years' exposure clearly indicate the highway environment as being the most aggressive with regard to external frost damage. The influence of climate on the internal frost damage is less pronounced. The results after ten years' exposure show that concrete with CEM I, CEM II/A-LL, CEM II/A-S, CEM I + 30% slag and CEM I + 5% silica as binder, with entrained air and a water/binder ratio of 0.5 or below, has good resistance to internal and external frost damage. Results show that concrete containing large amounts of slag in the binder (CEM III) have the severest scaling, whether with or without entrained air. For concrete without entrained air, qualities containing CEM I + 5% silica as binder seem to be more susceptible to internal damage than do the other qualities. Carbonation depth was measured on specimens exposed for eleven years at the test site without salt exposure. Results show a small carbonation depth for all concrete qualities, except for concrete with CEM III as binder, for which the carbonation depth was markedly greater.

Key words: Freeze/thaw, Carbonation, Field test, Laboratory test, Secondary cementitious materials

1. INTRODUCTION

Resistance to external and internal frost damage is of great importance in determining the durability of concrete in the Scandinavian countries, as well as in other European countries with cold climates. To be able to prove concrete qualities to be resistant to external damage (scaling),

test methods such as CEN-TS 12390-9[1] or CEN TR 15177 [2] have been developed, Finnish Standard SFS 5448 for dilation [3], and the measurement of critical degree of saturation [4] for internal damage.

These test methods have been developed primarily on the basis of experience of traditional concrete made with ordinary Portland cement. When new types of concrete are introduced - for example, with new types of binders, filler materials, admixtures etc. - we do not know how to evaluate the test results or even if the freeze/thaw test methods used are relevant. More knowledge and experience of the salt/frost resistance of these new concrete qualities in the field is needed. One way to acquire this experience is to expose concrete specimens to representative outdoor environments. Such an investigation was started in Sweden in the mid-nineties. Three field exposure sites were established in the south-west of the country: one in a highway environment beside highway 40 (60 km east of Gothenburg), one in a marine environment at Träslövsläge harbour (80 km south of Gothenburg) and one in an environment without salt exposure on SP's premises in Borås (70 km east of Gothenburg). The air temperature and relative humidity ranges are the same at the highway exposure site and at SP's premises, with minimum temperatures between -15 °C and -20 °C, and with a precipitation of about 900 mm per year. The temperatures at the marine exposure site at Träslövsläge harbour are somewhat milder, and the precipitation is about 700 mm per year. The micro-climates surrounding the test specimens, however, vary significantly between the three sites, with the highway microclimate being the most moist and saline, and the climate at SP being the 'mildest', with no salt and only pure precipitation.

A large number of concrete mixtures of varying quality with different binder types/combinations, varying water/binder ratios and air contents were produced and placed at the field exposure sites. The frost damage has been regularly evaluated by measurements of the volume change of the specimens and the change in ultrasonic transmission time through each specimen. This paper presents results after ten winter seasons for concrete qualities produced with six different binder combinations: one with a CEM I (Ordinary Portland Cement), one with a CEM II/A-LL, one with a CEM II/A-S, one with a CEM III cement type and two with CEM I and secondary cementitious materials (silica and slag).

2. MATERIALS AND SPECIMENS

The binder types/combinations studied in this investigation are shown in Table 1. For chemical composition, see [5].

Binder type/combination	Comments
CEM I ¹⁾	Low alkali, sulphur-resistant
CEM $I^{1)}$ + 5 % silica by binder weight	Silica in the form of slurry
CEM II/A-LL	Cement with 15 % limestone filler
CEM II/A-S	Finnish cement with ~15 % slag
CEM I^{11} + 30 % slag by binder weight	Ground blast furnace slag added in the mixer
CEM III/B	Dutch slag cement, ~70 % slag

Table 1 - Binder types/combinations investigated

¹⁾ CEM I = Degerhamn Standard [6] is a low-alkali, sulphur-resistant cement

Ten different concrete qualities were produced for each of the binder types/combinations. Concrete qualities with five different water/binder (w/b) ratios (0.30, 0.35, 0.40, 0.50, 0.75), and with and without entrained air, were produced for all binder combinations. 0-8 mm natural and 8-16 mm crushed aggregate was used for all mixes. A naphthalene-based plasticizer, Melcrete, was used for mixtures with w/b-ratio of 0.40 and lower. The air-entraining agent used, L16, is a tall oil derivative. A summary of concrete constituents and properties is presented in Table 2 below: for a complete presentation, see [5].

Binder type	w/b-	Equiv.	Cement	SCM ⁽²)	aea	Air content	Slump	Com	pressive	Scaling	$(kg/m^2)^{(6)}$
	ratio	w/c-	(kg/m^3)	(kg/m ³		fresh (%)	(mm)	streng	th (MPa)	28	56
		ratio ⁽¹)				SS ⁽⁴	Recalc ⁽⁵		
CEM I	0.30	0.30	500	-	Yes	4.8	240	95	87	0.02	0.04
	0.35	0.35	450	-	Yes	4.8	190	95	87	0.05	0.09
	0.40	0.40	420	-	Yes	4.6	125	67	60	0.01	0.02
	0.50	0.50	370	-	Yes	4.6	90	49	44	0.02	0.02
	0.75	0.75	260	-	Yes	4.7	100	21	18	0.13	0.14
	0.30	0.30	500	-	No	1.1	120	102	93	0.16	0.26
	0.35	0.35	450	-	No	1.2	140	91	83	1.94	4.39
	0.40	0.40	420	-	No	0.8	130	87	79	3.11	7.92
	0.50	0.50	385	-	No	0.8	70	56	50	5.09	14.5
	0.75	0.75	265	-	No	0.9	60	31	27	4.34	>15
95% CEM I	0.30	0.29	475	25	Yes	4.6	100	103	94	0.04	0.12
+ 5% silica	0.35	0.33	427.5	22.5	Yes	4.5	90	91	83	0.02	0.04
	0.40	0.38	399	21	Yes	4.8	105	72	65	0.02	0.04
	0.50	0.48	361	19	Yes	4.6	70	57	51	0.02	0.03
	0.75	0.71	237.5	12.5	Yes	4.3	70	25	22	0.19	0.20
	0.30	0.29	475	25	No	1.1	125	121	111	0.12	0.20
	0.35	0.33	427.5	22.5	No	1.1	90	105	96	0.36	0.89
	0.40	0.38	399	21	No	0.5	100	84	76	1.67	3.25
	0.50	0.48	370.5	19.5	No	1.2	60	67	60	1.86	4.61
	0.75	0.71	256.5	13.5	No	0.3	75	35	31	3.45	6.58
CEM II/A-S	0.30	-	520	-	Yes	4,4	70	62	56	0.09	0.11
	0.35	-	450	-	Yes	4,5	100	62	56	1.40	2.23
	0.40	-	420	-	Yes	4,5	80	53	47	1.75	2.45
	0.50	-	380	-	Yes	4,8	100	39	34	0.15	0.16
	0.75	-	260	-	Yes	4,7	100	25	22	1.12	1.25
	0.30	-	540	-	No	2,2	30	69	62	0.32	0.42
	0.35	-	450	-	No	2,6	35	64	57	2.37	4.06
	0.40	-	420	-	No	2,0	90	58	52	3.93	6.71
	0.50	-	400	-	No	1,7	65	48	43	5.28	7.65
	0.75	-	275	-	No	0,5	100	30	26	TD	TD
CEM II/A-	0.30	0.30	520	-	Yes	4,4	110	89	81	0.03	0.05
LL	0.35	0.35	450	-	Yes	4,3	110	85	77	0.14	0.25
	0.40	0.40	420	-	Yes	4,6	90	72	65	0.11	0.12
	0.50	0.50	390	-	Yes	4,8	70	52	46	0.17	0.20
	0.75	0.75	260	-	Yes	4,7	70	34	30	2.92	5.26
	0.30	0.30	530	-	No	2,3	100	95	87	0.16	0.28
	0.35	0.35	470	-	No	2,4	115	86	78	0.52	1.05
	0.40	0.40	420	-	No	2,4	110	76	69	3.04	5.67
	0.50	0.50	400	-	No	1,8	70	64	57	5.05	11.6
	0.75	0.75	280	-	No	1,2	75	36	32	TD	TD
						7	-	-			
					~~~~~~~~~~~~~~~~~~~~~~~~~~~~~~~~~~~~~~~						<u> </u>
Binder type	w/b-	Equiv.	Cement	SCM ⁽² )	aea	Air content	Slump	Com	pressive	Scaling	$(kg/m^2)^{(0)}$
	ratio	w/c-	(kg/m°)	(kg/m³		fresh (%)	(mm)	streng	th (MPa)	28	56
		ratio		)				SS ⁽⁴	Recalc		

Table 2 - Concrete constituents and properties.

70% CEM I	0.30	0.34	350	150	Yes	4.8	230	90	82	0.03	0.05
+ 30% slag	0.35	0.40	315	135	Yes	4.8	130	86	78	0.09	0.14
-	0.40	0.45	294	126	Yes	4.4	110	65	58	0.04	0.07
	0.50	0.57	259	111	Yes	4.8	80	49	44	0.02	0.03
	0.75	0.85	175	75	Yes	4.4	100	20	17	0.49	0.54
	0.30	0.34	350	150	No	0.7	220	101	92	0.10	0.13
	0.35	0.40	315	135	No	1.1	140	91	83	1.78	3.61
	0.40	0.45	294	126	No	0.9	120	78	71	1.78	3.83
	0.50	0.57	273	117	No	1.3	80	52	46	0.86	1.94
	0.75	0.85	185.5	79.5	No	0.5	80	25	22	1.58	4.37
CEM III	0.30	0.30	520	-	Yes	4.8	200	78	71	0.25	0.36
(~70% slag)	0.35	0.35	460	-	Yes	4.7	200	74	67	0.43	0.62
	0.40	0.40	420	-	Yes	4.3	120	61	55	0.57	0.85
	0.50	0.50	380	-	Yes	4.5	70	46	41	0.99	1.66
	0.75	0.75	255	-	Yes	4.4	90	26	23	2.05	3.16
	0.30	0.30	520	-	No	0.8	200	99	90	0.22	0.28
	0.35	0.35	470	-	No	0.7	200	80	72	0.49	0.65
	0.40	0.40	420	-	No	0.9	125	68	61	0.84	1.14
	0.50	0.50	400	-	No	1.0	65	54	48	1.21	1.61
	0.75	0.75	265	-	No	0.1	100	31	27	3.94	6.89

⁽¹ Equiv. w/c-ratio=water/(cement + 2*silica + 0.6*slag). Not applicable for CEM II/A-S, CEM II/A-LL and CEM III.

⁽² SCM – Secondary Cementitious Materials

⁽³ AEA – Air Entraining Agent

⁽⁴ Dry stored cubes tested in accordance with SS 13 72 10 [7] at the age of 28 days

⁽⁵ Recalculated to wet stored cubes in accordance with  $f_{wet,cube} = 0.76*(f_{dry,cube})^{1.04}$ 

⁽⁶ In accordance with the 'Slab test', SS 13 72 44 [8], freeze/thaw started at the age of 31 days

TD = Totally disintegrated

All concrete batches were produced in the autumn of 1996, and a number of 150 mm cubes were cast from each batch. The cubes were demoulded 24 hours after casting, and stored in lime-saturated water for six days. They were then stored in a climate chamber (50 % RH / 20 °C) for a period of between one and a half and three months. Between eight and twelve days before the specimens were placed at the field test sites, the cubes were cut, resulting in two specimens with the shape of a half 150 mm cube with one cut surface and the rest mould surfaces. After cutting, the specimens were stored in a climate chamber (50 % RH / 20 °C) until placed at the test site. During this second conditioning period, the volume of, and transmission time through, each specimen were measured. Two specimens of each mixture were then placed at each test site.

At the highway environment test site, the specimens were placed in steel frames close to the road, so that they were splashed by the passing traffic. At the marine test site, the specimens were mounted on top of a pontoon, thus exposing them to the saline marine environment but with no direct contact with the sea water, except when splashed over them by storms. The specimens at the test site without salt exposure were placed on top of loading pallets: here, they were exposed only to water from precipitation. At all sites, the specimens were exposed with the cut surface turned upwards.

### 3. TEST PROCEDURES

In order to be able to detect both internal and external frost damage, the change in volume and ultrasonic pulse transmission time was measured regularly. The first measurement was carried

out before placing the specimens at the test sites. The specimens at the highway site have subsequently been measured once a year during the first years of exposure and thereafter every second year. The specimens at the other two sites were measured after every second to third year.

The volumes of the specimens are calculated from results obtained from measuring the weight of the specimens in water and in air respectively. The ultrasonic pulse transmission time through the specimen (150 mm) is measured as a mean of three measurement positions, where possible, on each specimen.

The following laboratory tests were carried out on each concrete mix in order to determine the concrete characteristics (results are given partly in Table 2 above, and fully in Reference 5):

Testing the fresh concrete:

Remoulding test

- Testing the hardened concrete:
- Air content

- Density •
- Compressive strength in accordance with SS 13 72 10 [7]. Salt/frost resistance in accordance with SS 13 72 44 [8]. •

Slump •

•

Microscopical determination of the air void system, in • principal in accordance with ASTM C 457 [9].

The carbonation depth was measured on specimens exposed for eleven years at the test site without salt exposure. Carbonation was measured in accordance with SS 13 72 42 [10]. From the half cubes exposed at the test site, 40 mm thick slabs were cracked perpendicular to the cut surface, and phenolphthalein (3 % solution in ethanol) was sprayed on the cracked surface. The cracked surface was photographed, and the change in colour was measured at ten positions along both on the upper surface and the lower parts of each specimen.

#### 4. RESULTS

Figures 1-6 presents results from measurements of the volume change (%) after ten years of exposure at the highway and marine test sites, and after eleven years at the test site without salt exposure. The reference value is the initial volume before exposure. Figures 1-3 show the results for concrete produced without entrained air, exposed at the three exposure sites, while Figures 4 - 6 shows the results for concrete with entrained air (4-5 %). Each point is a mean value of measurements on two specimens.

Tables 3-5 show the change in transmission time (%)after ten years' exposure at the highway and marine test sites, and after eleven years at the test site without salt exposure. The reference value is that of measurements before exposure. Each point is a mean value of up to three measurements on each two test specimens, i.e. a mean value of up to six measurements. For some qualities with w/b-ratio 0.75, no value is presented: this is because damage to the concrete surfaces was so severe that measurements were not possible.

Table 6 shows the carbonation depth in millimetres measured on the top and bottom surfaces.



*Figure 1 - Volume change after ten winter seasons at the highway exposure site (Highway 40). Concrete with different binder combinations and water/binder ratios. No entrained air.* 



Figure 2 - Volume change after ten winter seasons at the marine exposure site (Träslövsläge harbour). Concrete with different binder combinations and water/binder ratios. No entrained air.



Figure 3 - Volume change after eleven winter seasons at the no-salt exposure site (SP in Borås). Concrete with different binder combinations and water/binder ratios. No entrained air.



Figure 4 - Volume change after ten winter seasons at the highway exposure site (Highway 40). Concrete with different binder combinations and water/binder ratios. With entrained air (4-5 %).



Figure 5 - Volume change after ten winter seasons at the marine exposure site (Träslövsläge harbour). Concrete with different binder combinations and water/binder ratios. With entrained air (4-5 %).



Figure 6 - Volume change after eleven winter seasons at the no-salt exposure site (SP in Borås). Concrete with different binder combinations and water/binder ratios. With entrained air (4-5 %).

	55				
Concrete mix \ w/b-ratio	0.30	0.35	0.40	0.50	0.75
CEM I, air	-4.9	-3.4	-3.9	-3.8	-7.9
CEM I, no air	-5.4	-4.0	-1.6	-2.0	9.6
CEM I + 5% silica, air	-0.7	-2.7	1.0	-0.4	-9.6
CEM I + 5% silica, no air	-2.2	-2.2	8.2	31.4	30.7
CEM II/A-LL, air	-5.5	-6.3	-5.6	-4.9	-3.4
CEM II/A-LL, no air	0.0	-6.4	-5.0	-3.8	3.3
CEM II/A-S, air	-7.9	-5.9	-6.1	-2.5	-9.8
CEM II/A-S, no air	-6.8	-6.6	-5.4	-3.2	2.7
CEM I + 30% slag, air	-3.4	-5.2	-4.4	-7.1	-17.6
CEM I + 30% slag, no air	-5.5	-1.4	-1.4	-3.9	-14.4
CEM III/B, air	-2.5	-2.0	-0.2	-1.4	-
CEM III/B, no air	-1.6	-2.9	-2.2	-3.2	-

Table 3. Change in transmission time (%) after ten years exposure at the highway exposure site (Highway 40). Concrete with different binder combinations and water/binder ratios.

Table 4. Change in transmission time (%) after ten years' exposure at the marine exposure site (Träslövsläge harbour). Concrete with different binder combinations and water/binder ratios.

	0.20	0.25	0.40	0.50	0.75
Concrete mix \ w/b-ratio	0.30	0.55	0.40	0.50	0.75
CEM I, air	-4.0	-4.0	-4.9	-2.4	-8.7
CEM I, no air	-4.6	-4.4	-5.0	-5.8	101
CEM I + 5% silica, air	-4.4	-4.0	-3.9	-5.4	-12.9
CEM I + 5% silica, no air	-3.2	-3.7	1.3	13.4	254
CEM II/A-LL, air	-4.9	-6.9	-9.8	-4.2	-5.8
CEM II/A-LL, no air	-4.8	-5.4	-4.9	-2.8	335
CEM II/A-S, air	-7.6	-7.0	-8.2	-3.7	-6.6
CEM II/A-S, no air	-7.7	-8.4	-6.5	-5.5	263
CEM I + 30% slag, air	-4.8	-5.6	-5.2	-8.3	-15.7
CEM I + 30% slag, no air	-6.1	-4.9	-4.9	-8.3	-16.3
CEM III/B, air	-6.5	-2.9	-3.6	-5.1	-
CEM III/B, no air	-4.1	-3.5	-1.3	-3.3	-

Table 5. Change in transmission time (%) after eleven years' exposure at the no-salt exposure site (SP in Borås). Concrete with different binder combinations and water/binder ratios.

Concrete mix \ w/b-ratio	0.30	0.35	0.40	0.50	0.75
CEM I, air	-4.0	-3.0	-4.2	-1.4	-7.5
CEM I, no air	-4.0	-4.0	-2.3	-1.7	0.8
CEM I + 5% silica, air	-2.1	-3.2	-1.7	0.0	-10.0
CEM I + 5% silica, no air	-1.7	0.4	1.7	7.5	26.8
CEM II/A-LL, air	-4.7	-5.5	-3.4	-3.1	-4.8
CEM II/A-LL, no air	-4.3	-3.4	-2.8	-1.5	2.1
CEM II/A-S, air	-5.5	-5.1	-2.3	-0.8	-5.0
CEM II/A-S, no air	-4.3	-4.7	-0.8	-0.9	8.7
CEM I + 30% slag, air	-1.1	-2.9	-2.7	-3.1	-13.6
CEM I + 30% slag, no air	-4.8	-3.0	-3.7	-4.9	-12.3
CEM III/B, air	-4.8	-2.3	-2.4	-2.6	1.8
CEM III/B, no air	-2.4	-2.8	-1.7	-1.6	0.9

Concrete mix \ w/b-ratio	0.3	0.35	0.4	0.5	0.75
CEM I, upper	0	0	0.5	0.8	2.3
CEM I, lower	0	0	0.8	1.7	6.1
CEM I + 5% silica, upper	0	0	0.2	0.4	1.1
CEM I + 5% silica, lower	0	0	0.6	0.9	3.8
CEM II/A-LL, upper	0	0	0.2	0.8	2.1
CEM II/A-LL, lower	0	0	0.1	0.5	1.8
CEM II/A-S, upper	0	0	0.1	0.3	3.6
CEM II/A-S, lower	0	0	0.8	0.8	4.1
CEM I + 30% slag, upper	0	0	0.4	0.9	3.3
CEM I + 30% slag, lower	0	0	1.0	1.2	8.1
CEM III/B, upper	0	1.5	2.5	4.4	12.1
CEM III/B, lower	1.0	2.9	1.6	3.2	9.2

Table 6. Carbonation depth in millimetre after eleven years' exposure at the no-salt exposure site (SP in Borås). Concrete with different binder combinations and water/binder ratios.

# 5. DISCUSSION

#### 5.1 External frost damage

Figures 1, 2 and 3 show the external damage in the form of volume change after ten winter seasons (highway and marine exposure sites) and eleven winter seasons (exposure site without salt) for different concrete qualities, all without entrained air. Without comparing results from the different concrete qualities, it is clear that the climate has a marked influence on the amount of external frost damage, i.e. surface scaling. Concrete exposed in a saline highway environment, Figure 1, shows much more extensive surface scaling than does concrete exposed in a salt-free environment. Concrete exposed in the marine environment shows external damage, but in general less severe than that of concrete exposed in the highway environment, at least for qualities with w/b-ratios of 0.40 and over. These differences in resistance to surface scaling can probably be explained by differences in:

- **Temperature**. The temperature in the marine environment is milder and fluctuates less than the temperature at the highway environment or the salt-free exposure site. However, the air temperature at these two latter sites is about the same.
- **Moisture conditions**. The moisture conditions at the different exposure sites vary significantly. During the winter months, the specimens at the highway site are subjected to a very moist environment, being constantly splashed by passing traffic. The specimens at the SP test site, however, are exposed only to natural precipitation.
- Salt concentration. The salt concentration in the water splashed over the samples at the highway site is, at least occasionally, much higher than the salt concentration in the marine environment. At the SP test site there is no exposure to salt.

For concrete qualities with entrained air and with w/b-ratio 0.75, all qualities are more or less damaged after ten years' exposure at the highway test site: see Figure 4. The most severely damaged is concrete with CEM III/B as binder, less damaged is CEM I and CEM II/A-LL. All concrete qualities, except with CEM III as binder, with w/b-ratio 0.50 or lower and with entrained air, show only small volume changes. CEM III with w/b-ratio 0.50, however, shows

significant damage. At the marine exposure site, all concrete qualities with entrained air and a w/b-ratio of 0.50 or lower show only small amount of damage: see Figure 5. For qualities with w/b-ratio 0.75, concrete with CEM III/B as binder respectively with CEM I + 30 % slag as part of the binder show most damage. At the exposure site without salt exposure, all concrete qualities with entrained air show only limited damage after 10 years' exposure: see Figure 6.

For concrete without entrained air exposed at the highway exposure site, all concrete qualities with w/b-ratio 0.75 show severe scaling: see Figure 1. For concrete with w/b-ratio 0.5, the quality with CEM III/B as binder shows the highest scaling. For concrete with w/b-ratio 0.4 and below, the differences in scaling are relatively small. However, concrete quality CEM II/A-S and w/b-ratio 0.35 shows a much higher scaling than do other concrete qualities. The major part of this scaling occurred during the first winter, after which the scaling was very small. One explanation could be due to a problem with the compatibility between the cement and the plasticizer used, resulting in a poor air void structure. The plasticizer was used for qualities with w/b-ratio 0.40 and 0.35, the scaling is significantly higher than for the quality with w/b-ratio 0.50. The same tendency can be seen for concrete with entrained air and with w/b-ratio 0.35 in Figure 4. In addition, the results from testing in the laboratory indicate lower scaling resistance for concrete with w/b-ratios 0.35 and 0.40 compared to 0.50 (see Table 2), indicating a possible compatibility problem between the cement and plasticizer.

At the marine exposure site, all concrete qualities without entrained air and with w/b-ratio of 0.50 or below show only small volume change. For concrete with w/b-ratio 0.75, all qualities are damaged to a greater or lesser extent.

At the test site without salt exposure, all concrete qualities without entrained air, except with CEM III/B as binder, show only limited damage, even at w/b-ratio as high as 0.75. However, concrete with CEM III/B as binder and with w/b-ratio of 0.75 shows significant damage.

One significant difference between the concrete qualities is the increase in volume for some qualities without entrained air with CEM I + 5 % silica as binder and with a w/b-ratio over 0.35 (see Figures 1 and 2), and for one quality with CEM I and a w/b-ratio of 0.75 (see Figure 2). This increase is seen primarily at the highway and marine test sites. An increase in volume is probably caused by internal damage, i.e. micro-cracks. A limitation of the volume measurements procedure is that the measured volume is a net volume of both a negative and a positive element, in the respective forms of a volume loss due to surface scaling and a possible increase in volume due to internal cracking. Concrete qualities with an apparent volume loss might, therefore, also have a small increase in volume caused by internal cracking without this being observed. It is therefore important to complement volume measurements with other techniques in order to detect possible internal damage, e.g. ultrasonic pulse transmission time measurements.

In general, the volume changes for concrete with high water/binder ratios and entrained air are less than for concrete without entrained air (compare results in Figure 1 and Figure 4). However, this is not valid for concrete qualities with CEM III/B as the binder. The volume change for qualities with CEM III/B as the binder and with entrained air is of the same order as that for concrete without entrained air (w/b-ratios 0.50 and 0.75). For these qualities, entrained air does not seem to improve the scaling resistance. This behaviour is confirmed by freeze/thaw testing in the laboratory: see Table 2. For concrete with CEM III/B as the binder, the air-entrained

qualities show damage of the same order as for concrete without air. For concrete with other binder types in this investigation, a significant improvement in scaling resistance is seen, as expected, for qualities with entrained air compared to qualities without entrained air.

#### **5.2 Internal frost damage**

Tables 3, 4 and 5 show the change in transmission time in percent after ten years' exposure at the highway and marine exposure sites, and after eleven years' exposure at the test site without salt exposure. A negative value (decrease in transmission time through the specimen with age) is expected for sound, undamaged materials due to the densification of the paste as a result of continued hydration. A positive value indicates possible internal damage. The results presented in Tables 3-5 show that a number of concrete qualities probably have internal damage, and that there does not seem to be any significant difference between the different exposure climates. The results show that most concrete qualities without entrained air and with a w/b-ratio of 0.75 show indications of internal damage. For concrete with CEM III/B as part of the binder, the surfaces of the specimens exposed at the highway and marine exposure sites were too severely damaged to be able to measure ultrasonic transmission time. At the no-salt exposure site, ultrasonic transmission time could be measured on CEM III/B specimens, and the result indicates a slight increase in transmission time through the specimen. This is not necessarily an indication of internal frost damage, but can be a result of a relatively deep carbonation zone in these specimens possibly resulting in a increase in transmission time. CEM I + 30 % slag as part of the binder shows no indication of internal frost damage, not even at high w/b-ratio without air.

Combining the results from volume measurements with the transmission time measurements gives clear indications of internal damage for the concrete qualities with CEM I + 5 % silica as binder and without entrained air, also for concrete with w/b-ratios down to 0.40. Both an increase in volume and an increase in transmission time clearly indicate internal damage, probably micro-cracking. The surfaces of the qualities that show increased transmission time but no detectable increase in volume are too severely damaged to permit the detection of an increase in volume due to internal cracking. Microscopic techniques, such as analysis of polished sections or thin sections, could be used for finding further evidence of internal damage. This was carried out and the results were confirmed for some qualities after five years' exposure at the exposure sites. The results are presented in [11] Utgenannt & Petersson 2001.

From the results described above, it can be noted that concrete with CEM I or CEM I + 5 % silica, CEM II/A-LL, CEM II A/S and CEM I + 30 % slag as part of the binder with entrained air and a water/binder ratio of 0.50 or lower, seems to be frost and salt/frost resistant in an aggressive highway environment, at least after ten years of exposure. However, concrete qualities containing large amounts of slag as binder seem to be less resistant to external salt/frost damage than concrete with the other binder types studied in this investigation. The results regarding concrete with high slag content are in agreement with those from an extensive field exposure investigation presented in [12, 13, 14]. In that investigation, a large number of concrete qualities with different amounts of slag in the binder were exposed to the marine climate at the Treat Island exposure site, Canada. Results from that investigation clearly show that salt/frost resistance decreases with increasing amounts of slag as part of the binder; a result that is confirmed by the present investigation. As in the present investigation, the investigation at Treat Island also showed the positive effect of low water/binder ratio on the salt/frost

resistance of concrete. The negative effects of high slag contents in the binder on the salt/frost resistance of concrete have been reported by several researchers: see [15, 16, 17, 18]. One probable explanation for this negative effect is the coarsening of the pore structure found in the carbonated skin of concrete containing slag [15, 17, 19, 21]. Another probable explanation, presented in [17], is the existence of metastable carbonates in the carbonated zone of concrete rich in slag.

# 5.3 Correlation between laboratory and field tests

When testing the durability in the laboratory, e.g. salt/frost resistance, results are wanted that are relevant to durability in the actual field conditions. In the present investigation, each concrete quality was tested in the laboratory at the prescribed age of 28 days in accordance with the Swedish Standard for salt/frost resistance, SS 13 72 44 (the 'Slab test') [8]. Comparing the laboratory results with results from the field exposure site at SP's site (no salt exposure) is not relevant, since salt/frost-resistant concretes are not prescribed for this climate. Concrete exposed at the marine and highway test sites is, however well suited for 'calibration' of the laboratory test method. Since the climate at the highway has proven to be most aggressive with regard to salt/frost resistance, the laboratory results are compared with results from this test site.

Figure 7 shows results from the laboratory tests and the volume change after ten years of highway exposure. The diagram shows the scaling  $(kg/m^2)$  after 56 freeze/thaw cycles as a function of the volume loss (%) after ten years' highway exposure. The acceptance criterion in the laboratory test is 1 kg/m² (illustrated by a horizontal line). An acceptance criterion of 2.5 volume % (shown by the vertical line) after ten years' exposure has been chosen for the field exposure specimens, corresponding to scaling of approximately 1.0 kg/m². Filled symbols represent concrete with entrained air. Symbols with a white centre represent concrete without entrained air.



Figure 7 - Scaling resistance (tested in the laboratory) as a function of the decrease in volume for specimens exposed in the highway environment for ten winter seasons. Filled symbols represent concrete with entrained air. Symbols with a white centre represent concrete without entrained air.

From the results presented in Figure 7, it can be seen that only three qualities fall into the IV quadrant, which is the worst case, and means that they are accepted by the test method, but fail in field exposure. These are air-entrained qualities, however, with high water/binder ratios (0.75). The standard test method is primarily intended to be used for bridge concrete, with entrained air and with a w/b-ratio below 0.5.

Most qualities falls into Quadrants II or III, which means that the test method and 'reality' correspond. Some concrete qualities fall into Quadrant I, which means that the test method rejects them. However, as the concrete in Quadrant I show only limited damage in the field, the test method results are on the safe side. Only two of these qualities have entrained air, both with binder type CEM II/A-S. All other qualities in Quadrant I are without entrained air, which makes them especially susceptible to frost damage. During the first ten years, the climate has not been aggressive enough significantly to damage these qualities. However, one winter season with a more aggressive climate might cause internal damage as well as scaling on these qualities without entrained air, moving them into Quadrant II. Two concrete qualities show an increase in volume and an increased transmission time, indicating internal frost damage. These qualities also fail the acceptance criterion when tested in the laboratory.

One explanation for the limited scaling in the field for the concrete qualities in Quadrant I might be a positive effect of ageing. In a field investigation reported in [20], it was found that concrete aged and exposed in a marine climate showed better scaling resistance when tested in the laboratory after ageing than did virgin concrete. This increase in scaling resistance was especially apparent for concrete without entrained air, as was also found in this investigation. One effect of ageing that markedly improves the scaling resistance for OPC concrete is carbonation: see [21].

On the whole, the results for concrete with w/b-ratios equal to or below 0.5, and with entrained air, shown in Figure 7, indicate that the slab test classifies these concrete qualities as could be expected. This is true for all binder combinations tested.

The results presented here are valid only for the materials, e.g. cement and binder types, used in this investigation. Other materials may give different results.

# 5.4 Carbonation

The results presented in Table 6 show that the carbonation is relatively limited after eleven years of exposure. All concrete qualities, except concrete with CEM III as the binder, show carbonation depths less than 2 mm for qualities with w/b-ratios of 0.50 or below. For qualities with w/b-ratio 0.75, all concrete qualities show larger carbonation depth. Only concrete with CEM III as binder has a carbonation depth of more than 2 mm for qualities down to w/b-ratio 0.35. Carbonation was measured on the upper as well as on the lower part. The results do not show any clear indication of whether the carbonation depth is greater on either of the parts. It could be expected that the lower part, which is sheltered from direct precipitation, would have a somewhat greater carbonation depth. However, the specimens were placed on wooden loading pallets, and the bottom surface in contact with wood ought to be as wet as the top surface, thus giving similar or even smaller carbonation depths on the lower part compared to the upper part.

# 6. CONCLUSIONS

The following conclusions can be drawn after ten years' exposure at the field exposure sites in a highway environment and a marine environment, and after eleven years' exposure in a salt-free environment:

- There are substantial differences in external frost damage, depending on the environment. The most extensive external frost damage is observed on concrete specimens exposed in the highway environment. Concrete exposed in the salt-free environment shows only small changes in volume after eleven winter seasons.
- For all concrete qualities with water/binder ratio of 0.5 or below, scaling takes place only when exposed to salt. Internal damage, however, is observed on concrete qualities exposed at all three test sites, even when no salt is present.
- Concrete with CEM I, CEM II/A-LL, CEM II/A-S, CEM I + 30 % slag and CEM I + 5 % silica as binder, with entrained air and a water/binder ratio of 0.5 or below, has good resistance to internal and external damage. Concrete with CEM III, however, suffers from severe scaling, even with w/b-ratio below 0.5 and with entrained air.
- Internal damage is observed only for concrete qualities without entrained air and, furthermore, in most cases for concrete qualities with high water/binder ratios. However, for concrete qualities with CEM I + 5 % silica as binder, internal damage is found at lower w/b-ratios, down to w/b 0.4.

- Comparing results from laboratory testing in accordance with SS 13 72 44 (the 'Slab test'), with results after ten years' exposure at the highway exposure site, shows that the laboratory standard classifies most concrete qualities correctly.
- Carbonation depth measured after eleven years' exposure at the test site without salt exposure show that all concrete qualities, except concrete with CEM III as binder, show carbonation depths less than 2 mm for qualities with w/b-ratio 0.50 or below. Only concrete with CEM III as the binder has a carbonation depth of more than 2 mm for qualities down to w/b-ratio 0.35.

# 7. ACKNOWLEDGEMENTS

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Chloride ingress and corrosion from the Swedish field exposures under the marine environment



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# ABSTRACT

This paper presents the results from several Swedish national research projects dealing with durability of marine concrete structures. In the beginning of 1990's, some 40 types of concrete specimens were exposed to seawater at the field station at the western coast of Sweden. Totally about 240 chloride profiles measured after 0.5, 1, 2, 5 and 10 years exposure and about 100 moisture profiles after 5 and 10 years exposure have been collected. These collected data supply unique opportunities for validation of the rapid test methods and the prediction models for chloride ingress and corrosion of reinforcement steel in concrete.

**Key words:** Concrete, chloride, corrosion, durability, field exposure, moisture.

#### 1. INTRODUCTION

Chloride induced reinforcement corrosion is one of the most important degradation processes in reinforced concrete structures exposed to marine environment and road environment where deicing salt is used in the winter. The degradation of reinforced concrete structures, especially infrastructures, has very important economical and social consequences due to the need for diverting resources for repairing damaged structures and sometimes the need to close the facility for carrying out the repair work. Owing to its long coastline and intensive application of deicing salt, the topic of chloride ingress in concrete has special significance in Sweden. In the beginning of 1990's, a Swedish national project called "BMB" – Durability of Marine Concrete Structures – was initiated [1]. As a part of work in the BMB project, some 40 types of concrete specimens were exposed to seawater at the Träslövsläge field site at the west coast of Sweden. The specimens were periodically sampled for chloride penetration profiles, which serve as the "first hand's" information about chloride ingress into concrete and are believed valuable for the examination of modelling for chloride penetration. The chloride ingress profiles up to five years exposure to seawater at the field site have been measured during the lifetime of the BMB project.

After the BMB project, many concrete slabs are remained at the field site for continuous exposure. Under the financial support of Swedish Road Administration, the field data after 10 years exposure were measured again. Together with the previously collected data, these serial field data supplied unique opportunity for evaluation of chloride ingress and verification of prediction models.

With the development of a new rapid technique RapiCor for corrosion measurement, the corrosion status of steel embedded in the concrete slabs exposed at the field site were investigated in 2005, after 13 years exposure, under the financial support of SBUF – Swedish Construction Industriy's Organisation for Research and Development.

This paper presents the some results from the above mentioned research projects.

# 2. CONCRETE SPECIMENS AND EXPOSURE CONDITIONS

# 2.1 Concrete slabs

The relevant mixture proportions of concrete are summarised in Table 1. The main variations include water-binder ratio (0.25, 0.3, 0.35, 0.4, 0.5, 0.6 to 0.75), binder type (four types of cement with different additions of silica fume and fly ash), and air content (6% entrained air and non-AEA). Concrete slabs of  $1000 \times 700 \times 100$  mm were cast in the end of 1991 and the beginning of 1992. After about two weeks' moist curing, the slabs were transported to the Träslövsläge field site and mounted on the sides of the pontoons for the exposure with the bottom side of the slab outward the seawater. The parallel slabs called "noll" (reference) specimens were used for measurement of chloride transport properties, as will be described later in section 3.3.

It should be noticed that the slurry silica fume was used in concrete H-series, while the powder one was used in the other types of concrete.

# 2.2 Exposure conditions at the Träslövsläge field site

An overview of the Träslövsläge field site is shown in Fig. 2.1. The chloride concentration in the seawater varies from 10 to 18 g Cl per litre, with an average value of about 14 g Cl per litre. The typical water temperature is illustrated in Fig. 2.2 and has an annual average +11 °C.
Mix No.	Binder type	Binder kg/m ³	Water- binder ratio ¹⁾	Fine aggreg. 0-8 mm kg/m ³	Coarse aggreg. 8-16 mm kg/m ³	Sp ²⁾ % of binder	AEA ³⁾ % of binder	Air content %	28d compr. Strength ⁴⁾ MPa
1-35	100% Anl ⁵⁾	450	0.35	839	839	1	0.041	6.0	70
1-40	100%Anl	420	0.40	873	806	0.8	0.03	6.2	58
Ö	100%Anl	430	0.38	813	840	1	0.04	6.2	58
1-50	100%Anl	370	0.50	876	808	-	0.033	6.4	41
1-75	100%Anl	240	0.75	1013	796	-	0.029	6.1	21
2-35	100% Slite ⁶⁾	450	0.35	801	868	1.7	0.038	5.7	60
2-40	100% Slite	420	0.40	871	804	1.3	0.029	6.2	54
2-50	100% Slite	390	0.50	853	787	-	0.026	5.8	42
2-60	100% Slite	310	0.60	936	797	-	0.022	6.3	35
2-75	100% Slite	250	0.75	999	785	-	0.02	5.8	26
3-35	95% Anl+ $5%$ SF ⁷⁾	450	0.35	801	868	1.2	0.08	5.8	72
3-40	95%Anl+5%SF	420	0.40	835	835	0.8	0.043	6.1	61
3-50	95%Anl+5%SF	370	0.50	840	840	-	0.04	6.0	45
3-75	95%Anl+5%SF	240	0.75	966	823	-	0.039	5.9	21
4-40	90%Anl+10%SF	420	0.40	803	870	1.17	0.043	6.6	65
5-40	95%Anl+5%SF	420	0.40	878	878	1.5	0.006	2.9	81
6-35	95% Anl+5% SF	450	0.35	858	929	1.5	-	2.1	93
6-40	95% Anl+5% SF	420	0.40	898	898	1.5	-	1.7	87
7-35	100%Anl	450	0.35	898	898	1.5	-	2.4	91
7-40	100%Anl	420	0.40	939	867	1	-	2.1	79
7-75	100%Anl	265	0.75	1044	821	-	-	1.1	32
8-35	100% Slite	470	0.35	847	918	1.8	-	2.1	73
8-40	100% Slite	440	0.40	882	882	1.5	-	2.1	67
8-50	100% Slite	410	0.50	893	924	-	-	1.4	56
8-60	100% Slite	330	0.60	977	833	-	-	1.6	45
8-75	100% Slite	270	0.75	1040	817	-	-	1.4	37
9-40	95%DK8)+5%SF	420	0.40	839	839	1.2	0.037	6.5	63
10-40	$78.5\% DK {+} 17\% FA^{9)} \\ {+} 4.5\% SF$	420	0.40	770	905	1.7	0.063	6.1	69
11-35	85%DK+10%FA+5%SF	450	0.35	781	917	2.33	0.04	5.7	84
12-35	85% Anl+10% FA+5% SF	450	0.35	781	917	1.87	0.055	6.4	73

Table 1 – Mixture proportions of concrete exposed at the Träslövsläge field site

(See the next page for the notes)

Fine Coarse Water-AEA 28d compr. Sp Air Binder Binder type Mix aggreg. aggreg. binder % of % of content strength kg/m³ 0-8 mm 8-16 mm No. binder ratio binder % MPa kg/m³ kg/m³ 9-40 95%DK⁸⁾+5%SF 420 0.40 839 839 0.037 6.5 1.263 78.5%DK+17%FA⁹⁾ 10-40 420 0.40 770 905 1.7 0.063 6.1 69 +4.5%SF 85%DK+10%FA+5%SF 781 917 5.7 11-35 450 0.35 2.33 0.04 84 85%Anl+10%FA+5%SF 450 0.35 781 917 73 12-35 1.87 0.055 6.4 H1 95%Anl+5%SF 500 0.30 836 942 2.3 0.8 112 _ H2 90% Anl+10% SF 500 0.30 820 963 2.1 1.1 117 H3 100%Anl 492 0.30 791 892 2.7 3.6 96 _ H4 95% Anl+5% SF 420 840 840 0.055 5.9 0.40 0.8 63 H5 95% Anl+5% SF 551 0.25 806 946 3 1.3 125 _ 95% Anl+5% FA H6 518 0.30 791 892 2.5 2.8 95 95%Deg400¹⁰⁾+5%SF H7 500 0.30 836 942 2.3 1.3 117 _ H8 80% Anl+20% FA 0.30 680 98 616 865 2.8 3.0 _ H9 100%Deg400 500 0.30 812 916 2.3 2.9 102 _

Table 1 (Continuation)

1) Assuming that the efficiency factor of silica fume is 1 and fly ash is 0.3.

2) Sp - Super-plasticizer. Cementa 92M

3) AEA - Air entraining agent. Cementa L14

4) According to SS 13 72 10

5) Anl – Anläggningscement (Swedish SRPC)

6) Slite - Slite cement (Swedish OPC)

7) SF – Silica fume (Elkem. Norway)

8) DK – Aalborg Lav cement (Danish SRPC)

9) FA - Fly ash (Aalborg . Denmark)

10) Deg400 – Degerhamn 400 cement (another type of Swedish SRPC)



Figure 1 – Overview of the Träslövsläge field site



In the sine function:  $T_{max} = 20$  °C,  $T_{min} = 2$  °C,  $t_0 = 119$  days 1994-12-30 is taken as the original point

Figure 2 – Annual temperature in the seawater

# **3. MEASUREMENTS**

## 3.1 Sampling

In the previous investigations up to 5 years' exposure, the concrete slabs were taken back to the laboratory. A 30~50 mm thick slice was cut away from one of the outmost sides of each slab to avoid the influence of two-dimensional penetration, and a prism of size  $1000 \times 100 \times 100$  mm was then successively cut as the sample specimen for different penetration zones, as shown in Figure 3. After cutting for the specimens, the slabs were sent back to the field site for continuous exposure. The cores were drilled in the exposure direction from different zones of each prism. In the investigation after 10 years' exposure, cores were directly drilled at the field site. Two cores, one for chloride profile and another for moisture profile, were taken in respective zones. The sampling positions were chosen in such a way that the least distance between the curved surface of a core and the outmost side of a slab is about 50 mm to avoid the influence of twodimensional penetration. From the previous investigations it has been known that the chloride profile from splash zone varies very much due to the unstable climate in this zone, but the profile is normally between those from atmospheric zone and submerged zone. To minimise the laboratory work in the 10 years' investigation, the cores were taken from the splash zoon only from three types of concrete (1-40, 2-40 and 3-40), which might be most commonly used in Sweden for infrastructures. Each core was sealed in double thick plastic bags when its surface was slightly dry, and then was transported to the laboratory for further profiling.



Figure 3 – Sampling illustration of a concrete slab after exposure

# 3.2 Measurement of chloride profiles

The cores individually sealed in double thick plastic bags were stored in the laboratory at the room temperature not longer than two weeks prior to sampling. Powder samples were then taken from each core by means of dry-grinding on a lathe with a diamond tool, successively from the exposed surface to a certain depth. The depth of each sample was measured from the lathe with an accuracy of 0.5 mm. After the grinding, the powder samples were immediately dried at 105 °C and then stored in a desiccator for later chloride and calcium analysis. The calcium analysis is for an estimation of binder content in the same sample for chloride content so as to be able to more precisely express the chloride content by mass % of binder.

The acid soluble chloride content in each sample was determined principally in accordance with AASHTO T260 using potentiometric titration on an automatic titrator with chloride selective electrode and the 0.01 N AgNO₃ solution. The soluble calcium in the same sample for chloride analysis was determined using the same potentiometric titrator but with calcium selective electrode and the 0.1 N EDTA solution. Form the calcium content the binder content in the sample can be estimated. A more detailed description of the method for determination of chloride and calcium contents can be found in [2]. Since similar techniques were employed in the precious investigations even though by different operators, the data from various investigations should be reliable and comparable.

# **3.3** Measurement of moisture profiles

The measurement of moisture profiles was carried out at Chalmers. The cores individually sealed in double thick plastic bags were stored in the laboratory at the room temperature not longer than a few days prior to sampling. A slice of about 10~20 mm thick was split from each concrete core, starting from one of the ends, with the help of a compression jack. A large piece of sample of about 10~30 g and a number of small pieces of sample were immediately taken,

using hammer and chisel, from the central portion of the freshly split slice. The large piece was immediately weighed and then placed in a box for measurement of degree of capillary saturation, while the small pieces were stored in a glass test tube for measurement of RH (Relative Humidity). The technique for measurement of RH has been well described by [3] and for degree of capillary saturation by Hedenblad and Nilsson [4]. After the above sampling, another slice was successively split and samples were taken. The above sampling process was repeated until all the samples were taken from each core.

# **3.4** Measurement of chloride migration coefficient

In the BMB project, a rapid test method for determination of chloride transport property in concrete was developed at the Chalmer, called the CTH method [5-6]. The test is based on the principle of ionic migration under an external electrical field. This external electrical field can greatly accelerate the transport process of chloride ions in concrete. This method has been used to measure  $D_{\text{CTH}}$  on all the types of "noll" concrete specimens and the detailed information about the test conditions can be found elsewhere [7]. Owing to its simplicity and rapidity, the CTH method is very useful for evaluating the resistance of concrete to chloride ingress and has been adopted as a Nordtest method NT BUILD 492 in 1999.

# **3.5** Measurement of corrosion

In the past years, a handheld instrument RapiCor was developed for rapid measurement of corrosion of steel in concrete, see Figure 4. The detailed descriptions of this measurement technique have been published elsewhere [8, 9]. The instrument measures half-cell corrosion potential, and then generates two galvanostatic pulses for corrosion rate and resistivity measurements. The measurement is quick and only needs a few seconds to obtain three parameters: corrosion potential, corrosion rate of steel and resistivity of concrete. These three parameters contribute a more accuracy estimation of the corrosion status of steel.



Figure 4 – Handheld instrument RapiCor (left) and it measurement principle (right)

Since there were more than 40 concrete slabs from the previous BMB project continuously exposed at the field site, to make a rough estimation of the corrosion status the corrosion measurement was, as the first step, carried out with the slabs kept in place, that is, a half of the slab was exposed under the seawater and another half to the air, as shown in Figure 5 (left). From this rough estimation, taking into account both the roughly measured corrosion risk index and the type of concrete, five slabs (see Table 2) were taken to the laboratory for more detailed investigation including non-destructive corrosion measurement using RapiCor at the positions as shown in Figure 5 (right), destructive examination by releasing steel bars from the concrete, and determination of chloride contents in concrete at the cover depth along the steel bars. Based on the visual examination of the steel bars from the five selected slabs, it has been verified that the non-destructive RapiCor technique gives satisfactory estimation of corrosion [10, 11]. Further corrosion measurement using this technique was, therefore, carried out on the rest of concrete slabs exposed at the Träslövsläge field site. In this case, the slabs were lifted up and the measurement was carried out at the positions as shown in Figure 5 (right).

Mix No.	Binder type	Binder kg/m ³	Water- binder ratio	Air content %	28d compr. strength MPa	Rebar diam. mm	Cover mm Bar 1/2	Cl % of binder ²⁾ at cover depth Bar 1/2	Corrosion risk index ³⁾ Bar 1/2
1-352 ¹⁾	100% Anl	450	0.35	6.0	70	20	20/15	2/2.4	4/4
4-40	90%Anl+10%SF	420	0.40	6.6	65	12	15/15	1.6/1.6	4/2
7-40	100% Anl	420	0.40	2.1	79	12	10/15	2.6/2.1	3/2
10-40	78.5%DK+17%FA + 4.5%SF	420	0.40	6.1	69	12	15/10	2.5/3.3	3/4
H8	80% Anl+20% FA	616	0.30	3.0	98	12	15/10	1.7/2.3	4/3

Table 2 – Concrete slabs taken from Träslövsläge for the laboratory investigation

1) The last number "2" denotes the second batch in concrete casting.

2) Data quoted/interpolated from the investigation after 10 years' exposure [12].

3) According to the preliminary field investigation, see Appendix 1.



Figure 5 – Steel bars in concrete slab and positions for corrosion measurement using the handheld instrument RapiCor (left: in place, right: in the laboratory or lifted up)

# 4. **RESULTS AND DISCUSSIONS**

# 4.1 Chloride and moisture profiles

The collected data of chloride profiles after exposure for 0.5, 1, 2, 5 and 10 years and moisture profiles after exposure for 5 and 10 years are compiled in [12]. In this paper some of the results will be presented.

## Effect of exposure zones

The chloride profiles as well as the binder content in the corresponded concrete sample with three typical types of binder and water-binder ratio are summarised in Figure 6. It should be noticed that, if not otherwise stated, the profile data are hereafter referred to 10 years exposure. It can be seen that the chloride ingress in submerged zone is in general the severest among all the three exposure zones, while the chloride ingress in splash zone may be similar to or less than that in submerged zone. This is in agreement with the results from the previous investigations after 5 years exposure [13], where it was shown that chloride ingress in various types of concrete follows the order of submerged > splash > atmospheric zone. From the moisture profiles as shown in Figures 7 it can be seen that the concrete exposed in submerged zone shows highest moisture content, which provides paths for chloride ions to diffuse into concrete. It is interesting to see that the moisture profiles in splash zone and atmospheric zone are not far different, but the chloride ingress in splash zone is remarkably severer than in atmospheric zone. This can be explained by side diffusion and capillary suction.

It can also be seen from Figure 6 (left) that chloride ingress from the backside (corresponding to the topside in casting) of the slab is in general severer than that from the front side (corresponding to the bottom side in casting), even though the moisture profiles do not show such a tendency. Apparently, this is attributed to segregation, as indicated in Figure 6 (right), where the backside of slabs contains more binder than the front side. This segregation makes the concrete in the topside more permeable than in the bottom side.

The measured degree of capillary saturation as shown in Figure 7 (right) is sometimes larger than 1, implying that these specimens lost weight when put in capillary contact water in the laboratory after having been exposed in seawater for ten years at a small depth. Most probably, it is the air voids that were partly water filled after the exposure, but were emptied when the specimens were taken out of the seawater. Further study is needed to clarify this phenomenon.

When comparing the 10 years profiles with the 5 years profiles as shown in Figures 8 and 9, it can be observed that, in the splash zone, even though the moisture contents (see Figure 9) did not significantly increase, the chloride ingress (see Figure 8) in this zone significantly increased in the past 5 years, especially in the concrete with Anl cement. This could imply that the chloride ingress in the splash zone may gradually exceed the one in the submerged zone after a long-term exposure. According to Tang and Sandberg [14], the chloride penetration profiles in the splash zone is, however, very sensitive to the position where the core is taken. Little difference in the coring position may result in large difference in chloride profile. Further observations from a longer exposure period are needed to verify the above phenomenon.



*Figure* 6 – *Profiles of chloride ingress in concrete under various exposure zones (left) and the binder content in the corresponded concrete sample (right)* 



Figure 7 – Moisture profiles (left: relative humidity and right: degree of capillary saturation) in concrete under various exposure zones after 10 years exposure.



Figure 8 – Comparison between chloride profiles after 5 and 10 years exposure.



Figure 9 – Comparison between moisture profiles after 5 and 10 years exposure.

# Effect of water-binder ratios

Since the chloride ingress in submerged zone is severest as shown in the previous section, the discussions hereafter will be limited to the submerged zone. The chloride profiles in concrete with different water-binder ratios are summarised in Figure 10. As expected, lower water-binder ratio results in less chloride ingress, especially in the concrete with Anl cement. There exists a limitation: when water-binder ratio is very low, e.g. 0.25, it may not help to reduced chloride ingress, as shown in Figure 10.

The moisture profiles in concrete with different water-binder ratios are summarised in Figure 11. Also as expected, low water-binder ratio in general results in less moisture inside the concrete. Addition of silica fume in concrete significantly reduced relative humidity and degree of capillary saturation in concrete. Although it seems difficult to find a clear relationship between chloride and moisture profiles, less chloride ingress is in general related to a lower moisture profile. Clearly, water is the medium for chloride to ingress in concrete.



Figure 10 – Chloride profiles in concrete with different water-binder ratios



Figure 11 – Moisture profiles in concrete with different water-binder ratios

### Effect of entrained air

The chloride profiles in concrete with and without addition of AEA (Air Entraining Agent) are summarised in Figure 12 and the corresponded moisture profiles are shown in Figure 13. It seems that the 6% entrained air tends to increase chloride ingress. The least chloride ingress in the concrete with 3% entrained air (Figure 12, right side) might be attributed to a better compaction under such air content, but the conclusion cannot be drawn from this single profile.

The relative humidity in the concrete without entrained air is generally lower than that with entrained air, especially in the deeper portion of the concrete, where there are less chloride ions. This is probably due to the reason that part of the air pores was filled with water, resulting a higher total water content (capillary + part of air pores).



Figure 12 – Chloride profiles in concrete with and without entrained air



Figure 13 – Moisture profiles in concrete with and without entrained air

# Effect of binder type

The chloride profiles in concrete with various types of binder are summarised in Figure 14 and the corresponded moisture profiles are shown in Figure 15. It can be seen from Figure 13 that Slite cement reveals lower chloride profile than Anl cement, but the significance becomes

negligible when water-binder ratio is reduced to 0.35. There is no significant difference in moisture profiles between Slite cement and Anl cement.

No significant difference in chloride profile between Swedish (Anl) cement and Danish (DK) structural cement could be found (Figure 14), even though there exists some differences in their moisture profiles (Figure 15).

Addition of silica fume and fly ash in Swedish (Anl) cement and Danish (DK) structural cement effectively increases the resistance to chloride ingress (Figure 14, right side), but not in Deg400 cement, which already reveals a better resistance to chloride ingress when compared with pure Anl cement (Figure 14, upper left). It seems that addition of fly ash by 5% did not significantly improve the resistance, while 20% fly ash can remarkably increase the resistance (Figure 14, upper left).

Again, addition of silica fume or fly ash in concrete resulted in lower moisture profiles, to a great extent due to finer pore structures contributed by the secondary hydration of the pozzolanic materials.



Figure 14 – Chloride profiles in concrete with different binders and water-binder ratios



Figure 15 – Moisture profiles in concrete with different binders and water-binder ratios

## 4.2 Relationships between migration coefficient and apparent diffusion coefficient

By curve-fitting the data of a measured chloride profile to the error-function solution to Fick's second law, the apparent diffusion coefficient can be obtained. It should be noted that Fick's law is a very simple model which is in many cases not suitable for chloride ingress in concrete. The curve-fitted apparent diffusion coefficient should, therefore, be regarded as a regression coefficient rather than a diffusion coefficient. Nevertheless it is valuable to compare this regression coefficient from the field exposure with the chloride migration coefficient from the laboratory, as shown in Figure 16.



Figure 16 – Comparison between migration coefficient  $D_{CTH}$  measured at about 6 months age and  $D_{F2}$  curve-fitted from the profiles after different exposure periods in submerged zone.

It can be seen that there exist fairly good linear relationships in logarithmical scale between  $D_{F2}$  and  $D_{CTH}$ . The variation in regression coefficients with different exposure periods is probably due to the fact that the curve-fitted  $D_{F2}$  varies with exposure time. Since the parameters  $D_{F2}$  and  $D_{CTH}$  involve both the material property and the exposure condition, it is not expected to compare their absolute values, but the sensitivity to ranking the concrete with regard to chloride ingress. From the above figure and regression results it is easy to know that  $\Delta D_{CTH}/D_{CTH}$  is always larger than  $\Delta D_{F2}/D_{F2}$ , implying that the CTH method is really a sensitive tool for ranking concrete's resistance to chloride ingress. When  $D_{CTH}$  is less than  $5 \times 10^{-12} \text{ m}^2/\text{s}$ , the value of  $D_{F2}$  after 10 years exposure in seawater is in most cases less than  $1 \times 10^{-12} \text{ m}^2/\text{s}$ . Comparing Figs. 4.19 to 4.21 with the  $D_{CTH}$  values in Appendix 3 it can be found that the lowest  $D_{CTH}$  value in the *w/c* 0.4 group is concrete 10-40, in the *w/c* 0.35 group is concrete 11-35, and in the *w/c* 0.3 group is concrete H2. All these three types of concrete reveal the lowest chloride ingress in their group, as shown in Figs. 4.19 to 4.21. This indicates that the CTH method is indeed measuring the resistance of concrete to chloride ingress and is applicable to various types of concrete including silica fume and fly ash.

# 4.3 Corrosion of steel in concrete

As can be seen in Figure 5, the lower end of steel bar was completely embedded in the concrete slab. When casting the concrete, a piece of mortar was used as distance spacer to support the steel bar. It has been found from the corrosion measurement that, on almost a half of the concrete slabs exposed at the Träslövsläge field site, the maximum corrosion rate appeared at the lower part of the slab, near the interface between concrete and distance spacer, indicating a poor interface, as shown in Figure 16, Rebar 1. Otherwise the maximum corrosion rate often appeared at the position about 10-30 cm under the water level, where the chloride ingress is severest (see Figure 6) and oxygen is sufficient, as shown in Figure 16, Rebar 2.

It is worthy to point out that the half cell potential can give information about something happened on the steel but cannot tell exactly what has happened. For instance, the potential on the stainless steel without any corrosion became also very negative. The resistivity only gives information about the moisture or ionic content in concrete. It is the corrosion rate which supplies the real information about corrosion status of steel in concrete.

The values of maximum corrosion rate on each steel bar in concrete are summarised in Figure 17. It seems not clear to find the effect of water-binder ratio and type of binder on corrosion rate, due to the fact that the poor interface between concrete and distance spacer resulted in early corrosion. On the other hand, the corrosion rate was found relatively low when cover thickness is larger than 30 mm. Therefore, as general conclusions, thicker cover is an effective way to protect the steel from corrosion.



Figure 16 – Left: Measurement values of half-cell potential (upper), corrosion rate (middle) and resistivity of concrete (lower). Right: Example of maximum corrosion rate at the lower end of steel bar due to poor interface between concrete and distance spacer (Rebar 1) and at about 20 cm under the water level (Rebar 2).



*Figure 17 – Summary of corrosion rate in concrete with different binder (upper) and water-binder ratios (lower)* 

# **CONCLUDING REMARKS**

The marine exposure data collected up to 10 years provide very important first hand information for improving knowledge and understanding of chloride transport in concrete. These data are very valuable for validation of the laboratory test methods and verification of prediction models.

The results from 10 years exposure in marine environment show that

- Chloride ingress in submerged zone is the severest, while in atmospheric zone is the mildest.
- Chloride ingress in splash zone may be similar to or less than that in submerged zone due to side diffusion and absorption. Although there is a tendency that chloride ingress in splash zone may exceed that in submerged zone after a long-term exposure, further investigations are needed to verify this phenomenon.
- Chloride ingress significantly decreases with water-binder ratio, especially for the concrete with SRPC.
- Addition of silica fume and fly ash increases the resistance of concrete to chloride ingress, especially when both silica fume and fly ash are added in concrete.
- High volume (6%) air entraining may increase chloride ingress to some extent, although it is necessary to assure a good resistance to frost attack.

From up to 10 years in-field data it seems that the curve-fitted diffusion coefficient  $D_{F2}$  decreased with the exposure time only in the first five years period, and afterwards this decrease tendency becomes unclear. On the other hand, the surface chloride content  $C_s$  increased in the first 5 years and then kept more or less unchanged. This increased  $C_s$  may mislead a decreased  $D_{F2}$ , because the assumption of constant  $C_s$  in the curve-fitting equations does not hold. Therefore, the hypothesis of a constantly decreasing  $D_{F2}$  in some empiric models for service life prediction is very questionable.

The rapid migration test, or called the CTH method (NT BUILD 492), is a sensitive tool for ranking the resistance of concrete to chloride ingress. The method has been proven applicable to various types of concrete including those with silica fume and fly ash. There exist reasonably good linear relationships in logarithmic scale between  $D_{\text{CTH}}$  and  $D_{\text{F2}}$ . The lowest  $D_{\text{CTH}}$  value well corresponds to the lowest chloride ingress in the group of concrete with similar water-binder ratios.

Based on the results from the corrosion measurement, it has been found that the visible corrosion (excluding that due to poor interface between concrete and distance spacer) normally occurred about 10-20 cm under the seawater level, where the oxygen may be sufficiently available for initiating the corrosion. Chloride may easily penetrate through a poor interface between concrete and mortar spacer and, as a consequence, initiate an early corrosion.

According to the findings from the field investigations, the following suggestions can be proposed:

- Reducing water-binder ratio is an effective way to improve the resistance of concrete to chloride ingress, but when w/b is less than 0.3, the effect becomes not significant.
- An alternative way to improve the resistance of concrete to chloride ingress is adding silica fume by 5~10% or fly ash by 20%, or more effectively, a combination of 5% silica fume and 10~20% fly ash. Adding a high quantity of fly ash or other types of additive materials may also improve the chloride resistance, but more experimental work is needed to verify their effect.
- The CTH method is a useful tool for a quick evaluation of the resistance of concrete to chloride ingress. The test results can be used as input data for prediction of chloride ingress, and further for prediction of service life of reinforced concrete structures.

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# AALBORG PORTLAND's durability project – 25 years judgement.



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# ABSTRACT

Information from 25 years exposure of 16 different types of concrete is presented in this paper. Three types of cement as well as secondary cementitious materials as fly ash and silica fume have been used.

The results demonstrate clearly, that fly ash and silica fume can improve the durability and the mechanical properties of concrete.

The data serves as a basic information for judgement and modelling equivalent behaviour of new types of concrete mixes, utilising new cements and new types of secondary cementitious materials.

**Key words**: Mineral additives, Flyash, silica fume, seawater, outdoor exposure, durability, mechanical properties

# **1 INTRODUCTION**

Aalborg Portland initiated in 1983 a project to document the durability of concrete containing secondary cementitious materials as fly ash and silica fume. Mechanical properties have also been evaluated.

Three different qualities of concrete are included in the project. The quality is characterised by the cement content, which is 260 (w/c: 0.52 - 0.59), 340 (w/c: 0.40 - 0.46) and 390 (w/c: 0.36 - 0.42) kg/m³ respectively. The concrete types with 390 kg/m³ are generally judged as high performance types of concrete.

The project focuses on exposure to seawater and natural weather as well as exposure to de-icing chemicals in the same amount as is used on the main roads in North Jutland. The project comprises both exposure sites in the harbour of Hirtshals and on Aalborg Portland's area. In this test series, 16 different concrete mixes are included. As reference, specimens have been stored in water at 20  $^{\circ}$ C.

The project was started in close co-operation with companies from the Danish concrete industry, including ready mix producers, contractors, consultants as well as additive producers.

# 2 DESCRIPTION OF THE FULL SCALE TESTS

The elements were cast in-situ at the inland exposure site at Aalborg Portland, figure 2A. The elements comprised of  $1.0 \cdot 1.0 \cdot 0.2$  meter flags to be exposed at the inland exposure site, as well as some  $1.0 \cdot 2.0 \cdot 0.2$  meter elements to be exposed in Hirtshals harbour. Some  $1.0 \cdot 2.0 \cdot 0.2$  m elements have also been exposed at the inland exposure site in order to judge on the influence of horizontal versus vertical casting and exposure. In addition, more than 60 cylinders, beams and cubes for laboratory measurements were cast for each concrete type.

Experimental set-up for measuring corrosion of the reinforcement was planned. Reinforcement mesh with varying cover thickness was placed in some of the elements. However, neither the set-up nor the actual measuring technique did result in consistent and reliable results, and the very time consuming measurements have been stopped.

Freeze/thaw resistance was judged with the former Nordic method. This method was withdrawn in 1985, after a recommendation from a Nordic Miniseminar "Beton & Frost" held in Køge 1983. /1/. The temperature control as well as the multidirectional temperature gradients made the method so in exact, that the results are meaningless. Hence, no initial information on laboratory freeze/thaw durability test are available.

Figure 1 shows the arrangement of the test specimens at the inland exposure site at Aalborg Portland.



- Figure 1. Arrangement of the test specimens at the inland exposure site at Aalborg Portland. Elements B, D & E have reinforcing mesh. Element E is exposed to de-icing salt during the winter season (3% NaCl solution, technical grade). Element F is the element situated in Hirtshals Harbour, only situated at the test area during casting of the elements /2/.
- Figure 2A & 2B shows the actual test sites.



*Figure 2A: Inland exposure site at Aalborg Portland. Elements E to the left (Photographed in July 2001)* 

For exposure to seawater in Hirtshals Harbour, a special rig of galvanised steel was made and mounted on the quay. The elements are placed in a depth so they are totally immersed during tide, and the upper  $\frac{1}{2}$  meter is exposed to air during ebb tide, as seen on figure 2B.



Figure 2B. Elements in Hirtshals Harbour, ebb tide. (Photographed in July 2004)

### **3 MATERIALS**

Chemical composition of the cement and the mineral additives are given in table 1. The mix design for the 16 different concrete types is presented in table 2.

ement ana	minerai aaan	1105			
	CEM I	CEM I	CEM II / B-V	Elvenh	Cilian Euro
	42.5	42.5 SR	42.5	Fly ash	Silica Fullie
%	21	24	27	53	92
%	5.2	2.5	10.3	31	2.2
%	3.0	3.1	3.6	7.4	0.3
%	63	66	52	4.9	0.4
%	1.0	0.7	1.1	1.4	1.0
%	2.9	1.8	2.3	0.7	0.7
%	1.5	0.9	2.3	2.4	3.0
O, %	0.6	0.3	0.8	1.8	2.6
tent, %	< 5	-	20 - 25	-	-
n²/kg	430	310	450	-	-
$C_3S$	55	60	55	-	-
$C_2S$	20	26	20	-	-
$C_3A$	10	2	10	-	-
$C_4AF$	9	10	9	-	-
gth, MPa	54	50	51	-	-
	$\frac{2}{3}$ $\frac{2}$	$\begin{tabular}{ c c c c c } \hline CEM I \\ \hline 42.5 \\ \hline & 21 \\ \hline & 42.5 \\ \hline & 21 \\ \hline & & 3.0 \\ \hline & & 5.2 \\ \hline & & 3.0 \\ \hline & & 5.2 \\ \hline & & & 3.0 \\ \hline & & & 5.2 \\ \hline & & & & 3.0 \\ \hline & & & & 5.2 \\ \hline & & & & & 3.0 \\ \hline & & & & & 5.2 \\ \hline & & & & & & 3.0 \\ \hline & & & & & & 5.2 \\ \hline & & & & & & & 5.5 \\ \hline & & & & & & & c_{3}S \\ \hline & & & & & & & c_{3}S \\ \hline & & & & & & c_{3}S \\ \hline & & & & & & c_{3}S \\ \hline & & & & & & c_{3}S \\ \hline & & & & & & c_{3}S \\ \hline & & & & & & c_{3}S \\ \hline & & & & & & c_{3}A \\ \hline & & & & & & c_{4}AF \\ \hline & & & & & & ght, MPa \\ \hline & & & & & 54 \\ \hline ext{tabular}$	$\begin{tabular}{ c c c c c } \hline CEM I & CEM I \\ \hline 42.5 & 42.5 & SR \\ \hline 42.5 & 42.5 & SR \\ \hline 42.5 & 42.5 & SR \\ \hline 5.2 & 2.5 \\ \hline $	CEM I         CEM I         CEM I         CEM I / B-V $42.5$ $42.5$ SR $42.5$ $\%$ $21$ $24$ $27$ $\%$ $5.2$ $2.5$ $10.3$ $\%$ $3.0$ $3.1$ $3.6$ $\%$ $63$ $66$ $52$ $\%$ $1.0$ $0.7$ $1.1$ $\%$ $2.9$ $1.8$ $2.3$ $\%$ $1.5$ $0.9$ $2.3$ $0, \%$ $0.6$ $0.3$ $0.8$ tent, $\%$ $< 5$ - $20 - 25$ $1^2/\text{kg}$ $430$ $310$ $450$ $C_3S$ $55$ $60$ $55$ $C_2S$ $20$ $26$ $20$ $C_3A$ $10$ $2$ $10$ $C_4AF$ $9$ $10$ $9$ $gth, MPa$ $54$ $50$ $51$	CEM I         CEM I         CEM I         CEM I         CEM I/B-V         Fly ash $42.5$ $42.5$ SR $42.5$ Fly ash $32.5$ $42.5$ SR $42.5$ Fly ash $32.5$ $21$ $24$ $27$ $53$ $36$ $5.2$ $2.5$ $10.3$ $31$ $30$ $3.1$ $3.6$ $7.4$ $30$ $3.1$ $3.6$ $7.4$ $30$ $3.1$ $3.6$ $7.4$ $30$ $3.1$ $3.6$ $7.4$ $30$ $0.7$ $1.1$ $1.4$ $40$ $0.7$ $1.1$ $1.4$ $42.9$ $1.8$ $2.3$ $0.7$ $30$ $0.9$ $2.3$ $2.4$ $0.8$ $0.8$ $1.8$ $1.8$ tent, $\%$ $< 5$ $ 20 - 25$ $ C_3S$ $55$ $60$ $55$ $ C_2S$ $20$ $26$

Table 1: Cement and mineral additives

When type II additions were used, activity factors of 3 and 0.3 respectively for silica fume and fly ash were used in the mix-design. These activity factors are related to the strength giving properties of the products. In accordance with EN 206-1 /3/, calculation of equivalent w/c-ratios is done with activity factors of 2 and 0.4 respectively for silica fume and fly ash.

		1	2	3	4	5	6	7	8	9	10	11	12	13	14	15	16
			260	kg cer	nent		-		340 kg	cemen	t			390	kg cer	nent	
Cement ^{a)}		BC	RP	SR	RP	BC	BC	RP	SR	BC	SR	RP	BC	RP	SR	SR	BC
Cement	kg/m ³	261	200	264	261	199	333	256	340	260	327	334	397	300	390	300	300
Flyash	kg/m ³	-	-	-	-	-	-	-	-	-	65	-	-	-	-	-	-
Silica fume	kg/m ³	-	20	-	-	21	-	26	-	26	-	-	-	30	-	30	32
0/4 mm	kg/m ³	718	780	732	725	775	630	725	653	703	600	635	565	640	592	670	635
4/8 mm	kg/m ³	125	125	125	125	125	125	125	125	125	125	125	125	125	125	125	125
8/16 mm	kg/m ³	1100	1100	1100	1100	1100	1100	1100	1100	1100	1100	1100	1100	1100	1100	1100	1100
Water	kg/m ³	141	125	138	140	143	146	141	142	140	141	144	165	140	142	136	143
w/c-ratio #)	-	0.54	0.52	0.52	0.54	0.59	0.44	0.46	0.42	0.45	0.40	0.43	0.42	0.39	0.36	0.38	0.39
Slump	mm	60	50	45	50	40	55	60	60	55	65	50	50	70	60	50	40
Air content	%	5.8	5.6	5.7	5.8	5.5	5.6	5.5	6.0	5.9	5.8	5.8	5.4	5.8	5.4	5.6	5.5
m)				~ -					~ ~ ~ ~								

Table 2:Concrete mix-design

^{III}): Cement – types: RP: CEM I 42.5. SR: CEM I 42.5 – SR. BC: CEM II / B-V 42.5

#): w/(c+2SF+0.4FA)

Originally it was planned to use absolutely sound aggregates in order to avoid aggregate related durability problems. However, at too late stage it appeared that the sand fraction did contain alkali reactive materials in the form of porous opaline flint. Thus problems related to alkali silica reactions (ASR) must be foreseen. The coarse aggregates consisted of sound sea dredged rounded granite particles. However, even after 25 years exposure in Hirtshals Harbour, ASR has only been found in concrete types 1, 3, 4, 11 and 15. On flags from the inland exposure site, surface cracks presumably originating from ASR are seen on concrete types 1 (only the horizontal salted element), 4 (horizontal elements) and 11 (both horizontal and vertical elements). In particular, concrete types 4 and 11 suffer from tremendous crack formation.

## **4 PROPERTIES OF THE HARDENED CONCRETE**

A comprehensive amount of investigations on the 16 types of concrete have been carried out, both initially and during the succeeding years. The tests and measurements have involved:

- a) Macro analysis on drilled cores.
- b) Structural analysis on thin sections.
- c) Measurements of air void characteristics on plane polished sections.
- d) Freeze/thaw resistance according the former Nordic method.
- e) Measurement of development of compressive strength, splitting tensile strength and Emodulus, both on samples stored in water at 20 deg. C, and on drilled cores.
- f) Measurement of drying shrinkage.
- g) Measurements of chloride profiles on the elements in Hirtshals Harbour.
- h) Carbonation depths.
- i) Leaching of heavy metals

In this paper only c), e), g), h) & i) are dealt with. Survey reports for 1, 5, 10 and 18 years exposure are published in  $\frac{2}{\frac{4}{5}}$  &  $\frac{6}{6}$  respectively.

## 4.1 Air void characteristics, ad c)

Air void characteristics were measured on plane polished sections from cores from the hardened concrete elements. The data are presented in table 3

 Table 3:
 Air void characteristics, hardened concrete (1983)

10010 01 11		0			,				- (	<i></i> ,							
Concrete type		1	2	3	4	5	6	7	8	9	10	11	12	13	14	15	16
Air content	%	2.8	2.8	4.7	4.5	2.6	1.9	1.6	5.6	4.7	4.0	4.3	4.7	5.6	3.5	4.7	2.8
Spec. surface	$mm^{-1}$	50	41	28	52	32	59	35	29	37	36	30	32	29	36	30	42
Spacing factor	mm	0.13	0.16	0.18	0.10	0.20	0.13	0.23	0.16	0.14	0.15	0.17	0.15	0.16	0.16	0.17	0.15

#### **4.2** Development of mechanical properties, ad e)

#### *Compressive strength*

For the 16 different types of concrete, measurement of the development of compressive strength properties have been made at laboratory water stored specimens as well as on drilled cores from the exposed elements. The following tables 4 - 8 present the results.

Table 4: Development of compressive strength, MPa. 20 °C watercured specimens

							<u> </u>									
Concrete type	1	2	3	4	5	6	7	8	9	10	11	12	13	14	15	16
7 days	20	21	17	21	16	24	30	31	21	33	30	28	30	38	39	29
28 days	27	36	25	28	27	32	51	40	34	44	38	38	49	48	62	46
28 days $^{\tt m}$	32	43	30	34	32	38	61	48	41	53	46	46	59	58	74	55
1 year	44	49	35	36	40	50	64	57	47	70	47	54	57	67	69	57
5 years	44	46	38	31	46	49	65	63	54	75	48	58	64	73	73	60
10 years	43	44	37	30	44	47	59	63	50	72	47	59	62	77	74	62
25 years	44	43	39	29	47	46	64	63	50	77	45	60	66	76	77	65

[#]: Cube strengths calculated in accordance with the introductory remarks

*Table 5: Compressive strength (MPa) of concrete water cured 7 days, successively stored in air at 20 °C and relative humidity of 60 % for 25 years* 



■ 28 days ref. ■ 10 years water ■ 25 years indoor ■ 25 years water



Due to reinforcement mesh in elements B, E & F, no compression strength data were measured after 25 years, since the cores contains rebars, which influenced the compressive strength measurement.

Table 6: Development of compressive strength, MPa. Vertical drilled cores from element Band E. (Elem. E has been salted during winter season).

											• /•					
Concrete type	1	2	3	4	5	6	7	8	9	10	11	12	13	14	15	16
28 days	26	38	19	20	29	28	48	33	38	40	35	-	-	52	61	46
1 year	39	49	36	28	38	44	69	47	43	52	46	43	57	65	69	52
1year, el. E	39	39	43	28	42	44	50	48	44	65	43	53	54	58	65	50
10 years	36	44	35	18*	37	44	60	47	42	55	**	43	46	65	59	50
18 years	37	47	37	20*	48	45	69	53	52	66	38*	51	60	76	76	55

*: Cracks from ASR

**: Disintegrated from ASR

Table 7:	Development o	f compressiv	e strength,	, MPa. Horizon	tally a	drilled cores,	element D

Concrete type	1	2	3	4	5	6	7	8	9	10	11	12	13	14	15	16
28 days	32	31	28	24	28	33	50	38	36	43	40	-	-	51	67	52
1 year	42	41	36	36	36	46	62	48	67	62	52	51	47	64	68	55
10 years	44	37	32	31	38	47	61	46	51	60	41*	55	56	59	61	61
25 years	44	35	31	31	37	46	54	48	53	70	-	54	57	65	77	
*: Cracks from AS	R															

Concrete type	1	2	3	4	5	6	7	8	9	10	11	12	13	14	15	16
<b>v</b> 1							-									

1 year	39	39	43	28	42	44	49	48	44	65	43	53	54	58	65	50
10 years	47	43	48	12*	50	48	56	58	44	70	35*	59	56	71	67	57
* Creates from AS	D															

*: Cracks from ASR

## Tensile splitting strength

Development of tensile splitting strength has been measured on 20°C water cured cast cylinders, see table 9 and figure 4.

Table 9. Development of tensue splitting strengths, MPa. 20 C water curea cast cyl	Fable 9: 1	Development o	of tensile splittin	g strengths, MPa.	$20 \ ^{o}C$	water cured	cast cylinders
------------------------------------------------------------------------------------	------------	---------------	---------------------	-------------------	--------------	-------------	----------------

Concrete type	1	2	3	4	5	6	7	8	9	10	11	12	13	14	15	16
28 days	4.2	4.8	4.3	4.0	4.4	4.6	5.7	5.1	5.0	3.4	4.6	4.8	5.2	6.1	6.4	5.7
1 year	5.1	5.5	4.8	4.2	6.0	5.8	6.8	5.9	5.7	6.9	5.2	6.3	5.6	6.6	7.3	6.5
10 years	5.8	5.8	4.6	3.8	6.5	6.6	7.3	6.6	6.3	7.5	5.4	7.3	5.9	6.7	8.0	6.8
25 years	3.8	3.8	3.4	2.5	4.1	4.3	4.7	4.3	4.1	5.2	3.8	4.3	4.3	4.9	5.1	4.4



*Figure 4:* Tensile splitting strength, MPa for concrete continuously water cured at 20 °C.

#### Elastic modules

For the 16 different types of concrete, measurement of development of elastic properties has been made at laboratory water stored specimens as well as on drilled cores from the exposed elements. The following tables 10 - 12 and figures 5 - 7 present the results.

*Table 10: Development of static modulus, GPa. 20 °C water cured specimens. For 28 days even cores from elements B (vertical) and D, also 25 years, (horizontal).* 

				1				-		~	1	-				
Concrete type	1	2	3	4	5	6	7	8	9	10	11	12	13	14	15	16
7 days	26	25	24	25	26	28	27	30	25	30	29	29		33		29
28 days	30	33	28	27	30	29	32	34	32	31	31	30		34		32
28 days, el. B	27	29	27	23	28	24	27	28	27	28	27	33	32	36	43	34
28 days, el. D	29	29	25	25	27	26	30	28	30	28	26	31	31	36	46	34
1 year	34	36	32	31	36	38	40			38	35	39	36	38	37	35
5 years	37	38	38	27	38			42	37	42	33	38	39	41	39	
10 years	35	33	34	30	35	32	32	37	30	42	36	34	33	41	39	38
25 years	31	30	26	21	31	29	32	31	27	33	26	31	31	33	33	31
25 years, el. D	27	25	19	22	27	24	28	24	27	39	25	28	31	27	29	



Figure 5: Variation of static modulus with age; 20 °C water cured specimens

		vaici	Chite	i spe	cinic	115										
Concrete type	1	2	3	4	5	6	7	8	9	10	11	12	13	14	15	16
7 days	45	46	42	42	43	45	49	52	44	51	47	48	48	55	43	47
28 days	50	51	57	47	51	51	54	56	51	53	53	53	52	56	49	50
1 year	54	58	48			54	57	56	50	52	57	54	56	59	57	57
5 years	57	59	50	48	59	59	61	64	56	61	55	58	60	64	63	58
10 years	49	50	46	42	49	49	53	53	47	52	48	51	49	55	54	50
25 years	50	51	47	41	51	50	53	54	50	53	49	52	50	55	54	51

Table 11: Development of dynamic modulus, GPa. 20 °C watercured specimens

Table 12: Dyn	amic	moa	uius,	GPU	i. 23 _.	years	)									
Concrete type	1	2	3	4	5	6	7	8	9	10	11	12	13	14	15	16
Water cured	50	51	47	41	51	50	53	54	50	53	49	52	50	55	54	51
Element B	33	47	40		46	41	46	45	45	48		48	46	47	45	43
Element D	40	43	39	40	40	39	42	45	42	43	45	43	42	46	46	43
Elem. E, Salted	25	41	44		44	32	46	52	44	52		50	46	50	51	48
Hirtshals			46			47	50		48	52		49	49	54	54	50

Table 12: Dynamic modulus, GPa. 25 years



🔲 7 days 🖾 28 days 🗖 1 year 🔳 5 years 🖾 10 years 🗮 25 years

Figure 6: Variation of dynamic modulus with age; 20 °C water cured specimens



Figure 7: Dynamic modulus, MPa, after 25 years, for five different storage conditions.

## 4.3 Measurement of chloride profiles, ad. g).

After  $8\frac{1}{2}$  year, 18 years and 25 years, chloride profiles in the elements exposed in Hirtshals Harbour have been measured. Data from  $8\frac{1}{2}$  & 18 years are published in /6/. The chloride content in the seawater is 19 g/l. /7/. The chloride profiles measured after  $8\frac{1}{2}$  and 25 years were determined with a chemical analysis, while the 18 years profile were determined with another technique. This technique gave rise to much higher chloride content than measured with chemical titration after 25 years. For this reason, these data are omitted in the following graphs, figures 8 - 10.

Some of the elements have been removed from the harbour after the  $8\frac{1}{2}$  years measure. This is partly due to alkali silica reaction, #4 & #11, and replacement of some elements, #1, #2, #5 &

#8 with some other concrete mixes. Chloride profiles after 25 years are given in table 13 (continuously submerged) and table 14 (splash zone)

A chloride content of 0.07 weight pct. of concrete is generally accepted as a critical content regarding chloride induced corrosion of reinforcement.

The elements contain reinforcement mesh in depths of 10, 20 and 30 mm. Eventual corrosion was judged on drilled cores.

	•	0		Depth from s	surface [mm]		
Element	Binder	2.5	7.5	12.5	17.5	32,5	37.5
3	SR	0.15	0.22	0.19	0.19	0.15	0.17
6	BC	0.33	0.30	0.24	0.26	0.12	0.08
7	<b>RP+MS</b>	0.27			0.24	0.14	0.09
9	BC+MS	0.18	0.21	0.19	0.17	0.03	0.01
10	SR+FA	0.425	0.34	0.32	0.27	0.08	0.02
12	BC	0.64	0.58	0.54	0.38	0.04	0.02
13	RP+MS	0.43	0.44	0.40	0.30	0.19	0.14
14	SR	0.56	0.49	0.36	0.34	0.22	0.20
15	SR+MS	0.38	0.34	0.29	0.25	0.11	0.07
16	BC+MS	0.34	0.34	0.29	0.21	0.03	0.00

 Table 13: Chloride profiles (weight pct. of concrete) after 25 year's exposure in Hirtshals

 Harbour. Samples from the continuous submerged part of the element.

RP: CEM I 42.5. SR: CEM I 42.5 – SR. BC: CEM II / B-V 42.5

Table 14: Chloride profiles (weight pct. of concrete) after 25 year's exposure in Hirtshals Harbour. Samples from the part of the element placed in the splash zone, above sea level..

				Depth from s	surface [mm]		
Element	Binder	2.5	7.5	12.5	17.5	32.5	37.5
3	SR	0.16	0.18	0.17	0.15	0.12	0.11
6	BC	0.58	0.38	0.30	0.23	0.03	0.00
7	RP+MS	0.39	0.30	0.25	0.19	0.07	006
9	BC+MS	0.07	0.16	0.15	0.13	0.02	0.01
10	SR+FA	0.68	0.50	0.41	0.31	0.01	0.00
12	BC	0.45	0.43	0.35	0.28	0.03	0.02
13	RP+MS	0.40	0.32	0.23	0.17	0.03	0.02
14	SR	0.26	0.23	0.22	0.17	0.14	0.13
15	SR+MS	0.42	0.36	0.30	0.23	0.06	0.03
16	BC+MS	0.20	0.21	0.15	0.08	0.01	0.00

RP: CEM I 42.5. SR: CEM I 42.5 – SR. BC: CEM II / B-V 42.5



*Figure 8: Chloride profiles for concrete types with CEM I 42.5 after 8¹/₂ and 25 years exposure. 28 days compressive strength given in brackets. "asl" means above sea level, i.e. the splash zone.* 



*Figure 9: Chloride profiles for concrete types with CEM I 42.5 - SR after 8¹/₂ and 25 years exposure. 28 days compressive strength given in brackets. "asl" means above sea level, i.e. the splash zone.* 



Figure 10: Chloride profiles for concrete types with CEM II / B-V 42.5 after 8¹/₂ and 25 years exposure. 28 days compressive strength given in brackets. "asl" means above sea level, i.e. the splash zone.

Figures 8 - 10 illustrate, that the chloride ingress in the splash zone is less than in the submerged part of the element. This is the case even if the elements are flooded often due to tide as well as waves from ferries and large fishing vessels.

### 4.4 Carbonation depth, ad. h)

After 10, 20 and 25 years indoor exposure, 60 % relative humidity, 20 deg. C, the depth of carbonation was measured with phenolphthalein, se table 15. Figure 11 a-c shows the relationship between age and carbonation depths, for the three concrete quality classes.

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Figure 11: Carbonation depths for the three quality classes, indoor storage, 60 % RH, 20 °C

Table 15: Depth of carbonation after 10, 20 and 25 year's indoor exposure																	
Concrete type		1	2	3	4	5	6	7	8	9	10	11	12	13	14	15	16
10 years	mm	27	28	41	30	34	24	18	20	25	16	18	16	16	15	14	17
20 years	mm	38	37	¤	42	¤	32	22	23	35	22	23	20	21	12	21	22
25 years	mm	38	43	¤	¤	¤	37	27	27	38	26	27	24	25	23	23	25
¤: > 50 mm																	

Figure 11 and table 15 illustrates clearly, that carbonation is dependent on the quality of concrete. There is no direct explanation for the spread in results in figure 11a) and 11b). Table 16 gives data for carbonation depths on vertical surfaces exposed to natural weather.

Table 16: Depth of carbonation on a vertical surface (element D) at the inland exposure site after 25 years exposure.

	-0 900	15 0.0	$P^{ODU}$														
Concrete type		1	2	3	4	5	6	7	8	9	10	11	12	13	14	15	16
25 years	mm	6	10	8	2	19	2	3	2	9	1	1	0	1	0	1	3

# 4,5 Leaching of heavy metals, ad i)

In 2007, after 24 years exposure in sea water, cores were taken from the elements in Hirtshals Harbour. Content of heavy metals, As, Cd, Cu, Ni, Pb, Sb, Ti, Co, Mn, V & Zn were measured both from the central part of the element, and from the outer 5 mm layer. The overall result is that no heavy metals have leached out during this time period.

# 5 **DISCUSSION**

## 5.1 Air void characteristics

In the Danish standards, requirements for the air void characteristics in air entrained concrete are spacing factor < 0.2mm, or alternatively a specific surface > 25 mm⁻¹. The latter requirement is no longer used. All the concrete types fulfil both requirements, as can be seen from Table 3. Visual inspection on the elements shows no degradation, except a slight abrasion on two elements having higher capillary porosity in the surface layer due to insufficient curing.

# 5.2 Development of strength and modulus of elasticity

## Compressive strength

The compressive strength increases with age during the whole 25 years period. The highest strengths are obtained by specimens continuously stored in 20 °C water. Dry indoor storage as well as strength of cores taken horizontally from a vertical element, had a lower strength, than those from water storage.

From time to time it has been postulated, that the strength of silica fume containing concrete decreases with age. The data in tables 4 to 8 demonstrate that this is not the case. Table 9 in  $\frac{8}{8}$  further support this.

## Tensile splitting strength

Tensile splitting strength was measured on samples which have been stored in 20 °C water for 28 days, 1, 10 & 25 year. From figure 4 a significant decrease, in average a decrease to 2/3 from 10 to 25 years can be seen.
Modulus of elasticity

Table 10 and 11, as well as figures 5, 6 & 7 shows the variation of static as well as dynamic modulus. It can be seen, that the modulus increase during he first 5 years, where after it decreases app 20 % and 10 % respectively for the static and dynamic modulus.

For most of the outdoor exposed concrete, element D, the static modulus after 25 years are smaller than the static modulus measured initially after 28 days.

# 5.3 Chloride profiles

On the chloride profiles presented in figures 8 - 10, some different changes over time can be seen.

Unfortunately, due to the ASR damage, no chloride profiles for concrete with CEM I 42.5 without type II additions could be measured after neither 18 nor 25 years.

In figure 9, a very flat chloride profile is observed for concrete with CEM I 42.5 - SR alone. Similar profiles have been measured in Sweden on same types of concrete /9/. It is believed, that this is caused by the low  $C_3A$  content, and hence the low chloride binding capacity. Addition of fly ash and silica fume reduces the chloride ingress, either because of an increased amount of aluminate or because of a more dense structure; 25 % fly ash being more effective than 10 % silica fume.

Figure 10 shows the chloride ingress in concrete with CEM II / B-V 42.5. Since this cement is identical with a binder of CEM I 42.5 + 25 % fly ash, the positive effect of fly ash can be seen by comparison of figures 10 and 8. As for the CEM I 42.5 - SR, fly ash is more effective than silica fume.

In spite of chloride contents above the 0.07 %, no corrosion on the reinforcement mesh was observed on cores taken from the elements, even in only 10 mm depth. This was the case for cores taken above sea level as well as below sea level.

# 5.4 Carbonation

Carbonation in dry indoor climate is not a durability problem. Hence, carbonation in outdoor stored concrete is more relevant. The worst situation is carbonation on vertical surfaces exposed for rain.

For such exposure, the Danish regulations require a w/c ratio below 0.55 and a concrete cover of 20 + 5 mm. Since the w/c ratio of concrete type 5 is 0.59, none of the 15 different concretes complying with the w/c-ratio requirement, have a carbonation depth above 10 mm after 25 years. Thus, for the stipulated minimum life time of 50 years according to EN 206-1, there is no risk of reinforcement corrosion due to carbonation.

# 5.5 Leaching

The European Construction Products Directive /10/ focuses much on the environmental impact from construction materials. For concrete, special interest concerns possible leaching of dangerous materials to drinking water and ground water. Hence, data confirming, that no, or very limited, leaching of heavy materials have occurred during 25 years in sea water, will have an impact on the coming generic classification of concrete.

# 6 CONCLUSION

25 years of exposure of 16 different types of concrete has demonstrated that

- Use of type II additions as fly ash and silica fume improves the long-term durability of concrete with regard to alkali silica reactions and chloride penetration.
- There is no evidence of a negative strength development in concrete with silica fume.
- The low chloride binding capacity of cements with low aluminate content can be improved by addition of fly ash. Addition of silica fume reduces the chloride ingress, probably due to the finer pore structure and lower permeability.
- The elastic modulus increase during the first five years, after which it decrease again.
- The tensile splitting strength follows the same trend as the elastic modulus. Due to lack of five years data, the apparent maximum is at ten years
- In high performance concrete, carbonation is independent of cement and addition type, in contradiction to lower concrete qualities.
- None of the 16 concrete types have suffered from frost damage; even the air content in the hardened concrete was very low in several of them.

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# Shore Approach 26 years experience with high quality concrete in XS3 exposure



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# ABSTRACT

A 590 m long submerged concrete tunnel was built in 1982 at the rocky and harsh west coast of Karmøy to protect two gas pipelines coming in from the North Sea. The tunnel was produced in two dry docks in 5 elements. The concrete quality was very close to today's requirements for concrete exposed to class XS3 in the Norwegian Standard. The tunnel element in the splash zone has been examined several times, latest during summer 2008, 26 years after installation. This presentation concentrates on chloride ingress. The results after 26 years of exposure are used to estimate the safety against failure (the reliability index) of the structure when the concrete cover is set equal to the requirements in today's Norwegian Standard.

**Key words:** Existing structure, chloride ingress, service life, requirements in Norwegian Standard.

# **1. INTRODUCTION**

A part of the Statpipe Development Project is the landfall for two gas pipelines on the exposed western coast of Norway. The pipelines are placed inside a submerged concrete tunnel which acts as an underwater protecting bridge over the rocky sea bed. The 590 m long tunnel was produced in five separate elements in two different dry docks. The tunnel starts at a water depth of 30 m and ends up at water level. The tunnel elements were produced and installed during summer and fall of 1982.

Due to a very tight construction schedule, no time was available for mix design tests, and a concrete quality with ample margin for strength requirement was chosen. The structure is exposed to a very severe environment, both from a statically and durability point of view. The structure has therefore been continuously inspected with more detailed inspections after 7 years

(1989), 12 years (1994) and 26 years (2008) in service, especially with respect to concrete technology, durability and rest service life point of view.

Durability performance was much higher than normal at that time, and very close to the specifications in Norwegian Standards today for the same exposure conditions. The results up to 26 years in service are of great interest for evaluating the requirements given in Norwegian Standards today.

# 2. STRUCTURE

A part of the Statpipe Development Project is a landfall for the gas pipelines coming from the Statfjord Field. The two pipelines are part of the 650 km long Statpipe gas line system in the North Sea.

In order to protect the pipelines at the shore approach on the rocky west coast of Karmøy, a submerged concrete tunnel was planned and built. It has a total length of 590 m, starting at a water depth of 30 m and ending up at water level. The tunnel consists of five elements ranging from 90 to 150 m length. The cross sectional area varies from 30 to 45 m², and the largest element has a displacement of 7000 tons. The tunnel elements rest on six heavy foundations, the lower part of which were cast under water, see Figure 1.



Figure 1 – Submerged concrete bridge (tunnels) at Karmøy [1]

Due to a very tight construction schedule, the tunnel elements were produced in dry docks in Stavanger and Kristiansand, while the foundation work progressed. The prefabricated tunnel elements were towed to the installation site, water-ballasted, pulled down to the foundations, and then flooded. After this, the pull in of the pipelines could be performed.

During the design process, it was found that high strength concrete was favourable. The mix design was partly based on experience from the constructions of oil platforms in the North Sea; however, even higher strength than common at that time was required. Super plasticizers and silica fume were available. The design concrete strength was C65 (characteristic cube strength), which was the highest strength grade covered by the Norwegian Standard NS 3473 at that time.

In general, the structure was heavily reinforced; on average there were 250 kg of reinforcement and 80 kg of pre-stressing cables in each cubic meter of concrete. The concrete mixes were tailored to meet these conditions and the concrete work was executed perfectly.

The elements were all installed with an accuracy of a few centimetres. The whole project, including design and construction was completed during nine months in 1982. The pull in of the gas pipes was performed, as scheduled, in the spring of 1983. A more detailed presentation of the structure is given in [1].

# **3.** CONCRETE MIX DESIGN

Table 1 shows the most important information about the concrete mixtures. The strength results from the total production period are also shown in Table 1. The concrete strength obtained is significantly higher than the necessary values used in the design. The proportions were, however, not changed during the construction period for two reasons:

- A reserve in the concrete strength was appreciated in order to be in the position of making small adjustments of the mix without causing concern for the strength.
- Due to the very short design and construction period, major changes in the mix design might lead to delay.

	Production site				
Property	Kristia	ansand	Stavanger		
Cement type P30	400 1	kg/m ³			
P30 4A				420 kg/m ³	
Silica Fume	32.5	kg/m ³	$30 \text{ kg/m}^3$		
w/(c+si)-ratio	0.36 0.3		0.34		
Slump	200-260 mm		200-260 mm		
Element no	1	2	3	4	5
28 days cube strength, MPa					
Mean value	77.5	79.7	82,9	85,4	84,7
Lowest value	69.9	68.7	76,0	73,9	73,6
Standard deviation	5.9	6.4	3,8	5,2	6,1
Characteristic value	68.9	70.4	76,8	77,7	75,1
Required characteristic value	60	60	65	65	65
Elastic modulus (mean), GPa	4	0		32.5	

Table 1 – Concrete for submerged tunnel elements at Shore Approach, Karmøy [1]

Cores from splash zone of element 1 are tested and reported in this report

The two types of cement shown in Table 1 meet both the requirements for CEM I according to EN 197-1. P30-4A was developed for the oil platforms. It has a higher strength potential than P30 and lower  $C_3A$  (approximately 5.5 %) content than in P30 (approximately 7 %). Silica fume was used for higher strength potential only, a more durable concrete was a bonus.

Mix design, especially for the elements produced in Kristiansand, is very close to the requirements in Norwegian Standards today for concrete exposed to marine environment. It is therefore of great interest to study durability properties of this existing structure in the right exposure in order to evaluate the durability requirements in our standards. Tunnel element 1 has been inspected continuously and more detailed quality inspection have been performed in 1989 (7 years of exposure), 1994 (12 years of exposure) and 2008 (26 years of exposure).

# 4. TEST PROGRAM AND METHODS

# 4.1 General

All test results in this report come from cores drilled in the splash zone of element 1, which was produced in Kristiansand. The coring area is located  $\pm 8$  m from the manhole closest to the shore.

The test program has not been the same for each term. However, the most important properties are tested at different times, using more or less the same test methods.

In this paper, only chloride ingress and some related results will be covered. A more comprehensive report will be issued within the COIN-project during 2009.

	Year of inspection					
Property	1989	1991	1994	2008		
Cl-profile	Х	Х	Х	Х		
Cl-resistance			Х	Х		
Electrical resistivity				Х		
Thin section analysis	Х			Х		
Microstructure analysis	Х			Х		
Capillary absorption	Х			Х		
Compressive strength	Х			Х		
Density				Х		
References	[2], [3]	[4]	[5], [6]	[7], [8], [9]		

Table 2 – Test program at different exposure times

# 4.2 Chloride profile on exposed surface

The chloride profile on exposed surfaces has been tested at all intervals. However, the number of points on each profile and the test method has varied. The results at the different terms can therefore not be compared directly.

**1989:** The results are based on one single cylinder drilled through the slab. The diameter was 69 mm. Chloride content was measured at six different depths from both ends, outer and inner surfaces, by the Quantab method. However, the depth intervals were too few where chloride had penetrated to calculate reliable diffusion coefficients [2].

**1991:** The results are based on six cylinders. The diameter is not known, but most probable in the range of 90 mm. Each profile consists of 3 - 4 measured points. The test method is not

found, but most probable the Quantab method was used. The number of measuring points was too few to calculate reliable diffusion coefficients [4].

**1994:** The results are based on six cylinders with diameter 80 mm. Dust was ground in intervals of 3 mm and the chloride content was measured according to a spectrophotometric method given in SINTEFs procedure KS 70 108. Each chloride profile was based on 14 measuring points and diffusion coefficients could be calculated in a reliable way [5].

**2008:** The results are based on four cylinders, three with diameter 74 and one with 95 mm. Dust was ground in intervals of 5 mm in the outer part and intervals of 10 - 14 mm further in. The chloride content was measured according to a spectrophotometric method given in SINTEFs procedure KS 14-05-04 128. Each chloride profile was based on 9 - 14 measuring points and diffusion coefficients could be calculated in a reliable way [7].

# 4.3 Chloride resistance on virgin surfaces and laboratory cast samples

The inner virgin surface of drilled cylinders was tested at the two latest intervals (1994 and 2008) by a bulk diffusion method. In 1994, the APM 302 method and in 2008 the Nordtest NT Build 443, respectively were used [6], [8]. APM 302 was the base for NT Build 443 method, and results from the two methods can be compared directly. The virgin surface was exposed to an aqueous NaCl solution of 165 g NaCl per liter solution for 35 days. After the exposure, dust was ground at intervals of 1 mm and chloride content was measured according to a spectrophotometric method. Each chloride profile was based on 14 measuring points and diffusion coefficients could be calculated in a reliable way.

As a part of a research project on concrete with lightweight aggregates, a comparison mix based on the Shore Approach mix was produced [10]. After 28 days curing, the concrete was exposed to natural sea water for 35 days and chloride ingress was measured. The method is equal to NT Build 443, except for the chloride content in water. This has an important effect on the surface chloride concentration, but not on the calculated diffusion coefficient. The results from this project can therefore be regarded as initial chloride diffusion coefficient after 28 days curing.

# 4.4 Microstructural analysis

This is a collective term for different analysis carried out partly in 1989 and 2008. It includes thin section analysis, back-scattering, X-ray diffraction and scanning electron microscopy (SEM). The objective of these analyses was to study any changes in the concrete surface exposed to sea water, especially ion exchange between cement paste and sea water [2], [3], [9].

# 5. TEST RESULTS

# 5.1 Chloride diffusion coefficients and surface concentration on exposed surface

Chloride profiles after 26 years of exposure are shown in figure 2. The mean curve is calculated based on the mean diffusion coefficient for the four samples.

Based on measured chloride profiles, all results are recalculated with the use of the same Excel spreadsheet based on Fick's second law of diffusion, equation 1. The results are given in table 3. The mean diffusion coefficient  $D_a$  is  $0.375 \cdot 10^{-12}$  m²/s with standard deviation  $0.111 \cdot 10^{-12}$  m²/s and the mean surface chloride concentration is 0.55 % of concrete mass with standard deviation 0.106 %.

$$C(x,t) = C_s - (C_s - C_i) \bullet erf\left(\frac{x}{\sqrt{4tD_a}}\right)$$
(1)

Where :

C(x,t) = Chloride content at depth x at time t.

- $C_s$  = Chloride content on the exposed surface.
- C_i = Chloride background content.
- $D_a$  = Apparent diffusion coefficient.
- x = Depth

t = Exposure time.

erf = Error function.



Figure 2 – Chloride profiles after 26 years exposure [7].

Table 3 also includes test results after 12 years of exposure, but none of the curves are included in this paper. Reference is made to [5]. The diffusion coefficient  $D_a$  is the so called "apparent" or in situ coefficient, meaning that it is a kind of an average value for the whole exposure period. The instantaneous coefficient at the time of inspection will be lower, how much lower is not known, but it will depend on the aging factor  $\alpha$ , see equation 9.

		1	$\mathbf{D}_{\mathbf{a}}$		Cs
Year	Age, years	10-1	² m ² /s	% of 0	concrete
		0.34		0.48	
		0.45	Mean	0.64	Mean
		0.32	0.420	0.72	0.497
1994	12	0.50		0.34	
		0.50	St. dev.	0.30	St. dev.
		0.41	0.078	0.50	0.164
		0.51	Mean	0.57	Mean
		0.41	0.375	0.69	0.55
2008	26	0.33	St. dev.	0.45	St. dev.
		0.25	0.111	0.49	0.106

Table 3 – Diffusion coefficient  $D_a$  and calculated surface chloride concentration  $C_s$ . [5], [7]

#### 5.2 Chloride diffusion coefficients on virgin surfaces and laboratory cast samples

The potential chloride diffusion coefficient  $D_p$  is calculated based on chloride profiles after bulk diffusion test at different terms. It is called the potential chloride diffusion coefficient. Results are shown in table 4 for 12 and 26 years old samples drilled from the structure. Table 4 also includes results from laboratory produced and tested concrete with the same mix design as the concrete in element 1 at Shore Approach. In principle the same bulk diffusion test method was used in all tests. However, the laboratory samples were exposed to natural sea water, not a NaCl solution. Based on experience, this has no significant influence on the calculated  $D_p$ , only the surface chloride concentration,  $C_s$ , will be influenced.

		$D_p$	Mean D _p	
Year	Age	$10^{-12} \text{ m}^2/\text{s}$	$10^{-12} \text{ m}^2/\text{s}$	Comments
	28 days	7.23	Mean	From laboratory cast concrete with
		8.24	7.75	mix design equal to Shore
		7.77	St. dev.	Approach concrete. Exposed to sea
			0.51	water for 35 days.
		3.34		Based on six samples drilled from
		2.38	Mean	structure. Exposed to NaCl-solution
		2.86	2.82	(165 g per litre solution) for 35
1994	12 years	2.97	St. dev.	days.
		2.51	0.34	
		2.84		
		2.15	Mean	Based on three samples drilled
		2.23	2.91	from structure. Exposed to NaCl-
2008	26 years	4.36	St. dev.	solution (165 g per litre solution)
			1.25	for 35 days.

Table 4 – Diffusion coefficient  $D_p$  based on bulk diffusion method. [6], [8], [10]

#### 5.3 Aging factor α

The aging factor  $\alpha$  can be calculated from equation 2 when results for two or more exposure periods are available.

$$D_t = D_0 \left(\frac{t_0}{t}\right)^{\alpha} \tag{2}$$

In this situation we have results for D_a after 12 and 26 years exposure, see table 3. In addition we have results for D_p for the same mix design produced in laboratory and tested according to a bulk diffusion method, as described in section 4.3. The result is given in table 4 and can be used as the diffusion coefficient after 35 days exposure. Based on this, the aging factor can be calculated as given in table 5. The spread of the diffusion coefficients used in the calculations are relatively high, meaning that the spread of the aging factors are high as well. The aging factor seems to be reduced with time. However, both after 12 and 26 years exposure, the apparent diffusion coefficients are so low that further chloride ingress will be very low independent of aging factor.

<u>Table 5 – Aging factors</u>	5	
From	То	Aging factor α
$D_{p35days} =$ 7.7 • 10 ⁻¹² m ² /sec	$D_{a12 years} = 0.42 \cdot 10^{-12} m^2/sec$	0.61
$D_{p35days} = 7.7 \cdot 10^{-12} m^2/sec$	$D_{a26 \text{ years}} = 0.375 \cdot 10^{-12} \text{ m}^2/\text{sec}$	0.54
$D_{a12 years} = 0.42 \cdot 10^{-12} m^2/sec$	$D_{a26 \text{ years}} = 0.375 \cdot 10^{-12} \text{ m}^2/\text{sec}$	0.13

#### 5.4 **Microstructural analysis**

The results from the testing in 1989 showed that calcium had been leached out and magnesium had replaced it in the outer 1-2 mm [3]. Such an ion exchange has been observed by others [11], [12], [13], [14]. Figure 3 shows the concentration of magnesium and calcium in the concrete.



*Figure 3 – Concentration (white dots) of magnesium (left) and calcium (right).* Surface of sample is up [3]

Thin cross section analysis, using crossed polarizers and lambda plate, turquoise coloured crystals were observed, which indicate brucite  $(Mg(OH)_2)$ . Magnesium is therefore most probable present as brucite, which results in a tighter surface layer than further into the concrete.

Test results from 2008 [9] did not confirm such a tight surface layer of brucite in two of three samples. The third sample showed a very thin (0.4 mm) layer with enriched magnesium content. However, the results in figure 2 shows that the mean curve calculated on the basis of the mean diffusion coefficient results in higher chloride contents in depths where the reinforcement is located compared to the curves based on measurements. This indicates that a tight surface layer results in a relatively steep chloride curve from the surface, which doesn't fit well to Fick's second law of diffusion.

# 6. ANALYSIS

### 6.1 General

The general question is how such data from an existing structure can be used in estimating the future development, rest service life and safety against failure. Mathematic models based on probabilistic principles exist and may be used. Such models are developed for estimating service life of new structures where all input parameters are more or less unknown. When the future estimate is based on test results from the structure itself after many years of exposure, many of the input parameters are known and simpler and more reliable methods may be used. Some of the input parameters are additionally most probable interconnected in some way, e.g. surface chloride concentration, diffusion coefficient and critical chloride content [15]. The analysis in this paper is based on the philosophy in EN 1990 [16] according to FORM (First Order Reliability Method) in its simplest form, the so called "mean value method", presented in [16]. The method is briefly presented with focus on the current situation.

# 6.2 Model for estimating service life

The philosophy is based on the European standards EN 1990 [16], where the following definitions are important:

- Failure meaning that a defined event occurs, in this situation that the chloride concentration at the depth of the reinforcement results in a risk of initiating corrosion equal to 10-15 %. This chloride content is called the threshold chloride concentration.
- Environmental action meaning the action or load that the concrete is exposed to, in this situation the depth where the chloride concentration is equal to the threshold chloride concentration.
- Resistance meaning the resistance of the concrete against the action and thereby failure, in this situation defined as the concrete cover.

The risk of failure, Z, is expressed as the difference between a resistance against failure, R, (concrete cover independent of time), and an environmental action, F, (depth where the chloride concentration is equal to the threshold chloride concentration, increasing with time). Both resistance and action are expressed in a statistical way by values for mean and standard

deviation. Probability of failure,  $p_f$ , is defined as the maximum acceptable probability,  $p_{max}$ , depending on safety philosophy. This may be expressed as:

$$p_f = p\{Z = R - F\} < p_{\max} \tag{3}$$

When the functions R and F are normal distributed, also Z will be normal distributed. Results show that normal distribution is accurate enough to make reliable calculations despite that other statistical distributions may fit a bit better to the measurements than the normal distribution [17].

When the resistance R (concrete cover) is normal distributed, the mean is like  $\mu_R$  and the standard deviation is like  $\sigma_R$ , constant with time.

When the action F (depth where the chloride concentration is equal to the threshold chloride concentration) is normal distributed, the mean is like  $\mu_F$  and the standard deviation is like  $\sigma_F$ , both increasing with time.

The probability function for failure, Z, is given by the limit state:

$$Z = R - F \tag{4}$$

When Z is normal distributed, the mean is  $\mu_Z$  and given by:

$$\mu_Z = \mu_R - \mu_F \tag{5}$$

and the standard deviation is  $\sigma_Z$  given by:

$$\sigma_Z = (\sigma_R^2 + \sigma_F^2)^{0.5} \tag{6}$$

The mean  $\mu_Z$  decreases with time and the standard deviation  $\sigma_Z$  increases with time since  $\sigma_F$  increases. This means that the probability for failure increases with time. When Z is normal distributed, the probability of failure may be expressed as:

$$p_f = \Phi(-\frac{\mu_z}{\sigma_z}) = \Phi(-\beta) \tag{7}$$

Where  $\beta$  is called the reliability index. When designing,  $\beta$  shall be greater than a  $\beta_0$  required to fulfil the safety level in a given situation.

The relationship between the probability distribution of failure Z, resistance R and action F is shown in figure 4 [18].

The relationship between the reliability index  $\beta$  and probability of failure  $p_f$  is shown in Table 6 when Z is normal distributed.

Table 6 – Relationship between reliability index, $\beta$ , and probability of failure, $p_{f}$ .								
$p_{\mathrm{f}}$	10 ⁻¹	0.668 10 ⁻¹	0.359 10 ⁻¹	0.227 10-1	10 ⁻²	10 ⁻³	10 ⁻⁷	
β	1.28	1.50	1.80	2.00	2.32	3.09	5.20	

The highest value of  $\beta$  (lowest probability of failure) shall be used when the consequence of failure is high and the reference period for the action is short. Typical examples are accidental situations with many people present.

Servicibility limit state is used when failure lead to economical consequenses only. This is typical for durability situations where the deterioration will be visible long before a risk of collaps is reached.



Figure 4 – Relationship between probability distribution of failure Z, resistance R and action F [18].

Chloride penetration represents a typical situation where probability of risk of failure (defined as risk of initiation of corrosion) should be reduced to an acceptable value. After initiation of corrosion, it may take many years before potential corrosion reduces the servisability or the safety of the structure to such a degree that danger situations develop. Additionally, the reference periode for the action is equal to the service life (increasing chloride content). The consequence of both these factors is that the limiting value  $\beta_0$  may be choosen relatively low, i.e. a relatively high probability for failure.

Based on the philosophy in [16], the probability of initiating corrosion due to chloride ingress is set equal to 10 % (10⁻¹), corresponding to a reliability index  $\beta_0$  equal to approximately 1.30.

The reliability index  $\beta$  may be calculated in different ways according to [16]. In this presentation, FORM (First Order Reliability Method) in its simplest form, the so called "mean value method" is used.

The reliability index  $\beta$  is calculated from:

$$\beta = \frac{\mu_Z}{\sigma_Z} \tag{8}$$

The resistance R is defined as the concrete cover, with mean value  $\mu_R$  and standard deviation  $\sigma_R$ , both constant and independent of time.

The action F is defined as depth where the chloride concentration is equal to the threshold chloride concentration, depending of w/c-ratio, time etc.

At time  $t_0$ , the structure is inspected and chloride profiles are determined. Based on Fick's second law of diffusion,  $C_s$ ,  $D_a$  and  $C_i$  are calculated for each curve according to equation 1.

 $D_a$  has been looked upon as constant over time. That is not correct [19], and this has to be considered when calculating future chloride penetration based on measured chloride profiles and  $D_a$ -values. The development of the diffusion coefficient is in [19] found to be:

$$D_{ati} = D_{at0} \bullet \left(\frac{t_0}{t_i}\right)^{\alpha} \tag{9}$$

Where

D_{ati} is apparent diffusion coefficient at time t_i

D_{at0} is measured apparent diffusion coefficient at time t₀

 $\alpha$  is an aging parameter between 0 and 1, depending on concrete and environment

Threshold chloride content for possible start of corrosion is set equal to  $C_{cr}$  and the time period  $t_i$  it takes  $C_{cr}$  to reach a given depth F may be calculated by equation 10 when equation 9 is included in equation 1 [18].

$$t_{i} = t_{0} \left( \frac{F}{\xi (t_{0} \bullet D_{at0})^{0.5}} \right)^{(\frac{2}{1-\alpha})}$$
(10)

Where

$$\xi = 2 \cdot \operatorname{erfc}^{-1} \left( \frac{C_{cr} - C_i}{C_s - C_i} \right)$$
(11)

erfc⁻¹ is the inverse error function

The resistance R is defined as the concrete cover, with mean value  $\mu_R$  and standard deviation  $\sigma_R$ .

The action F is defined as the depth where the chloride content is equal to the threshold chloride content, depending on w/c-ratio and time. At time  $t_0$ , chloride profiles are determined, giving data for calculating mean  $F_{t0}$  equal to  $\mu_{Ft0}$  and standard deviation equal to  $\sigma_{Ft0}$ , as well as diffusion coefficient  $D_{at0}$  at this time. To find mean  $F_{ti} = \mu_{Fti}$ , at a later time  $t_i$ , a transformed version of equation 10 is used:

$$\mu_{Fti} = \xi \left( \sqrt{t_0 D_{ato}} \right) \bullet \left( \frac{t_i}{t_0} \right)^{\frac{1-\alpha}{2}}$$
(12)

The standard deviation is supposed to be proportional with the mean value (constant coefficient of variation). It may be discussed if this is correct, but it is accurate enough for estimations.

When this method is used to estimate the risk of failure or the service life for an existing structure, it is not necessary to include the dispersion of the w/c-ratio, the chloride consentration at the concrete surface  $C_s$ , the diffusion coefficient nor the original chloride content in the concrete, they are all included in the standard deviation  $\sigma_F$ . This is not quite correct, but accurate enough for such calculations. This means that the number of equations may be reduced to:

Mean value for Z at time  $t_i$  is given by equation 13:

$$\mu_{Zti} = \mu_R - \mu_{Fti} \tag{13}$$

The standard deviation for Z at time  $t_i$  is given by equation 14:

$$\sigma_{Zti}^2 = \sigma_R^2 + \sigma_{Fti}^2 \tag{14}$$

When  $\mu_{zti}$  and  $\sigma_{zti}$  are calculated,  $\beta$  is calculated by equation 8. When  $\beta$  is calculated for the an age equal to the design service life, ist value shows if it is higher or lower than the limiting value  $\beta_0$ . When  $\beta$  is calculated for different ages  $t_i$ , giving  $\beta$ -values on both sides of  $\beta_0$ , the time giving  $\beta$  equal to the limiting value  $\beta_0$  can be found and this is equal to the service life.

#### 6.3 Input parameters in calculations

The main purpose with the following calculations are to find if the requirements in Norwegian Standard for concrete cover is sufficient for 50 and 100 years of exposure when the concrete mix design is equal to the Shore Approach mix design, which is very close to the mix design requirements in the standards today for exposure class XS3.

#### Concrete cover

Requirements in the standard for nominal values are used as mean values and standard deviation giving a minimum cover 10 mm lower than nominal with a 10 % risk of failure.

- 50 years service life:  $\mu_R = 60 \text{ mm}$  and  $\sigma_R = 6.1 \text{ mm}$
- 100 years service life:  $\mu_R = 70 \text{ mm}$  and  $\sigma_R = 6.1 \text{ mm}$

#### Threshold value and depth where chloride concentration is equal to threshold value

A threshold value of 0.4 % by weight of cement is regarded to result in 10 - 15 % risk of failure [20], [21]. For the actual concrete at Shore Approach, this number has to be divided by 5 to be comparable with concrete weight (0.08 % of concrete weight). The chloride curves, see figure 2, crosses the 0.08 %-level of chloride content at different depths. This is used for finding mean and standard deviation for depth where chloride content is equal to threshold value.

- Threshold value  $C_{cr} = 0.4$  % by weight of cement (0.08 % by weight of concrete)
- Mean  $\mu_{F26} = 35.5$  mm and standard deviation  $\sigma_{F26} = 5.5$  mm

#### Apparent diffusion coefficient

The apparent diffusion coefficient is after 26 years found to be  $0.375 \cdot 10^{-12} \text{ m}^2/\text{s}$ . This is a kind of an average coefficient during the 26 years. The instantaneous diffusion coefficient at 26 years has to be lower, however it is not known how much lower it is. The measured apparent coefficient is therefore regarded as a "charactheristic" value on the safe side and included in the calculation as a constant number.

•  $D_{a26} = 0.375 * 10^{-12} \text{ m}^2/\text{s}$  (Equal to 11.83 * mm²/year)

#### Surface chloride concentration and chloride background content

Both theoretical surface chloride concentration,  $C_s$ , and chloride background content,  $C_i$ , are found from the chloride curves. In the calculations, both values are included as constant numbers.

- Theoretical surface chloride content,  $C_s = 0.55$  % by weight of concrete
- Background chloride content,  $C_i$ , = 0.01 % by weight of concrete

#### Aging factor a

The aging factor is found to decrease from around 0.61 to 0.13, see table 5. The scatter in the results are relatively high at both terms. Since the aging factor is important for the service life estimation and its value in the future is uncertain, it is set equal to 0.1 in these calculations without any deviation. This is regarder to be on the safe side.

• Aging factor  $\alpha = 0, 1$ 

#### Estimation of reliability index

The two main parameters i) resistance (concrete cover) and ii) action (depth where chloride concentration is equal to threshold value) are included as probabilistic values. Most of the other input parameters in the calculations are more or less characteristic values. Some parameters are more or less interconnected. This means that the risk of failure should be below 10 %. Based on the presented model for estimating service life (or risk of failure) and the input parameters, the following reliability indexes  $\beta$  are calculated.

- 50 years service life:  $\beta = 4.6 > 1.3$
- 100 years service life:  $\beta = 1.44 > 1.3$

Reliability indexes both for 50 and 100 years service life is well higher than the limiting value 1.3, corresponding to a 10 % risk of failure. This means that requirements given in the present Norwegian Standards for concrete mix design and concrete cover, exposed to exposure class XS3, fulfil the design service lives.

### 7. CONCLUSIONS

The Shore Approach structure is a very important object for calibrating the requirements in the Norwegian Standards with respect to concrete mix design and concrete cover. The current concrete mix design for the structure is very close to the mix design requirements in the standards today for exposure class XS3.

Samples are drilled from the structure at different exposure periods up to 26 years. The results are used in a probabilistic model, according to the philosophy in EN 1990, to calculate if the concrete cover requirements in today's standards in Norway are sufficient.

The results show that planned service lives of 50 and 100 years are exceeded with good margin when using concrete mix design and concrete cover as required in Norwegian Standard for exposure class XS3 today. The margin is highest for 50 years planned service life.

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# **Field Performance versus Laboratory Testing of Freeze-Thaw:** Various Cement and Concrete Qualities



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# ABSTRACT

A test series of 18 concrete mix designs was subjected to initial laboratory testing and ten years of field exposure in de-icing environment. Poor or intermediate laboratory results were not confirmed during the field exposure. The paper questions the high degree of moisture saturation prior to laboratory testing.

**Key words:** Freeze-thaw testing, property documentation, field performance, moisture level.

# 1. INTRODUCTION

# **1.1** The choice of testing concept

The choice of testing regime is of vital importance for the outcome of the laboratory freeze-thaw testing procedure [1, 7]. Principally, the moisture state should agree with the degree of saturation concept introduced by Fagerlund [3], when comparing various material combinations and concrete qualities. For practical testing however, we soon run into practical obstacles, like conditioning time required to obtain the desired state when lowering the w/c-ratio below a certain level – or even the ability to reach this state at all. Additionally for adequate ranking, it becomes of vital importance somehow to consider the moisture state in the field, since this is so crucial to the material's performance. Very often, the non-interrupted capillary suction approach suggested by the degree of saturation concept does not apply to the field conditions. For the field, of course, conditions vary widely with alternating wetting and drying periods of varying intensities. These changes not only affect the degree of saturation, but also modify the pore system [4, 5] and the suction properties of the concrete material. Hence, alternatively to a defined "level-of-state", "performance test" procedures tend to choose a defined "level-of-exposure":

Performance tests on freeze-thaw resistance like the reference method of CEN/TS 12390-9 expose the testing samples to a continuous wet and temperature-cyclic testing regime. The samples are not allowed to "breathe", a fact that leads to "pumping" and accumulation of water within the material structure [1]. Still, the very nature of performance testing is to accelerate the deterioration process in the field. It cannot simulate field conditions completely. Auberg [6] provides a compilation of the principles concerning sample preparation, damage attack and damage assessment for a large number of testing methods. Referring to Powers questioning the ASTM C 666 procedure in this respect already in 1955, he points out that the experience from

testing not necessarily coincides with that of practical experience. Too often, testing methods seem to distinguish between superior and poor only, with no intermediate differentiation. The answer might be various testing methods for various applications. Auberg [6] however, further refers to an ACI committee and concludes that the main question of field correlation is solved by degree of precision and laboratory versus field "calibration". Consequently, not only testing principles and exposure conditions (incl. pre-conditioning) must be discussed, but also acceptance criteria concerning degree of degradation in the laboratory tests – "calibrated" to insitu field observation. Lack of definition and precision concerning "field conditions" of course complicates the issue further.

Still, even if a 1:1 correlation between field performance and accelerated testing is not possible, the regime of moisture exposure – leading to internal moisture accumulation or the opposite - should follow the same principles in the field and in the laboratory. If not, a correlation or "calibration" between the two exposure conditions will not be possible to obtain, at least not adequately "fair" for a range of material qualities.

It is not the objective at this stage to discuss testing principles in details. Without elucidating the issue further, the reference method of CEN/TS 12390-9 [7] (Based on SS 13 72 44 - the "Borås" method) is believed to represent relevant environmental conditions in the following aspects:

- one exposed face and one-dimensional freezing front
- application of de-icing salt
- temperature regime
- deterioration pattern
- useful both for different materials and ways of surface finishing

One concern, however, was to which extent this method could provide fair testing conditions for a range of different material qualities, incl. various w/c-ratios and binders with different timedependent properties' development. Previously, this testing method has been subjected to correlation tests in marine environment [8]. Then, 13 years ago, it was decided to establish another major study on field versus laboratory behaviour for a wide range of concrete mix designs [8, 9, 10]. The new exposure site was at the highway RV 40 between Gothenburg and Borås in Sweden. This chapter describes the concrete mixes manufactured by the present author that formed our part of the field exposure. Reported results include initial laboratory results and some of the field results.

### 1.2 Objective

The objective of the present section is to report and discuss results of the field versus laboratory "performance testing" for such an environment after 10 years of exposure. The study includes a range of concrete mixes made with Norwegian cements and/or "Norwegian" mix design tradition. Other binders and issues related to the project have been reported elsewhere [11, 9].

## 2 EXPERIMENTAL

## 2.1 General

Concrete mixes were manufactured in the laboratory and subjected to the normal curing procedure of SS 13 72 44 for the first month. At this stage, one series of samples from each mix design was subjected to the normal pre-qualification test of the same standard. Mix nos. 1-12 was placed at the field station at the highway at the age of four to five months, i.e. in July 1997. Prior to this, they had been water cured until the age of seven days and then stored in laboratory climate of 20  $^{\circ}$ C and 65 % RH. Mix nos. 13-18 was placed at the site in December 1997, at the age of two months after same type of curing.

# 2.2 Field exposure

The field exposure site constitutes the "saline environment" along the highway RV 40 between Gothenburg and Borås, Sweden. Application of de-icing salt follows standard procedures of that of the Swedish road authorities. The salt exposure is considered relatively severe. The location is displayed in the figures 2.1 & 2.2. The former illustrates the exact placing of the 70 x 150 x 150 mm samples below the road safety barrier. Due to the safety of the traffic and snow clearance, the samples could not be placed closer to the traffic. The samples were tilted 1:20 compared to the horizontal plane.



Figure 2.1: Location of 150 x 150 x 70 mm³ samples at the site. Also larger samples for other purposes like corrosion measurements etc. are located here. Front side faces traffic and is directed towards south. From personal communication with the author of [9].



*Figure 2.2: The site on a winter day. From personal communication with the author of [9].* 



*Figure 2.3: Determination of temperature and relative humidity at site during the winter 1997-98. From personal communication with the author of [9].* 

Data on climatic characteristics are available in the project reports. Utgenannt compiled and reported the temperature and relative humidity at the site, based on automatic measurements. He also reported the number of hours below 0  $^{\circ}$ C and the number of crossings of this temperature to be approximately 1750 and 70, respectively. Both values are for the winter 1997-98. It is assumed that these values are representative for many corresponding locations in Scandinavia, while – as mentioned – the amounts of salt applied is in the upper range.

## **2.3** Testing methods

The standard test SS 13 72 44 was performed in order to rank the mixes according to existing acceptance criteria and to relate the results to the subsequent field performance. Samples for the site exposure comprised four (mix no. 1-12) or two (mix no. 13-18) 150 mm cubes cut in two halves and placed with the sawn surface upwards.

Initially (just before placing at the field station) and after the 1rst, 2nd, 3rd, 4th, 5th, 7th and 10th winter season(s), the samples were subjected to the following procedure :

- Volume determination by weighing in air and water.
- Ultrasonic pulse velocity measurements, reported as the mean value of three separate measurements (different locations) on each of all the samples.
- Visual inspection and classification.

One of the mix designs (no. 3) was – together with some mixes from other parts of the project – subjected to periodic determinations of the capillary degree of saturation during a part of the first winter season. This test was performed on samples drilled from the top and front (towards the traffic) surface of 300 x 300 x 400 mm blocks (not shown in the previous figures). These cores were first weighed, left immersed in water for a fortnight, weighed, dried at 105  $^{\circ}$ C and weighed again. The cores had been cut into sections in order to determine the gradient of the capillary degree of saturation. The concrete had been cast on September 25, 1997, and was placed in field on November 6, 1997. The measurements took place in the period mid February to the end of April, 1998.

The air void characteristics of the fresh concrete have been determined by the "DBT-method" [12]. Additionally, air void characteristics were obtained on hardened concrete in accordance with ASTM C 457, with assistance from an external laboratory.

# 2.4 Mix design and materials

The objective of the mix design matrix was to answer a number of questions. A list of the mixes is provided in table 2.1, and an overview is presented in table 2.2.

The overall objective was to investigate the laboratory behaviour vs. field performance on a set of mix design relevant to the type of field conditions in question. On doing so, the various cement types have been adopted in the design matrix only within their reasonable range of application, i.e. their "design range". This implies e.g. that the use of a fast reacting and finely ground cement at low w/c-ratios – by applying excessive amounts of superplasticizers – is not considered relevant.

The design matrix allows the comparison of two cements (of low water demand) at three different w/c-ratios; 0.35 - 0.40 - 0.45. Both of them are low alkali and designed for this type of application.

The fly ash cement is designed to meet the properties needed for intermediate as well as low strength levels. (The intermediate level is met by the clinker composition of the cement, the lower level by its fineness and concrete surface finishing properties. The fly ash is favourable in

both respects and also is a measure to deal with alkali reactive aggregates.) The fly ash cement is included at two different w/c-ratios; 0.40-0.45, the lower one in this case at the border of its design range (intended use of application).

The cement type and w/c-ratio matrix is partly designed to fit the Swedish binder matrix, reported elsewhere. However, the Norwegian mixes are the only ones to include the w/c-ratio of 0.45, motivated by Norwegian design codes for the environment in question.

Introducing the w/c-ratio of 0.45 also enables a comparison of all the Norwegian manufactured cement types without possible interference of water reducing admixtures interfering with the air void structure. Thus, the "Standard" and rapid hardening ("Industri") cement are added to the matrix at 0.45.

The effect of air entrainment vs. non-AE is investigated at the w/c-ratio of 0.35, but only with the two cements considered relevant for such types of concrete.

Last but not the least, the "academic" matrix above is supplemented with two mixes in an apparently not very systematic approach. Still, mix no. 18 (with 4 % silica fume) meets the standard specifications of the concrete class "SV40" of the Norwegian road authorities [13]. It shall have a maximum w/(c+2s)-ratio of 0.40 and contain 3 - 5 % silica fume. Mix no. 17 (9 % silica fume) is very close to the same design code's "SV30" (w/(c+2s)  $\leq$  0.38 and 8-10 % silica fume) and (except for the silica fume) to that of mix no. 16. Hence, both these mix designs are of considerable practical interest.

In order to compare binder-to-binder effects, mix nos. 1-12 were designed for aggregate grading received for comparison with series beyond the ones presented here. However, this lead to unwanted features : A "quality assurance test" by thin section analyses displayed a number of flaws for five of the mixes. The defects were determined to be caused by bleeding mainly, with increased porosity of the paste along the paste-aggregate interface. This phenomenon was evident for mix nos. 1, 4, 6, 7 and 8, see figure 2.4. It was attributed to the high dosages of superplasticizer that was required to maintain the fresh concrete workability with the given aggregate characteristics and aggregate-binder combinations. Such flaws of increased porosity are weak points and might locally increase the capillary degree of saturation. The possible implication is that more water can be retained in these areas.



Figure 2.4: Example of structural flaws caused by inadequate mix design: Thin section analyses of mix no. 1, exhibiting bleeding (lighter areas) around the coarse aggregate particles at numerous locations. The picture is 3 mm across. From [14].

	Cement type	Trade			Nominal air
Mix no.	Acc. to EN 197-1	name	w/c-ratio	w/(c+2s)	content [%]
1	CEM I 42.5-R-SR-LA	SR	0.35	0.35	-
2	CEM I 42.5-R-SR-LA	SR	0.35	0.35	4.5
3	CEM II/A-V 42.5-R	Std.FA	0.40	0.40	4.5
4	CEM II/A-V 42.5-R	Std.FA	0.45	0.45	4.5
5	CEM I 52.5-R-LA	Anlegg	0.35	0.35	-
6	CEM I 52.5-R-LA	Anlegg	0.35	0.35	4.5
7	CEM I 52.5-R-LA	Anlegg	0.40	0.40	4.5
8	CEM I 52.5-R-LA	Anlegg	0.45	0.45	4.5
9	CEM I 42.5-R-SR-LA	SR	0.40	0.40	4.5
10	CEM I 42.5-R-SR-LA	SR	0.45	0.45	4.5
11	CEM I 42.5-R	Std.	0.45	0.45	4.5
12	CEM I 42.5-RR	Industri	0.45	0.45	4.5
13	CEM I 42.5-R-SR-LA	SR	0.35	0.35	-
14	CEM I 52.5-R-LA	Anlegg	0.35	0.35	-
15	CEM II/A-V 42.5-R	Std.FA	0.45	0.45	4.5
16	CEM I 52.5-R-LA	Anlegg	0.40	0.40	4.5
17	CEM I 52.5-R-LA	Anlegg	0.46	0.39 ¹⁾	4.5
18	CEM II/A-V 42.5-R	Std.FA	0.42	$0.39^{2}$	4.5

Alternatively, they might be the first pores to become saturated when subjected to external suction. In both cases, these locations are potential sites for the initiation of structural damage.

Table 2.1 : Mix design.

¹⁾9 % silica fume (s/c), ²⁾4 % silica fume. See text for objectives.

	w/c-ratio (entrained air content targeted)						
Cement type	0.35	0.35	0.40	0.45	<b>SV</b> ¹⁾		
	(-)	(4.5 %)	(4.5 %)	(4.5 %)	(4.5 %)		
CEM I 42.5-R-SR-LA (SR)	Х	x,y	Х	Х			
CEM I 52.5-R-LA (Anlegg)	Х	x,y	x,y	Х	Х		
CEM II/A-V 42.5-R (Std.FA)			х	x,y	Х		
CEM I 42.5-R (Std.)				х			
CEM I 42.5-RR (Industri)				Х			

Table 2.2 : Overview of mix design.

"y" designates additional, re-designed mix due to reasons explained in the text. ¹⁾Design close to that of specifications by the Norwegian road authorities, see text for explanation.

Hence, it was decided to re-design some of the mixes : Less water demanding aggregate was used and a more industry relevant mix design applied. Plasticizer was applied in order to reduce the amount of superplasticizer. The former contains lignosulphonate, and this admixture (combination) was known to normally interfere less with the air void structure than the use of superplasticizer only. Both measures are in line with Norwegian domestic practice and considered improving the practice-relevance of these mixes (nos.13-16).

## **3** LABORATORY TEST RESULTS

## 3.1 General

Compressive strength values are reported elsewhere and were in line with expected level and meant for indication only. Comments are provided in [1] and are not considered to question the present discussion.

# 3.2 Air void structure

Obtained air void characteristics are included in table 3.1. Spacing factor and specific surface area values determined by the two different methods (See Ch. 2.3) are included. Dubious results are indicated. Comments and plots are provided in [1]; the present paper does not provide space for an adequate discussion.

Mix	Cement type according to FN 197	w/	Nominal air	Total air content obtained [%]		Spacing factor L obtained [mm]	
110.		(C+23)	[%]	Fresh	Hardened	Fresh	Hardened
1 2 9 10	CEM I 42.5- R-SR-LA	0.35 0.35 0.40 0.45	4.5 4.5 4.5	2.4 5.5 5.0 4.9	1.9 3.0 3.8 4.0	1 0.30 0.40 0.26	$\begin{array}{c} 0.30^{2)} \\ 0.33 \\ 0.31 \\ 0.29 \end{array}$
13		0.35	4.5	2.3	1.0	_1)	0.57
5 6 7 8 14 16 17	CEM I 52.5- R-LA (17:+Silica)	$\begin{array}{c} 0.35 \\ 0.35 \\ 0.40 \\ 0.45 \\ 0.35 \\ 0.40 \\ 0.39 \end{array}$	4.5 4.5 4.5 4.5 4.5 4.5 4.5	1.8 5.3 4.8 5.2 2.3 4.8 4.5	2.5 5.5 4.0 3.6 2.9 3.9 4.9	>1 0.36 0.40 0.26 _ ¹⁾ 0.13 0.36	$\begin{array}{c} 0.47^{2)} \\ 0.25 \\ 0.27 \\ 0.27 \\ 0.43 \\ 0.17 \\ 0.35 \end{array}$
3 4 15 18	CEM II/A-V 42.5-R (18:+Silica)	0.40 0.45 0.45 0.39	4.5 4.5 4.5 4.5	5.6 4.3 4.5 4.4	4.8 3.0 4.1 4.2	0.29 0.33 0.24 0.38	0.24 0.31 0.20 0.31
11	CEM I 42.5- R	0.45	4.5	4.4	4.6	0.40	0.30
12	CEM I 42.5- RR	0.45	4.5	5.0	3.9	0.29	0.32

Table 3.1: Obtained air void characteristics.

¹⁾Expected poor value, not considered relevant to measure. ²⁾Not to be trusted. See [1].

### 3 Scaling

The scaling results of the laboratory tests are displayed in the figures 3.1-5. The first one contains all the series made with the CEM I 42.5 R-SR-LA cement, of various w/c-ratios and with/without air entrainment. Mix nos. 1 and 13 are identical except for the choice of aggregate and required admixture dosage, none of them are air entrained. Still, they behaved differently, the latter fulfilling the acceptance criteria of 1 kg/m², the other not. See subsequent sections for discussion and evaluation.

With the CEM I 52.5 R cement, only the air entrained mixes would fulfil any acceptance criteria in the pre-test (figure 3.2).

Except for mix no. 11 (CEM I 42.5 R), all air entrained mixes have scaling levels below 0.5  $kg/m^2$  at 56 cycles.

Several air-entrained mixes exhibit accelerating scaling between 56 and 112 freeze-thaw cycles.



Figure 3.1: Initial scaling test, part 1.



Figure 3.2: Initial scaling test, part 2.



Figure 3.3: Initial scaling test, part 3.



Figure 3.4: Initial scaling test, part 4.



Figure 3.5: Initial scaling test, part 5.

#### 3.4 Scaling versus spacing factor

Freeze-thaw scaling versus air void spacing factor obtained from hardened concrete is plotted in figure 3.6. Equivalently, scaling versus fresh concrete spacing factor is displayed in figure 3.7. See subsequent discussion.



Figure 3.6: Freeze-thaw scaling in initial test versus air void spacing factor determined according to ASTM C457. Note mix nos. of different w/c-ratios (but the same binder) having the same legend in the plot.



Figure 3.7: Freeze-thaw scaling in initial test versus air void spacing factor determined on fresh concrete by the DBT-method [12]. Note mix nos. of different w/c-ratios (but the same binder) having the same legend in the plot.

#### 4 FIELD TEST RESULTS

Plots of ultrasonic pulse velocity, weight, volume and density are included for some of the mixes (figures 4.1-10), exhibiting typical pattern. These exhibit the typical patterns of all the series after ten winter seasons, see subsequent discussion.

The in situ capillary degree of saturation obtained from periodic measurements on a mix identical to mix no. 3 during the first winter is also included in figure 4.11.



Figure 4.1: Ultrasonic pulse velocity (UPV), volume and weight change on field exposed samples, Mix no. 1, values obtained before exposure ("0"), after the first winter ("1") etc. No registration logged after  $6^{th}$ ,  $8^{th}$  and  $9^{th}$  winter.



Figure 4.2: UPV, volume and weight change on field exposed samples, Mix no. 2.



*Figure 4.3: UPV, volume and weight change on field exposed samples, Mix no. 3.* 



Figure 4.4: UPV, volume and weight change on field exposed samples, Mix no. 4.



Figure 4.5: UPV, volume and weight change on field exposed samples, Mix no. 12.



Figure 4.6: UPV, volume and weight change on field exposed samples, Mix no. 13. Similar results with mix no. 14, same mix design with "Anl" cement.



Figure 4.7: UPV, volume and weight change on field exposed samples, Mix no. 15.



Figure 4.8: UPV, volume and weight change on field exposed samples, Mix no. 16.



Figure 4.9: UPV, volume and weight change on field exposed samples, Mix no. 17.


*Figure 4.10: UPV, volume and weight change on field exposed samples, Mix no. 18.* 



Figure 4.11: Capillary degree of saturation of a mix identical to mix 3. Cast and placed during the autumn of 1997. Top surface. Results from the front surface, i.e. facing the road, were of the same order.

## 5 DISCUSSION OF TEST RESULTS

#### 5.1 General

As already stated, strength level results were in line with the expectations and correlating with the air void content. Dubious air void characteristics are indicated above – and the reasoning accounted for in [1]. The same is the case for the discussion of the two different ways of obtaining these values, and the issue is not further elaborated in the present paper.

## 5.2 Initial freeze-thaw test (Laboratory)

## <u>General</u>

Only air entrained concrete mixes performed well in the laboratory test, although mix no. 13 (with w/c-ratio of 0.35, the SR-LA cement and reduced amount of superplasticizer) exhibited a scaling level just below 1 kg.

The mixes re-designed with less water demanding aggregate and reduced amount of superplasticizer improved their performance in the scaling test. This appears to be evident for mix nos. 13 (vs. no. 1) and 14 (vs. no. 5), both non-air entrained. The same tendency is exhibited for the air-entrained mix nos. 15 (vs. no. 4) and 16 (vs. no. 7).

For the air-entrained mixes, this corresponds to the improved air void characteristics of these ("re-designed") mixes. For the non-air-entrained mixes, such a correlation is not evident : Generally, scaling plotted against air void spacing factor does not display a clear correlation. The spacing factor value of 0.30 of mix no. 1 appears peculiar as mentioned above. Using the fresh concrete measurements, the correlation is improved, indicating that the DBT-method may be favourable. We have previously recorded obviously incorrect air void characteristics obtained on (hardened concrete ;) samples with fine cracking. Structural flaws like the ones described above may – although not scientifically verified – lead to similar deviations. However, since investigation of this test method is not the major objective of this paper, we will leave the discussion at this point.

The improved performance after re-designing may not be attributable to the air void characteristics alone. In that case, it is reasonable to assume that less homogeneous structure of the matrix in mix nos. 1, 4, 6, 7 and 8 may have lead to local susceptibility to increased moisture ingress and local deterioration. The fact that all these mixes at some stage exhibit accelerated deterioration may support this. On the other hand, these are not the only mixes with this feature. Still, it would be reasonable to assume that accumulated moisture ingress [1 + many others], when occurring, accounts for accelerated freeze-thaw deterioration. This is one of the major concerns for the correlation between this type of testing and field conditions.

A preliminary conclusion would be that the lab test results vary, depending on the given characteristics including local degree of saturation and – to some extent as shown below – the choice of binder. The latter may be a direct or indirect factor:

## Cement type

The system outlined in table 5.1 may be useful for classifying the scaling results. This system is applied below for the different mix designs, organised according to the w/c-ratio and the laboratory testing performance. Values that are questionable are marked with "(?)".

For w/c-ratio = 0.45, the I-SR-LA and the I-LA cement appear somewhat superior to the other ones. This is not necessarily accompanied by better air void characteristics but higher strength. Still, the other combinations also performed well or fairly well.

When ranking the fly ash cement, it should be kept in mind that the fly ash component is a slower reacting or "maturing" one than the portland clinker. Hence, early age testing of this with respect to properties involving moisture transport may easily become "unfair", compared to field service conditions. The II/A-V (FA) cement mix also improved its performance when the superplasticizer dosage was reduced.

Scaling performance	Mix characteristics	Comments
Classification of scaling	Ref.: Mix no.	
results ¹⁾	Cement type (abbreviation)	
	Mean value $\overline{L}[mm]$	
	<u>Scaling 56 cycles [kg/m²]</u>	
	Strength $F_{c28}$ [MPa]	
	Structural defects/Bleeding ?	

Table 5.1 : Classification of freeze-thaw scaling.

¹⁾The classifications "very good", "good" and "acceptable" are in accordance with suggested classification for testing according to SS 13 72 44. Other classification in these tables is of more qualitative (subjective) nature.

Scaling performance	Mix characteristi	cs	Comments		
Very good	10	8			
	I-SR-LA	I-LA	Both :		
	0.275	0.265	Good strength		
	<u>0.03</u>	<u>0.04</u>	Reasonable $\overline{L}$		
	59	57			
	No	Yes			
Good	15	12	15 :		
	II/A-V (FA)	I-RR (IND)	Medium strength, Good $\overline{L}$		
	0.22	0.305	12:		
	<u>0.17</u>	<u>0.18</u>	Good strength, Dubious $\overline{L}$		
	44	52			
	No	No			
Good/Fair	4	11			
	II/A-V (FA)	I- $R$ (Std.)	Both :		
	0.32	0.35	Good strength,		
	<u>0.34</u>	<u>0.59</u>	Dubious or poor $\overline{L}$		
	50	50	-		
	Yes	No			

Table 5.2 : Classification of scaling test results for mixes of w/c-ratio = 0.45.

For w/c-ratio = 0.40, most of these mixes perform very well (table 5.3), even if the spacing factor is poor (partly). The CEM I-SR-LA and the CEM I-LA cement have a very similar performance. The II/A-V (FA) cement yields slightly higher scaling, but still within what should be classified as a very satisfactory level.

Scaling performance	Mix charac	teristics		Comments
Very good	9	16	7	9&7:
	I-SR-LA	I-LA	I-LA	Good strength,
	0.355	0.15	0.335	Poor L
	<u>0.04</u>	<u>0.03</u>	<u>0.07</u>	16 :
	69	57(?)	70	Good strength,
	No	No	Yes	Good $\overline{L}$
Good	3			
	II/A-V(FA)	)		
	0.265			Good strength, Reasonable $\overline{L}$
	<u>0.2</u>			
	53			
	No			

Table 5.3 : Classification of scaling test results for mixes of w/c-ratio = 0.40.

For w/c-ratio = 0.35, (table 5.4), the difference in laboratory test performance is considerable. All the mixes have a very good strength. The poorly performing mixes comprise both high and low levels of superplasticizer and/or structural defects. The best relation between test performance and characteristic properties is the spacing factor. Hence, these test results seem to confirm that air entrainment is necessary (for laboratory testing) even for the w/c-ratio of 0.35.

At last, for w/(c+2s)-ratio = 0.39 : w/(c+s) = 0.40 - 0.42 : Both of these combinations result in good performance (table 5.5). The I-LA cement plus 9 % silica fume yields somewhat better results than the II/A-V cement plus 4 % silica fume. However, both should be considered to perform at a very satisfactory level, considering the poor spacing factor. It should be considered that silica fume is a fast reacting pozzolanic material that is fully consumed at 28 days. Such early age testing may be a general disfavour of the fly ash cement, but favourable with respect to silica fume.

Scaling performance	Mix charac	teristic	S		Comments
Very good	<i>ood</i> 2 6				
	I-SR-LA		I-LA		Both :
	0.315		0.305	5	Very good strength,
	<u>0.09</u>		0.07		Dubious $\overline{L}$
	79		77		
	No		Yes		
Acceptable	13				
-	I-SR-LA				
	0.57(?)				Very good strength,
	<u>0.9</u>				Poor L
	88				
	No				
Poor	1	5		14	
	I-SR-LA	I-LA		I-LA	All :
	1(?)	1(?)		0.43(?)	Very good strength, Poor $\overline{L}$
	<u>3.4</u>	<u>3.3</u>		<u>2.5</u>	
	87	91		81	
	Yes	No		No	

Table 5.4 : Classification of scaling test results for mixes of w/c-ratio = 0.35.

Scaling performance	e Mix characteristics		Comments	
Good	17(0.40) I-LA + 9 % Silica 0.355 <u>0.14</u> 70 No	18 (0.42) II/A-V (FA) + 4 % Silica 0.345 <u>0.31</u> 55 No	Both : Good strength, Poor L	

Table 5.5 : Classification of scaling test results for mixes of w/(c+2s)-ratio = 0.39.

## Accelerated scaling beyond 56 cycles

Accelerated scaling from 56 to 112 freeze-thaw scaling is observed for the mix nos. 1, 2, 3, 4, 5, 6, 7, 8 (minor), 9, 11 (minor) and 12. This includes all the mixes investigated on this point, except for mix no. 10. It is difficult to attribute this to any particular property. Still, mix no. 10 (and no. 8) was made with 2 kg of superplasticizer only. If high amounts of superplasticizer are negative – without affecting the spacing factor – this might be attributed to changes of the capillary pore structure (not investigated).

The mix nos. 13 - 18 were not tested beyond 56 cycles. However, the mix nos. 13 & 14 exhibit acceleration from 28 to 56 cycles, mix nos. 15 - 18 not. The four latter mixes have higher water/binder-ratios. They also have less and partly other water reducing admixture combinations.

Not much is known about the reasons for such behaviour. Petersson [15] connects this "accelerating" phenomenon to dense concrete containing silica fume, which is not relevant here. However, the reasons for this behaviour in our present case remain unexplained but are clearly a negative property built into the acceptance criteria of SS 13 72 44. Fagerlund et al [16] reports breakdown of aged, self-desiccated high performance concrete subjected to long term water storage, but the time scale and exposure type is different from our case, the explanation probably also. Setzer explains accumulation by ice lens formation [17, 18].

However, it is reasonable to believe that the moisture accumulation in the pore system with prolonged freeze-thaw "pumping" exposure is a central factor for the accelerated behaviour, see review in section 7 of [1]. Considering the in-field moisture values above, the central issue for laboratory versus field performance becomes the way of moisture exposure prior to as well as during freeze-thaw exposure.

## 5.3 Field test

## Visual inspection:

Independent of the lab scaling results, all mixes had none or only very little visible surface damages after ten (!) winters of field exposure (classification "1" on a scale from 0 to 5) [U 2007]. No cracks were reported. Note that this corresponds to the classification of the best reference concrete mixes from the same exposure site (not yet reported).

## UPV:

The UPV-values are not very accurate due to (lack of) resolution of the equipment and reproducibility in measuring [19]. This means that values should change at least 2-4 % before being considered significant. Still, during the first years of assessment, it was considered somewhat suspicious that all the mix nos. 1-5 (and no. 14) have decreasing tendencies from the first to the end of the second winter. However, frost deterioration should result in much higher UPV loss, see section 4 in [1].

Theoretically, changing appearance ("up & down") could indicate temporary "healing" of any possible damages occurring, a phenomenon demonstrated in laboratory tests by Jacobsen [20]. In the present case, it is more likely to assume variation caused by different degree of moisture level in the samples at the time of recording, even if the samples have been subjected to water storage prior to UPV determination. Several features support this assumption :

The UPV variation proceeds in a very parallel way (time dependence) for several mixes, although to a different degree. Also, in no case, the fall in UPV value coincides with any loss in density, i.e. no indication of volume increase due to cracking. Still, comparing of UPV changes between certain series may support or contradict this hypothesis in different ways :

Comparing series nos. 1 & 2, differing in air void content, could support an idea of UPV value changes promoted by increased moisture diffusion facilitated by the air voids. However, the same pattern can not be found for mix nos. 5 & 6 (results not included in this paper). Hence, this hypothesis fails. Neither may the differences between series nos. 1 & 2 be attributed to larger diffusivity caused by the structural flaws, since this was a characteristic of mix no. 1 : Mix no. 2 was the one (of these two) exhibiting the largest UPV variation.

Still, UPV changes caused by moisture exchange should be related to w/c ratio – all other factors remaining constant: There is a very week indication amongst the rest results to support this idea, comparing mix nos. 3 & 4. Again, this is not supported when comparing mix nos. 6, 7 & 8 (graphs not included in the present paper) – or no. 9 versus that of no. 2. The comparable UPV results are all identical.

One feature remains, however, that all the re-designed mixes (improved mix design) exhibit higher UPV values during their "lifetime": This is valid for mix nos. 13 vs. 1 (SR/0.45/Nat.air), 14 vs. 5 (Anl/0.35/Nat.air), 15 vs. 4 (StdFA/0.45/4.5% Air) and 16 vs. 7 (Anl/0.40/4.5% Air). This is consistent also with the improvement in the initial scaling test results. Thus, it is reasonable to expect that the re-design actually improved the capillary structure. The first implication is the effect on the initial scaling test results. The second implication is that the UPV measurements were capable of detecting this, even if the structural changes were not severe enough to have impact on the visual rating in field.

The overall UPV picture is "up and down" until four years of age, then strong increase until five or seven years, then a strong drop at ten years.

The preliminary conclusion is that UPV measurements within the detected range (increase of 10 % from the initial stage and back to "bias") may well depend on material dependent parameters, but primarily on direct and indirect influence on the moisture state. The latter is a non-homogeneous "property" throughout the sample. The UPV changes could not be attributed to cracking.

Volume changes

Change in sample volume may be due to swelling/shrinkage or deterioration mechanisms, e.g. internal cracking or surface scaling

Mix nos. 1-12 exhibit small volume increase during the first year. This might be an indication of internal cracking. However, it does not correspond to the slightly negative UPV changes, taking place during the second winter, commented on above. During the second winter, the volume stays stable or decreases very slightly. The decrease is hardly connected to surface damage (stable surface rating). Hence, the former volume increase is most likely attributable to swelling during the first period, since the samples were rather dry at the time of placing at the field station.

Mix nos. 13-18 exhibits a slight volume decrease during the first winter, thereafter it remains stable. Since surface defects are close to zero, this indicates slight shrinkage, in spite of the weight increase mentioned below.

Only during the period of five to ten years of age, a minor (0.5 % or less) volume decrease is detected. Again, no relation to UPV.

## Weight changes

The weight changes from one winter to the next vary between the series, but are (from recalculation) all in the range 0 - 10 % (at the very most) degree of capillary saturation.

Mix nos. 1-12 exhibit a slight weight loss during the first winter, then it increases again during the second period. This is in contrast to swelling as an explanation of the volume increases above. It might indicate cracking as volume increase mechanism. However, low weight after the first winter might also be due to inadequate suction prior to weighing, i.e. the "saturated, surface dry"-condition (SSD) not being equally defined in the two cases. This may not necessarily be due to operator procedure, but may be caused by slower suction properties for the aged concrete.

Mix nos. 13-18 exhibit slight weight increase during both periods of field exposure. This seems evident in all these cases, in spite of the slight volume decrease/shrinkage mentioned above. The values are so small that the weight increase may be less than (re-filling of) hydrated and physically bound water during the period. These mixes were subjected to exposure at a later stage of the same year as mix nos. 1-12 and may have experienced less drying prior to the winter season.

Some of the series exhibit slight weight decrease from five to seven years of age, corresponding to similar volume change but - in some cases at constant density, in some not. This indicates a somewhat harsher exposure in this period, but it is not possible to relate this "performance" to any particular mix design parameter.

- - -

In other series than mix nos. 1-18, with more or less pronounced damage [19], the major part of these damages was localised at the bottom and lower parts of the samples. A possible implication is that the bottom of the samples is subjected to longer periods of suction. This

results in a higher degree of saturation - and therefore scaling and cracking. The upper parts of the sample are more exposed to evaporation/drying and shrinkage.

## Density changes

All mixes exhibit very slight density increase from seven to 10 years, indicating on-going hydration (increase in chemically bound water) and/or internal deposits. The former may contribute to increased density against moisture ingress and reduced degree of capillary saturation. Still, this has not been investigated. Reduced UPV value (increase in transmission time) might be a result (as actually measured).

## Degree of saturation

The figure 4.11 displays the degree of capillary saturation of the top surface and the front (road facing) surface, respectively. The accuracy of the single measurements is probably in the range of 3-5 % (0.03-0.05 on the plot scale).

Compared to a few other mixes also tested [21], the mix reported here (identical to mix no. 3, but one year younger) was in the lower range. However, mixes of equal w/c-ratio exhibited values at a similar level.

The most important result at this stage is probably the level of the degree of capillary saturation - and the profile of the sample: All values are at a level of 0.87 or below. Even more important may be that all the profiles appear to be at a "dry" or drying state. This is supported by Relling [22] who found that the moisture condition even of bridge piers in Norwegian, coastal climate was not affected below a few cm from the surface. The "drying state" coincides with the weight loss attained for the companion mix during this (first) winter. This leads to a concentration profile that increases from the surface inwards.

These figures were confirmed on companion chloride exposure samples at the same location after ten years [23]: Degree of capillary saturation from 0.79 - 0.65 at the surface to 0.75 - 0.60 at the interior. The ranking of mix design was not possible. RH measurements yielded values from 0.91 - 0.85 (surface) to 0.92 - 0.82 (interior) with "sensible" ranking.

## Preliminary conclusion:

A possible implication is that a high degree of saturation in field service is not possible, unless concrete is very permeable due to a high w/c-ratio or an open structure – or unless the surface is badly drained and covered with water for long periods. If this assumption is correct, internal cracking and/or scaling caused by high degree of saturation during long term laboratory testing is not representative for field service conditions. As mentioned earlier, there is also some evidence that high strength concrete without air performs better in the field than in lab tests.

## 6 CONCLUSIONS

The initial laboratory investigation on concrete provided valuable characterisation and information on laboratory performance. However, its direct application for the intended mix design ranking is questionable. There is no good correlation between the laboratory results ranking and field performance after ten years – for these relatively good or intermediate quality levels. This is probably due to the excessive degree of saturation, compared to field conditions

(depending on application area, of course). Increased discrepancy may be expected for slowly reacting binders, due to increased suction caused by chemical shrinkage during the preconditioning stage (fixed by time).

The following findings and indications have been made:

## Mix design / Air void system

- There seems to have been a considerable drop in total air content from fresh to hardened state for mixes with high amounts of superplasticizer.
- There are indications that other changes in air void characteristics from fresh to hardened state occur as well. Such changes may depend on fresh concrete properties (determined by mix design) or be due to difference in testing principles (See [1]).

## Laboratory freeze-thaw testing / Mix design ranking :

- Only air-entrained concrete performed well in the laboratory test, even with w/c-ratio of 0.35. Overall, a mean (of fresh and hardened ...) spacing factor in the range of 0.4 or less appears vital to ensure good performance in the laboratory freeze-thaw scaling test procedure.
- The I-SR-LA and I-LA cements appears somewhat superior to the other cements (w/c-ratio 0.40 or 0.45). However, the others also performed well, especially with an appropriate air void structure. The fly ash cement exhibited somewhat higher scaling, probably disfavoured by the young age of testing.
- Silica fume (4 %) had no distinct effect with the II/A-V (FA) cement at w/(c+s) = 0.42, its scaling level being close to those of the 0.40 and 0.45 mixes with the same cement. This may also be a "maturity" phenomenon not of the silica itself, but indirectly by affecting the reaction rate of fly ash.
- Silica fume (8 %) with the I-LA cement appears to be positive, but this may also be due to a simultaneous decrease of the spacing factor.
- High dosages of superplasticizer lead to structural defects in the form of bleeding pockets under aggregate particles and less homogeneous mortar/paste quality. This impaired the laboratory test results.

## Field exposure :

- All mixes had none or only very small visible surface damages after ten winters of field exposure. No cracks were evident. This applied whether the initial laboratory test of the specific mix design gave good results or not.
- The results of UPV, volume and weight measurements on the field samples were slightly mixed. However, this may be due to the very small magnitude of the changes. The significance of UPV in a pre-cracking stage is, however, questionable, and should be noted. The linking of weight, volume and density values may be useful, although the later is a property derived form the other two.
- The profile of the capillary degree of saturation exhibited values from 85 % and below. This should be considered with respect to choice of the test conditions of the testing methods.
- Damages (of other series) were found at the bottom and lower parts of the samples. This implies that the lower parts are subjected to longer periods of suction and a

higher degree of saturation. The upper parts of the sample are more exposed to evaporation/drying and shrinkage.

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# Field and Laboratory Testing and Service Life Modelling in Finland









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## ABSTRACT

Extensive data on the true weathering rate of concrete is needed to determine the correlation between field tests, laboratory tests and concrete mix designs. A wide range of concretes with different cements and binding materials are needed for these studies. Studies on integrated degradation must be included. All this data together with local weather data serves as a basis for thorough modelling, computer simulation and development of comprehensive and calibrated service life models. These all-out models can be used as tools in the development of more practical tools for engineering purposes. The current ongoing DuraInt-project aims at serving this objective.

**Key words:** Concrete, durability, field testing, frost-salt, frost, chloride penetration, carbonation, ageing, interacted deterioration, models, service life.

## 1 INTRODUCTION

In Finland there are today a total of three concrete durability field testing areas under evaluation. The first two field stations include concretes where frost and carbonation are evaluation, while the third station includes concrete samples for assessing frost-salt deterioration, carbonation and chloride penetration. The first station was established within the EU 5th Framework project "CONLIFE: Life-time Prediction of High-Performance Concrete with Respect to Durability" (2001-2004) and is located in northern Finland in the town of Sodankylä. In the parallel national project (YMPBETONI 2002 - 2004) with Finnish ecological binding materials, an additional station was established in southern Finland in the neighbourhood of Otaniemi, in the town of Espoo [1]. The most recent Finnish project (2008-2011) is entitled "Effect of Interacted Deterioration Parameters on Service Life of Concrete Structures in Cold Environments (DuraInt)". In the DuraInt project, one more testing area has been established in southern Finland, beside Highway 7 near the town of Kotka. The former so called Durafield-project [2, 3] is today included as one task in this more versatile DuraInt-project.

In addition to field and laboratory testing, DuraInt includes laboratory testing, theoretical studies and also service life modelling on the effect of interacted deterioration parameters. [4] In the DuraInt-project VTT works in co-operation with TKK (Helsinki University of Technology) and with foreign partners from Norway, U.S.A, Canada and Portugal. DuraInt is a public project funded by TEKES (the Finnish Funding Agency for Technology and Innovation) and also by several participants including organizations, companies, and cities.

The main strategy in Finland is to use modelling and computer simulation to treat such complex phenomena. This means accounting for simultaneous frost or salt-frost deterioration, chloride penetration and carbonation of different concrete structures. The simulations also need to consider different binding materials, concrete mix designs and different or constantly changing climatic conditions. Computer simulation is calibrated with field and laboratory testing. By the use of calibrated simulation, service life models for engineering purposes can be developed, i.e. most of the parameters in the factor method design formulas of the Finnish national codes can be defined. 'Interaction parameters' will be included in updated models developed in the project to take into account the effect of interacted deterioration.

## 2 FIELD AND LABORATORY TESTING

## 2.1 Testing field information

There are a total of three different concrete durability field testing areas in Finland. Two of these are in southern Finland and one in northern Finland. The main information on these field stations and the testing extent is summarized in Table 1. Some field station photos are presented in Figure 1. An example of temperature and relative humidity variation (Otaniemi field station, Espoo, autumn 2007 – autumn 2008) is presented in Figure 2.

Information, research field	Highway 7,	Otaniemi,	Sodankylä
Erost XF3	KOIKä	Espoo V	v
Frost-salt (de-icing) – XF4	X	-	-
Carbonation:	71		
- sheltered outdoors – XC3	-	Х	-
- not sheltered outdoors – XC4	$(X)^{1)}$	X	$(X)^{1)}$
Chloride penetration, road environment - XD3 (mainly 4.5 m from road line)	X	-	-
Projects and mix designs to date:			
- DuraInt (2007)	27	27	-
- CONLIFE (2001)	-	-	22
- YMPBETONI (2001)	-	19	19
Own meteorological station	Х	Х	-
Concrete temperature and humidity/RH measurements	Х	-	-
Location	N 67°22´ E26°39´	N60°11´ 24°48´	N67°24´ E26°35´
Elevation from sea level [m]	about 20	about 20	179
	for Helsinki,	Kaisaniemi	
Temperature information (1971-2000):	(elevatio	n 4 m):	Sodankylä:
-Year average [°C]	+6	<u>,</u>	-1
- January average[°C]	-4		-14
- June average [°C]	+1.	5	+12
Days in a year, when minimum temperature is below 0°C	16	9	230
Days in a year, when minimum temperature is below -10 °C	40		111
Relative humidity, year average [%]	79	)	62
Precipitation, year average [mm]	642	507	

Table 1 – Basic field testing area information.

1) No specific specimen for monitoring, but can be measured later on.



Figure 1 – Field testing areas in southern Finland beside Highway 7 near Kotka and in Otaniemi, Espoo.



*Figure 2 - Temperature and relative humidity variation at Otaniemi testing field in southern Finland autumn 2007 – spring 2008.* 

#### 2.1 Concrete mixes and general testing plans

#### DuraInt – Basic series

The mix designs of the first series ("Basic") of the DuraInt-project were performed with the Finnish concrete industry and represent mainly prevailing common industrial mixes (Table 2). About half of the castings were done in a ready-mixed concrete plant or in a pre-cast element factory. Of the 27 mixes, 23 were produced in autumn 2007 and then four additional mixes were made with a slight adjustment to the air entrainment dosage in spring 2008. Common Finnish cements (manufactured by Finnsementti Oy), blast furnace slag (BFS) and fly ash (FA) were used. The effective water-to-binder ratio, as defined in Table 2, was about 0.42, 0.50 or 0.60 and the compressive strengths were up to 60 MPa. Field testing concentrates on air entrained bridge concretes. Some concretes were intentionally produced with no or only inadequate air entrainment. In addition, 6 air entrained façade or balcony concretes are included. The DuraInt project includes a wide laboratory testing program, i.e. basic properties as well as salt-frost, frost, carbonation and chloride diffusion testing and thin section studies (Table 5). The suitable testing field and testing extent for each concrete mix was defined according to the range of use, e.g. façade concretes are not tested for chloride penetration. All the results in the future will be publicly available for anyone interested and the results will be preserved in a documentation database for decades.

#### DuraInt – Interacted deterioration

In autumn 2008 further laboratory test series will be cast for interacted deterioration studies. General information on the testing plan is presented in Table 5 as well as more general mix design information. Some mixes in this research are the same as in the DuraInt Basic series (Table 2). This also means that there will be field testing data for these mixes, and comparison of the results and verification of the models will be possible. E.g. results for the interaction of carbonation and salt-frost resistance can be compared with field testing results. Studies on the interacted deterioration are in the early phase. General mix design information and testing plans are presented in Table 6.

#### CONLIFE

In the CONLIFE-project all 22 mix designs were >60 MPa high performance concretes (HPC) with water-binder ratios of 0.30 - 0.42. The cement was Danish Type CEM I 52.5R cement from Aalborg Portland. Air entrained and non-air entrained high strength concretes with silica fume (SF), normal or fine blast furnace slag (BFS) or fly ash (FA) were included. Mixtures for

lab and field testing were cast and exposed in autumn 2001. Besides the Finnish field station in Sodankylä, CONLIFE-mixtures have also been exposed in 8 other marine and frost testing fields in Germany, Italy, Sweden and Iceland. The CONLIFE-project also included studies on acid attack, cyclic temperature attack, shrinkage and fire resistance, but these themes are not considered within this paper. Only the laboratory testing results corresponding to Finnish field test observations are presented here. The CONLIFE mix design information is presented in Table 3 and general information on testing in Table 5. [5]

### **YMPBETONI**

The YMPBETONI-project concretes were made with Finnish cements 'Yleis' (CEM II/A-M(S-LL) 42.5) and 'Rapid' (CEM II/A-LL 42.5 R) and they included up to 60 % fly ash (FA) or 70 % blast furnace slag (BFS). These ecological 30 – 45 MPa air entrained concretes were cured with or without heat treatment. The mix designs were based on the target strength for precast and ready mix concrete. Mixes were cast in autumn 2003 and were exposed at both southern and northern Finland field stations. The YMPBETONI mix design information for all 19 mixes is presented in Table 4 and general information on testing in Table 5. The results have been reported in Finnish literature through 2005 testing but will be further assessed within the DuraInt project.

DuraInt Short code	Cement type	W _{eff} /(Cement +2*SF+0.6*BFS+ 0.4*FA)	Cement [kg/m ³ ]	BFS [kg/m ³ ]	FA [kg/m ³ ]	Aggregate total [kg/m ³ ]	Total eff. water [kg/m ³ ]	Fresh concrete air [%]	Compressive strength 28 d (150 mm cube) [MPa]
1A	CEM II/B-S 42.5 N	0.41	405	0	0	1746	165	6.5	56.3
2A	CEM I 42.5 N - SR	0.42	387	0	0	1796	161	5.9	46.4
3A	CEM II/A-M(S-LL) 42.5 N	0.42	428	0	0	1709	179	5.9	38.0
5A	CEM II/A-LL 42.5 R	0.42	421	0	0	1748	176	5.0	41.2
6A	CEM I 52.5 R	0.42	417	0	0	1737	175	5.5	58.5
7A	CEM II/A-LL 42.5 R & Finnsementti SLG KJ400	0.47 3)	217	217	0	1725	163	6.1	46.0
8A	CEM II/A-LL 42.5 R & FA [EN 450-1. 2005] Fineness N. Class A	0.45	344	0	106	1706	173	5.0	54.6
3Ba	CEM II/A-M(S-LL) 42.5 N	0.42	410	0	0	1844	172	$2.6^{(1)}$	50.6
3Bb	CEM II/A-M(S-LL) 42.5 N	0.42	406	0	0	1784	170	4.5 ²⁾	46.1
3Bc	CEM II/A-M(S-LL) 42.5 N	0.42	407	0	0	1756	172	5.3 ²⁾	48.9
3Bc2	CEM II/A-M(S-LL) 42.5 N	0.42	406	0	0	1715	170	$7.0^{2}$	39.0
3Bd-SCC1	CEM II/A-M(S-LL) 42.5 N	0.42	435	0	0	1746	185	3.4 ¹⁾	51.0
3Be-SCC2	CEM II/A-M(S-LL) 42.5 N	0.42	426	0	0	1709	178	5.7	35.0
1C	CEM II/B-S 42.5 N	0.47	339	0	0	1808	160	6.9	45.5
3C	CEM II/A-M(S-LL) 42.5 N	0.49	333	0	0	1847	163	5.5	40.1
4C	CEM I 52.5 N	0.46	334	0	0	1816	154	5.5	46.3
5C	CEM II/A-LL 42.5 R	0.51	337	0	0	1833	172	5.0	44.9
6C	CEM I 52.5 R	0.40	451	0	0	1722	180	5.4	50.3
3D	CEM II/A-M(S-LL) 42.5 N	0.50	333	0	0	1895	166	3.4	44.5
1E	CEM II/B-S 42.5 N	0.60	273	0	0	1845	163	7.3	32.8
3E	CEM II/A-M(S-LL) 42.5 N	0.58	321	0	0	1828	185	4.3	35.5
4E	CEM I 52.5 N	0.54	300	0	0	1840	162	5.5	39.5
5E	CEM II/A-LL 42.5 R	0.54	322	0	0	1764	174	4.8	41.2
3Bf	CEM II/A-M(S-LL) 42.5 N	0.41	420	0	0	1812	174	1.7	64.0
3Bg	CEM II/A-M(S-LL) 42.5 N	0.41	420	0	0	1745	174	4.2	57.2
3Bh	CEM II/A-M(S-LL) 42.5 N	0.41	420	0	0	1691	174	6.8	46.3
3Bi	CEM II/A-M(S-LL) 42.5 N	0.41	420	0	0	1746	174	4.9	52.8

*Table 2 - DuraInt. Basic series for field and laboratory testing. Mix information and compressive strength 28 d.* 

1) No air entrainment; 2) Presumably extra compaction pores; 3)  $W_{eff}/(Cement + 2*SF + 0.8*BFS + 0.4*FA) = 0.42$ 

CONLIFE Short code	Code Meaning	Cement type	W _{eff} /(Cement +2*SF +0.8*BFS +0.4*FA)	Cement [kg/m ³ ]	BFS [kg/m ³ ]	fine BFS [kg/m ³ ]	FA [kg/m ³ ]	SF [kg/m ³ ]	Water [kg/m ³ ]	Aggregate total [kg/m ³ ]	Fresh concrete air [%]	Compressive strength (150 mm cubes) [MPa]
C21	0.30	CEM I 52.5 R	0.29	511	0	0	0	0	146	1859	2.0	81
C22x	0.42	CEM I 52.5 R	0.39	365	0	0	0	0	144	2096	5.5	78
C8	0.30 + 3%SF	CEM I 52.5 R	0.28	491	0	0	0	15	144	1960	2.0	87
C1	0.30 + 7% SF	CEM I 52.5 R	0.27	467	0	0	0	33	144	1960	2.0	98
C9	0.30 + 10% SF	CEM I 52.5 R	0.27	451	0	0	0	45	144	1960	2.5	98
C 2	0.35 + 7% SF	CEM I 52.5 R	0.32	401	0	0	0	28	145	2029	1.7	90
C10x	0.42 + 3% SF+AE	CEM I 52.5 R	0.39	351	0	0	0	11	146	1995	5.1	82
C3	0.42 + 7% SF	CEM I 52.5 R	0.38	334	0	0	0	23	146	2097	1.7	92
C11x	0.42 + 10% SF+AE	CEM I 52.5 R	0.38	322	0	0	0	32	146	1995	5.3	90
C4	0.30 + 7% fineBFS	CEM I 52.5 R	0.27	476	0	33	0	0	144	1960	1.8	90
C 5	0.30 + 7% SF + 30% BFS	CEM I 52.5 R	0.29	361	108	0	0	25	144	1960	2.2	99
C 6	0.42 + 7% fineBFS	CEM I 52.5 R	0.38	340	0	24	0	0	146	1995	1.2	84
C7x	0.42 + 7% SF + 30% BFS + AE	CEM I 52.5 R	0.41	258	77	0	0	18	146	1995	5.6	89
C12	0.30 + 7% SF + 10% FA	CEM I 52.5 R	0.29	416	0	0	42	29	144	1960	1.8	97
C13	0.30 + 7% SF + 20% FA	CEM I 52.5 R	0.36	313	0	0	125	22	145	1960	1.8	87
C14	0.30 + 7% SF + 40% FA	CEM I 52.5 R	0.32	375	0	0	75	26	145	1960	2.9	89
C15x	0.42 + 7% SF + 10% FA + AE	CEM I 52.5 R	0.42	297	0	0	30	21	146	1995	4.5	83
C16x	0.42 + 7% SF + 20% FA + AE	CEM I 52.5 R	0.45	268	0	0	54	19	146	1995	5.2	81
C17x	0.42 + 7% SF + 40% FA + AE	CEM I 52.5 R	0.50	223	0	0	89	16	146	1995	4.7	72
C18x	0.30+7%SF+AE	CEM I 52.5 R	0.27	467	0	0	0	33	144	1858	6.5	78
C19x	0.35+7%SF+AE	CEM I 52.5 R	0.32	401	0	0	0	28	146	1927	4.9	71
C20x	0.42+7%SF+AE	CEM I 52.5 R	0.38	334	0	0	0	23	145	1994	4.8	67

 Table 3 - CONLIFE mix design information and compressive strength 28 d.

Table 4 - YMPBETONI mix design information.

YMPBETONI Short code ¹⁾	Code Meaning	Cement type	W _{eff} /(Cement +2*SF +0.8*BFS +0.4*FA)	Cement [kg/m ³ ]	BFS [kg/m ³ ]	FA [kg/m ³ ]	Water [kg/m ³ ]	Aggregate total [kg/m ³ ]	Fresh concrete air [%]	Compressive strength (100 mm cubes/calculated 150 mm cubes) [MPa]
K30	K30 - Reference	CEM II/A-LL 42.5 R	0.58	250	0	0	144	1907	6.0	30/30
L20	K30 - 20% FA	CEM II/A-LL 42.5 R & FA [EN 450-1. 2005] Fineness N. Class A	0.55	231	0	58	139	1839	6.8	37/38
L40	K30 - 40% FA	CEM II/A-LL 42.5 R & FA [EN 450-1. 2005] Fineness N. Class A	0.52	208	0	139	136	1781	6.0	44/47
L60	K30 - 60% FA	CEM II/A-LL 42.5 R & FA [EN 450-1. 2005] Fineness N. Class A	0.47	173	0	259	129	1677	5.7	39/41
K45	K45 - Reference	CEM II/A-M(S-LL) 42.5 N	0.49	343	0	0	167	1786	5.0	43/46
M25	K45 - 25% BFS	CEM II/A-M(S-LL) 42.5 N & Finnsementti SLG KJ400	0.52	253	84	0	165	1748	6.9	37/38
M50	K45 - 50% BFS	CEM II/A-M(S-LL) 42.5 N & Finnsementti SLG KJ400	0.55	170	170	0	167	1755	6.2	36/37
M70	K45 - 70% BFS	CEM II/A-M(S-LL) 42.5 N & Finnsementti SLG KJ400	0.57	102	239	0	168	1761	5.6	33/34
K30U	K30-Ref NEW	CEM II/A-LL 42.5 R	0.60	279	0	0	168	1829	5.6	30/30
L40U	K30-40%FA NEW	CEM II/A-LL 42.5 R & FA [EN 450-1. 2005] Fineness N. Class A	0.62	208	0	139	163	1715	6.3	24/23
L60U	K30-60%FA NEW	CEM II/A-LL 42.5 R & FA [EN 450-1. 2005] Fineness N. Class A	0.57	172	0	257	156	1603	6.5	25/24

1) K means reference (only CEM II) with target strength; L means FA; M means BFS; U means new casting.

Table 5 – Information on field and laboratory testing in DuraInt Basic-, CONLIFE- and YMPBETONI–projects in Finland. (The general scheme for DuraInt Interacted deterioration studies are presented in Table 6).

	Number of mix designs/cases in					
	DuraInt Basic	CONLIFE	YMPBETONI			
	<u>Basic series</u> (field + laboratory), Interacted studies see Table 6	(Testing in different countries, different ages and test details as test duration and minimum temperature, see [5])	(Includes mixes with and without heat treatment and 3 re- done mixes)			
Testing scheme	(2007)	(2001)	(2002)			
Laboratory testing:						
- Fresh concrete basic properties	27	22	19			
- Compressive strength (always 28 d, for CONLIFE and YMBETONI more ages)	27	22	19			
- Thin section studies, air pore analysis, etc.	27	22	19			
- Carbonation: Cabinet: 1% CO ₂ ; RH 60%; T=21°C - Carbonation: RH 65 %, T=20 °C	23 23	-	- 19			
- Chloride diffusion coefficient - D _{nssm} (NT Build 492, CTH-method, different ages, e.g. 28 d, 3 months, 6 months)	12 +6 ¹⁾	22	-			
- Frost-salt: scaling and internal, slab test	21	-	-			
- Frost: scaling and internal, slab test	6	22	19			
Field testing:		-				
- Carbonation sheltered	23	-	-			
- Carbonation not sheltered	$(27^{2})$	_	19			
- Chloride penetration profiles	$12 + 6^{1)} + 3^{3)}$	(22 ⁴⁾ )				
- Frost-salt: volume change and internal deterioration	21	$(22^{4})$				
- Frost: volume (weight) change and internal deterioration	6	22 ^{5) 6)}	19			
- Frost healing at field	5	22	19 ⁷⁾			
- Optical microstructure (e.g. thin sections) and other further studies, as needed	27	22	19 ⁷⁾			

1) Effect of impregnation or form lining

2) No specific specimens, can be measured later on

3) Three more distances from road lining

4) Testing not in Finland but in Sweden and Iceland

5) Testing also in Iceland

6) Only weight change is monitored (=scaling+water uptake/drying)

7) Both in southern and northern Finland (Otaniemi and Sodankylä)

		(2000)	
Laboratory testing on interacted deterioration:	Number of mixes	r Mix information	Target
Carbonation – Frost-salt	17	Table 2, mixes:1A, 2A, 3A, 5A, 6A, 7A, 8A, 3Bb-e, 1C, 3C, 5C, 6C	- evaluate the effect of carbonation and ageing (also without carbonation) on salt- frost scaling
Frost-salt – Carbonation	7	Table 2, mixes: 3A, 3Bb, 3Bc2, 3C, 5A, 7A and 8A	- evaluate the effect of salt-frost scaling/deterioration on carbonation depth
Carbonation – Frost	10	Table 2, mixes: 1E, 3E, 4C, 4E, 5C, 5E and 4 new mixes w/c=0.60 and air 2 %, 3 % 5 % & 6 %; CEM II/A-M(S-LL) 42.5 N	- evaluate the effect of carbonation and ageing (without carbonation) on possible frost scaling and internal frost deterioration
Frost – Carbonation (internal)	6	6 mixes with w/c = 0.60 and air 2 %, 3 % & 5%; two binder materials: CEM I 42.5 N + BFS (50 %)	- evaluate the effect of internal cracking on carbonation
Carbonation – Chloride	5	5 mixes with $w/c = 0.50$ and air 2% and 1 with air 5%; 4 cements/binder materials (CEM L	- evaluate chloride diffusion coefficient in carbonated concrete and the effect of carbonation on chloride distribution, i.e. on chloride profiles
Chloride – Carbonation	5	CEM II, BFS 50 % and FA 24 %)	- evaluate the effect of chlorides on carbonation
Frost – Chloride (internal)	2	2 mixes with w/c = 0.50 and air 2 % & 3 %; CEM II/A-M(S-LL) 42.5 N	- evaluate the effect of cracking (internal) on chloride penetration
Chloride - Frost	2	e.g. 2 mixes with w/c = 0.50 and air 2 % & 3 %; CEM II/A-M(S-LL) 42.5 N	- evaluate the effect of chlorides on frost deterioration

 Table 6. – Information on mix design and testing in DuraInt Interacted deterioration studies.

 (2008)

## 3 **RESULTS**

## 3.1 Carbonation - XC3

#### DuraInt

Carbonation of all the DuraInt basic series mixes (Table 2, not for mixes 3Bf-i)) is studied at the field stations on samples sheltered from rain (XC3). Carbonation has been tested also in the laboratory in an accelerated procedure of - 56 days, 1%  $CO_2$ , T= 20 °C and RH 60 %. Before testing curing was 7 days in water and 21 days in RH 65 %. Carbonation is also tested in the laboratory in a non-accelerated environment of T=20 °C and RH 65 %. The samples have been exposed for 1 year already and exposure is continuing.

Carbonation depth was essentially measured as presented in prEN 13295 (2003) [19]: e.g. for each measurement one or two slices of 15 mm minimum thickness were taken from the prism (originally  $100 \times 100 \times 500 \text{ mm}^3$ ) and sprayed with phenolphthalein indicator solution. The average

value for each concrete was calculated based on single measurements (40 measurements for accelerated carbonation and 20 for a preliminary value of not-accelerated carbonation).

The results so far are presented in Table 7. In Figure 3 the calculated k-value (carbonation depth  $= k t^{\frac{1}{2}}$ ) for carbonation at 1 % CO₂ is compared with the values calculated for carbonation at RH 65 % and at the sheltered field location. In this calculation the carbonation time (days) is the time at the actual carbonation circumstance and the k-value is calculated considering the exact carbonation time separately for each concrete.

In the future, carbonation at both the field station and in the laboratory RH 65 % environment will be followed and measured. The next measurement time will be after 2 years of exposure time, in autumn 2009.

Mix	56 d at 1 % CO ₂ [mm]	RH 65 % (7.79.0 months; average 8.3 months) [mm]	Field sheltered about 270 d (Sept. 07- May 08) [mm]
1A	3.6	0.7	0.2
2A	3.6	0.7	0.3
3A	5.5	1.3	0.5
5A	5.0	1.1	0.4
6A	2.6	0.4	0.2
7A	5.7	1.9	0.7
8A	4.7	1.3	0.4
3Ba	4.7	1.2	0.3
3Bb	4.8	1.3	0.2
3Bc	4.5	1.2	0.2
3Bc2	5.6	1.5	0.4
3Bd-SCC1	4.5	1.1	0.2
3Be-SCC2	8.0	3.0	0.6
1C	5.9	1.8	0.5
3C	6.1	1.8	0.5
4C	2.7	0.5	0.3
5C	5.4	1.7	0.3
6C	4.0	0.6	0.1
3D	5.2	1.4	0.4
1E	10.5	3.7	2.2
3E	7.1	2.4	1.4
4E	4.3	1.4	0.7
5E	6.4	1.8	0.6

Table 7 – DuraInt. Carbonation depth measurement results ( $1 \% CO_2$ , RH 65 % and field sheltered).



Figure 3 – Correlation between k-values as calculated for carbonation at 1 % CO₂ (x-axle) and at RH 65 % and at field sheltered. Preliminary results after < 1 year.

### 3.2 Chloride penetration - XD3

#### DuraInt-project

Chloride penetration studies are made for samples at the DuraInt testing field site beside Highway 7 near Kotka. The results represent surfaces directly affected by de-icing salts or spray containing de-icing salts (XD3).

Only one winter period has passed so far and the results are thus somewhat preliminary. Chloride profiles were made from the surfaces facing the road by the so-called "profile grinding method" (on cores of 100 mm diameter). The main distance from the road lining is 4.5 m. There are also some results from samples placed at distances of 9 m, 11 m and 13 m from the road line. Total chloride content was determined, using one powder sample with one measurement per depth.

In the laboratory the non-steady state diffusion/migration coefficient ( $D_{nssm}$ ) was measured, with testing at the age of 3 months following the CTH-method, NT Build 492 [20].For these measurements  $\phi$ 98×200 mm cylinders were cast (3 parallel). The actual test specimens were prepared by first cutting the cylinder into two halves, and then cutting a 50 ± 2 mm thick slice from one half. The testing was done by Germann Instruments A/S device called PROOVE'it. An external electrical potential is applied axially across the specimen and forces the chloride ions outside to migrate into the specimen. After a certain test duration (normally after 24 hours), the specimen is axially split and a silver nitrate solution is sprayed on to one of the freshly split sections. The chloride penetration depth can then be measured from the visible white silver chloride precipitation, after which the chloride migration coefficient can be calculated from this penetration depth. Some results are presented in Figures 4 - 6.

From the results in Figure 4, it can be seen that the surface concentration after the first winter season varies between 0.03 - 0.07 w.-% and the maximum concentration is usually higher than the surface concentration. All mixes had a chloride penetration depth of less than 10 mm. These results after only the first year of field testing are not closely analyzed here. It is expected that after a few more years the results will be more stable and then closer analysis will be warranted. As an example of results, for concrete 7A (CEM II/A-LL 42.5 R & 50 % BFS) the surface

concentration is highest (0.067 w.-%), but the concentration at 5 mm depth is lowest (0.021 w.-%).

In Figure 5 the expected trend could be seen, as the depth of chloride penetration clearly increased as samples were moved closer to the roadway edge.

The study on the effect of hydrophobic impregnation is included in the DuraInt-project as well as the study on the effect of the use of form lining (3 + 3 cases/products, studies with concrete 3D, see Table 2). According to the laboratory tests, i.e. values for D_{nssm} at 3 months with mould surface in the testing by NT Build 492 method [20], and preliminary field testing results, both the use of form lining and hydrophobic impregnation clearly diminished chloride penetration (Figure 6).



Figure 4 – Chloride profiles after the first winter period and respective chloride diffusion coefficients measured at 90 day (NT Build 492). See Table 2 for mix information.



Figure 5 – Chloride contents in concrete specimen (3A and 5A) at different distances from road side (4.5 - 1 3meters). Total chloride content at 1.5 mm, 3 mm and 5 mm depth. of sample.



Figure 6 – The effect of hydrophobic impregnation (IMP) and the use of form lining (FL) on chloride penetration and diffusion coefficient (NT Build 492, form surface at test). A) Chloride diffusion coefficient at 90 d. B) Cl-content at field specimen (depth 5 mm) after the first winter period v.s.  $D_{nssm}$  at 90 days.

### 3.3 Freeze-thaw without de-icing salts - XF3

#### General

Field testing for freeze-thaw in Finland usually corresponds to exposure with high water saturation and horizontal surfaces (XF4). A typical Finnish winter experiences about 50 freeze-thaw cycles. In CONLIFE the testing field in northern Finland also represents a very low minimum temperature (see below), yet the number of actual freeze-thaw cycles is similar to southern Finland.

The so called Slab test is one test method for freeze-thaw and internal damage in CEN/TC 51 N 772 (2003) [21]. This method is mainly used here with the addition of scaling material being measured according to CEN/TS 12390-9 [22]. Internal damage or cracking is measured by ultrasound pulse velocity and a measure of internal damage is calculated as relative dynamic modulus percentage (RDM). Mixtures start with an RDM value of 100% and the concrete is considered deteriorated when the RDM falls below a certain level as defined by acceptance criteria. A typical 'failure' value may be an RDM falling below 85% or 67%, depending on the standard requirements [11].

### DuraInt

There are only 6 concretes in DuraInt for freeze-thaw testing with plain water (4C, 5C, 1E, 3E, 4E and 5E). These concretes have been tested in the laboratory using the slab test and they have been in the Otaniemi, Espoo testing field since autumn 2007. Internal deterioration and healing has been followed by ultrasonic transit time and fundamental frequency measurements. Both measurements have been calculated to provide the assessment of internal damage expressed as relative dynamic modulus (RDM %). Measurements of the specimen (75 x 150 x 150 mm³) are always made after 1 day in water and 7 days in RH 65%. Volume change is also monitored by weighing in water and air and always after 1 day in water immersion. Later on also thin section studies will be made to evaluate the internal structure, e.g. possible cracking.

Laboratory testing results including thin section results on air entrainment and results for the first field testing period 2007 - 08 are presented in Table 8. Compared to an initial RDM value of 100%, only minor changes in RDM can be seen after the first winter period noted as spring 2008. This first winter was considered to be extremely mild for Finland. Some strength gain or healing can bee seen after the first summer period. One concrete (5E) scaled during the lab testing. The next measurements will be done after the winter period 2008 - 09 and again after the summer. These field measurements are planned to continue for 10+ years.

	Laboratory testing				Field testing						
	Thin sections Air pores		Slab test 56 cycles		Internal deterioration						
Mix	Specific surface < 0.800 mm pores [mm ² /mm ³ ]	Spacing factor (< 0.800 mm pores) [mm]	Scaling [kg/m ² ]	$\begin{array}{c} \text{RDM} \\ 100 \ (t_0/t_n)^2 \\ [\%] \end{array}$	RDM by ultrasound $100 (t_0/t_n)^2 [\%]$		RDM by fundamental frequency $(f_n/f_0)^2$ 100 [%]		Volume change (+ is grow) [%]		
					spring 08	autumn 08	spring 08	autumn 08	spring 08	autumn 08	
4C	21	0.29	0.028	110	95.6	97.4	97.0	98.2	0.16	0.37	
5C	13	0.51	0.020	107	98.4	101.5	99.7	101.4	-0.03	-0.41	
1E	23	0.26	0.013	106	101.0	102.8	98.3	103.3	-0.45	-0.35	
3E	25	0.28	0.013	102	98.2	100.8	98.8	100.7	-0.03	0.21	
4E	25	0.25	0.023	107	96.5	98.2	97.6	99.2	0.06	0.31	
5E	16	0.41	0.259	101	97.6	99.0	99.3	100.7	0.02	0.35	

Table 8 - DuraInt-project. Results for frost deterioration, after 1 year. Slab test results and field testing results after the first winter period 2007 – 08 and subsequent summer.

## CONLIFE

Field testing of frost deterioration and also healing has been evaluated 8 times so far at the Sodankylä station during from 14.12.2001 - 15.9.2004. The criteria for a freeze-thaw cycle as defined by the project group was an exterior air temperature change, rising over +1 °C followed by dropping below -1 °C. There were 42 freeze-thaw cycles measured during the first (2001-02) winter, with the coldest temperature reaching -36°C. During the second (2002-03) season there were an additional 56 cycles and the coldest daily temperature recorded was -39°C.

The internal damage (RDM %, by fundamental frequency) measured on all concretes after two winter seasons is given in Figure 7.

Mixture C3 (w/b=0.42, 7% SF) was the only mixture that had severely deteriorated beyond the acceptance level of 80%. Mixture C6 (w/b=0.42, 7% special slag) also showed some damage but not beyond the unacceptable limit due to summer healing. Note that both of these mixtures had the higher w/b ratios and were non-air entrained. In all 22 concrete mixtures, the most severe deterioration occurred during the first winter (measured in June 2002 after placement in December 2001), followed by summer healing. The healing was significant, as expected, since all of the mixtures contained secondary binder materials that benefit from prolonged curing time to gain strength through further hydration.

The results after two winters of exposure showed heavy surface and internal cracking in both mixtures C3 and C6. The cracking did not show typical frost cracking, which would be cracks running parallel to the outer surface. Upon internal evaluation, it was seen that the cracks mainly ran along the bond areas of aggregate particles and in paste connecting the aggregates. No scaling was detected, but there was a high water uptake. A good correlation was found between the field and laboratory tests in this case (see Table 9). All the results can be found in CONLIFE deliverable reports [5].

Field measurements of the CONLIFE-concretes in Finland will continue and the next measurement time is scheduled for spring 2009. It will be interesting to see the future field performance as there were several mixes that performed poorly and failed during the lab testing; e.g. RDM % after 112 cycles with plain water or after 56 cycles with NaCl-solution (See Table 9).

	P	Plain water (	112 cycles	NaCl solution (56 cycles)			
Mix	Scaling [kg/m²]	$\begin{array}{c} \text{RDM} \\ \text{by ultrasound} \\ 100 \ (t_0/t_n)^2 \\ [\%] \end{array}$	Moisture Uptake [kg/m ² ]	Scaling [kg/m²]	RDM by funda- mental frequency $(f_n/f_0)^2$ 100 [%]	$\begin{array}{c} \text{RDM} \\ \text{by ultrasound} \\ 100 \left( t_0/t_n \right)^2 \\ [\%] \end{array}$	Moisture Uptake [kg/m²]
C21	0.004	51	1.353	0.067	_1)	130	1.763
C22x	0.010	106	0.163	0.054	-	104	0.657
C8	0.003	64	1.263	0.056	-	131	1.530
C1	0.014	53	1.848	0.138	10	-	1.975
C9	0.012	6	1.723	0.054	-	132	1.607
C2	0.016	38	2.250	0.520	2	-	2.415
C10x	0.006	107	0.140	0.061	-	101	1.113
C3	0.037	14	2.438	1.568	2	-	2.830
C11x	0.012	48	1.380	0.137	-	104	1.340
C4	0.027	29	2.410	0.043	94	-	0.765
C5	0.023	35	2.238	0.075	96	-	0.588
C6	0.032	50	1.883	1.013	2	-	2.343
C7x	0.005	92	1.190	0.165	102	-	0.410
C12	0.008	112	0.558	0.483	4	-	2.503
C13	0.013	113	0.565	0.718	2	-	2.625
C14	0.016	32	2.243	1.000	2	-	2.768
C15x	0.007	106	0.708	0.193	77	-	1.025
C16x	0.010	109	0.830	0.133	94	-	0.735
C17x	0.008	108	1.103	0.293	68	-	1.278
C18x	0.006	99	0.223	0.118	100	-	0.315
C19x	0.015	104	0.448	0.100	95	-	0.620
C20x	0.012	85	1.733	0.255	15	-	1.690

Table 9 - CONLIFE-project. Laboratory testing results for freeze-thaw (112 cycles) and also frost-salt testing (56 cycles), though there is no field testing for frost-salt.

1). Frost-salt measurements of internal damage were by either fundamental frequency or ultrasound. Non-measured specimens are marked with a dash (-).



Figure 7 – CONLIFE-project. Internal damage measured by frequency after 2 winters, at Sodankylä field station. Severe damage noticed for C3 (w/b=0.42, 7% SF) and C6 (w/b=0.42, 7% special slag). [5] See Table 3 for mix information and Table 8 for laboratory testing results.

## **YMPBETONI**

Almost all the tested concrete grades displayed excellent frost resistance properties in the slab test after 56 cycles, with respect to both surface weathering  $(0.008 - 0.019 \text{ kg/m}^2)$ , average 0.013 kg/m²) and internal damage (RDM = 95.2 - 101.7 %, average 98.6 %). No mixtures came close to the critical acceptance levels for frost damage. This was somewhat as expected, since all the mixtures were air entrained.

The field tests conducted over one winter season showed also very little surface and internal damage in the tested concrete grades. A trend was seen that the more fly ash or slag that was included, the more damage was detected. Heat treatment of samples during initial curing slightly improved the results (Figure 8). The first winter encompassed approximately 50 freeze-thaw cycles (south 52; north 43). Again a cycle was defined in this project as an exterior air temperature change, rising over +1 °C followed by dropping below -1 °C. At the southern station in Otaniemi, the weather is moister than in the northern Sodankylä location. At the same time temperature typically stays clearly below zero during the winter and the cycles are concentrated in late autumn and early spring. Some healing was noticed during the summer, which was detected as an increase in the RDM-value. Field testing results after the first winter and summer (2003-04) are presented in Figures 8 and 9. [1, 6, 7, 8] Field testing will continue and the next measurement is scheduled for spring 2009.



*Figure 8 - YMPBETONI. Field testing results for frost deterioration (internal) after 1st winter period at southern (Otaniemi) and northern (Sodankylä) Finland. ) K means reference (only CEM II); L means FA; M means BFS; C means heat treatment; U means new casting. [1, 6]* 



Figure 9 - YMPBETONI. Field testing results for frost deterioration (internal) after 1st winter period, first summer period and 2nd winter period in southern Finland (Otaniemi, Espoo). Healing or strength gain after 1st summer period can be seen (measurement Sept. 04). K means reference (only CEM II); L means FA; M means BFS; C means heat treatment; U means new casting. [1, 6]

## 3.4 Freeze-thaw with de-icing salts - XF4

## General

Field testing for freeze-thaw with salt exposure in Finland corresponds XF4, i.e. high water saturation with de-icing agent. This was only evaluated at a field station in the newest DuraInt project. Evaluation of frost-salt resistance in field tests of the CONLIFE project mixtures were evaluated by other partners in Sweden, Germany and Italy and are therefore not included in the scope of this paper. The test results for CONLIFE are reported in [5].

In the DuraInt project, information on the amount of de-icing salt spread every winter period is collected. The project also includes weather data collection and measurements of concrete temperature, relative humidity and water content at different season periods. Optical fibres are used to measure the concrete temperature and water content profiles. Frost-salt deterioration is followed by measuring volume change of 75x75x150x mm³ specimen. Internal deterioration is also measured in the same was as or the freeze-thaw specimen without de-icing salts (see above chapter 3.3). Microstructural alteration will be studied in the future as needed to supplement other results.

The so called Slab test is used here for scaling with salt solution (CEN/TS 12390-9) [22]. Internal damage is also measured by ultrasound (CEN/TC 51 N 772 (2003)) [21].

### Results

Laboratory and field testing results to date are presented in Table 10. After the first and very mild winter period of 2007-08, no visible scaling or cracking was detected. Perhaps the only possible sign of deterioration was a small volume increase in the poor quality concretes, meaning those with inadequate air entrainment. This volume increase had some correlation with lab testing results, i.e. scaling in the slab test after 56 cycles (see Table 10).

The field specimen for frost-salt damage will be measured again in spring 2009. In the future at least the mixes with inadequate or poor air entrainment are expected to show signs of damage at the field station along Highway 7. One series, mixtures 3Ba - Bi (10 mixes with the same cement, CEM II/A-M(S-LL) 42.5 N,) was specifically designed for studying the effect of air content and air pore quality on salt-frost deterioration and thus interesting and informative results are expected. So far laboratory results demonstrate the importance of proper air entrainment in normal bridge concretes with w/c 0.42 (see Table 10).

		Laboratory	testing	Field testing			
	Thin sec	tions	Slab test 56 cycles		Internal	deterioration	Volume change (+ is grow) [%]
Short code	Air pores. specific surface < 0.800 mm pores [mm ² /mm ³ ]	Air pores. spacing factor (< 0.800 mm pores) [mm]	Scaling [kg/m ² ]	$\begin{array}{c} \text{RDM} \\ \text{by ultrasound} \\ 100 \left(t_0/t_n\right)^2 \\ [\%] \end{array}$	$\begin{array}{c} \text{RDM} \\ \text{by} \\ \text{ultrasound} \\ 100 \left( t_0/t_n \right)^2 \\ [\%] \end{array}$	RDM by fundamental frequency $100 (f_n/f_0)^2 [\%]$	Volume change (+ is grow) [%]
					spring 08	spring 08	spring 08
1A	21	0.46	0.195	104	102.5	102.2	0.20
2A	16	0.35	0.045	110	102.3	102.0	0.26
3A	21	0.28	0.202	107	102.0	101.3	0.42
5A	28	0.24	0.205	109	104.2	101.7	0.31
6A	34	0.33	0.075	104	105.5	101.4	0.18
7A	37	0.18	0.320	99	101.6	101.3	0.27
8A	27	0.30	0.158	105	102.5	101.4	0.40
3Ba	14 (no air entr.)	1.15	3.500 1)	<86, no value, too much scaling	101.9	101.9	0.59
3Bb	23	0.51	1.750 1)	no value, too much scaling	102.1	101.6	0.73
3Bc	22	0.38	0.840 1)	no value, too much scaling	102.1	101.9	0.50
3Bc2	19	0.30	0.096	103	103.2	102.0	0.30
3Bd-SCC1	11 (no air entr.)	0.69	1.400 1)	no value, too much scaling	103.1	101.9	0.55
3Be-SCC2	12	0.34	0.128	99	103.9	102.8	0.54
1C	27	0.22	0.039	102	101.9	100.1	0.13
3C	21	0.28	0.084	103	100.8	101.2	0.34
5C	13	0.51	0.636	105	101.8	99.3	0.35
6C	23	0.29	0.390	101	101.0	100.1	0.38
3Bf	12 (no air entr.)	0.98	2.740 1)	<70, no value, too much scaling			
3Bg	23	0.41	0.496	101	to field tes	sting spring 2008.	, no results so far
3Bh	24	0.31	0.236	105		0 I 8 1000	,
3Bi	16	0.44	0.305	104			

Table 10 - DuraInt-project. Results after 1 year for freeze-thaw with de-icing salts. Slab test results and field testing results after the first winter period 2007 - 08.

1) This value is an extrapolation. Last measurement was after 42 cycles. The test was interrupted because of too much scaling causing leakage.

## 4 INTEGRATED DEGRADATION AND SERVICE LIFE MODELS

### **4.1 Principles**

Structures exposed to outdoor climate are usually subject to several types of degradation mechanisms simultaneously. If, for example, a concrete structure is exposed to normal Nordic climate, it is inevitably attacked by both carbonation and frost. The single degradation mechanisms, such as carbonation, chloride penetration and frost attack, are relatively well understood. There are also fairly good models for such degradation when treated separately. However, the interaction of these mechanisms is not well understood and there are hardly any

models for such combined effects. However, to be able to evaluate the service life of concrete structures understanding on combined effects of degradation mechanisms is necessary. Laboratory testing is needed to get data and verify deterioration models with interaction. [4]

Figure 10 shows a general scheme for the development of integrated degradation models. The process starts from tests in the laboratory and in the field. Laboratory tests are conducted both as 'single' and as 'coupled'. Based on these laboratory tests and theoretical/analytic reasoning, degradation models for 'single' and 'coupled' degradation mechanisms are developed.

As a first step in the theoretical reasoning process, simple time-related model functions on degradation are developed. As a second step computer simulation can be used for profound understanding of the combined effects of degradation. In computer simulation the 'first step' model functions are used but as differential approximations. That means that the changes in temperature and moisture conditions and the effects of other degradation mechanisms can be re-evaluated in every time step (typically 1 hour) according to the real situation in climatic conditions (local meteorological data) and the real progress of other degradation mechanisms. Computer simulation in this case refers to the following:

1. theoretical emulation of ambient climatic conditions,

2. determination of the temperature and moisture variations in a cross-section of a concrete structure, and

3. application of temperature and moisture sensitive degradation models so that the degradation over time and the service life can be predicted.

Computer simulation must be calibrated with tests results from both the laboratory and field. Calibration means that the simulation results of degradation are made fit with the experimental test results by changing the parameters of simulation models.



*Figure 10 - Development of integrated degradation and service life models. [4]* 

#### 4.2 Analytic models

#### Carbonation

Depth of carbonation is approximately proportional to the square root of time. The theoretical reasoning is not presented here; see [4].

$$X_{Carb} = k_{Carb} \sqrt{t} \tag{1}$$

where  $k_{Carb}$  is coefficient of carbonation [ $mm/\sqrt{a}$ ].

The values of coefficient of carbonation can be found experimentally.

Chloride penetration

The depth of the critical chloride content also approximately complies with the 'square-root-of-time' relationship in the same way as the depth of carbonation [4]:

$$X_{Cl} = k_{cl^*} \sqrt{t} \tag{2}$$

where

 $k_{cl^{\ast}}$ 

t

is coefficient of chloride penetration  $[mm/\sqrt{a}]$ , and is time [a].

The Bazant-simplification offers a mathematically easy way to treat the problem of chloride ingress. The coefficient of chloride penetration depends on the type of cement and it can be found experimentally. From Equations (1) and (2) follows that in both cases the following differential equation can be applied (Fagerlund, 1994 [4]):

$$\Delta X_{c} \approx \frac{1}{2} \cdot k_{c} \cdot t^{-\frac{1}{2}} \cdot \Delta t = \frac{1}{2} \cdot \frac{k_{c}^{2}}{X_{c}} \cdot \Delta t$$
(3)

where

 $X_c$ is depassivative depth (either carbonation depth or the depth of<br/>critical chloride content) [mm], and<br/>kc $k_c$ is coefficient of depassivation (either carbonation or the chloride<br/>penetration) [ $mm/\sqrt{a}$ ], and<br/>ttis time [a].

Both the carbonation depth and the depth of critical chloride content can be also modelled with two parameters as follows:

$$X(t) = A \cdot t^{B}, \tag{4}$$

where

X is the depth of carbonation or critical chloride content [mm], and A, B are parameters depending on the climatic conditions, material properties and possible structural measures.

In that case the differential approximation during  $\Delta t$  would be:

$$\Delta X_{c} \approx B \cdot A \cdot t^{B-1} \Delta t = B \cdot A^{\frac{1}{B}} X^{1-\frac{1}{B}} \cdot \Delta t, \qquad (5)$$

### Frost attack and frost-salt scaling

Modelling principles for frost attack and salt-frost scaling are presented e.g. in [4] and they are not presented here.

Frost attack is caused by freezing of water inside concrete. To model frost damage, the number of critical freezing events, degree of water saturation in natural weathering conditions and degree of degradation must be specified. The service life is ended when the degree of damage reaches the maximum allowable value (e.g. 30 %).

Frost-salt scaling is caused by freezing on a concrete surface when it is in contact with a chloride solution. The depth of scaling is related to the number of freeze-thaw cycles in the surface of concrete. An accelerating rate of scaling is usually a sign of poor frost resistance of concrete. A retarding scaling rate is a sign of inferior quality of concrete on the surface compared to the quality inside. In homogeneous concrete an even scaling rate is expected.

#### Service life based on frost-salt scaling

A simple formula was developed for predicting service life based on frost-salt scaling [13] (Vesikari, 1991). It separates the material factors from environmental factors. All material factors are incorporated in the P-value:

$$\mu(t_L) = k_e \cdot P, \tag{6}$$

 $\begin{array}{ccc} \text{where} & \mu(t_L) & \text{is the service life [a],} \\ & k_e & \text{is the circumstantial factor [-], and} \\ & P & \text{is P-value [-].} \end{array}$ 

According to the Finnish concrete code, service life can be determined using the following values of circumstantial factor [11] (BY50 2004):

$$XF2: \quad k_e = 2$$
$$XF4: \quad k_e = 1.25$$

P-value is determined from the following equations [11, 18]:

$$P = \frac{46 \cdot c_{cur} \cdot c_b}{\frac{10 \cdot (WAB)^{1.2}}{\sqrt{a}} - 1},$$
(7)

$$c_{cur} = 0.85 + 0.17 \cdot LOG_{10}(t_{cur}), \tag{8}$$

$$c_{b} = 1 - \left(\frac{Q_{h2o}}{Q_{b}}\right)^{1.5} \cdot \left(0.05 \cdot SF + 0.02 \cdot BFS + 0.01 \cdot FA\right), \tag{9}$$

$$Q_b = Q_{cem} + 2.0 \cdot Q_{sf} + 0.8 \cdot Q_{bfs} + 0.4 \cdot Q_{fa}, \qquad (10)$$

$$WAB = \frac{Q_{h2o} + 10 \cdot (a - 2)}{Q_{h}},$$
(11)
where	Р	is P-value [ - ],
	c _{cur}	is the curing factor [ - ],
	c _b	is the binding factor [ - ],
	WAB	is the reduced water-air-binder ratio [-],
	a	is the air content [%],
	t _{cur}	is the curing time [d],
	$Q_{h2o}$	is the effective water content $[kg/m^3]$ ,
	$\mathbf{Q}_{\mathbf{b}}$	is the total amount of effective binding material $[kg/m^3]$ ,
	SF	is the silica fume ratio [%],
	BFS	is the blast-furnace slag ratio [%],
	FA	is the fly ash ratio [%],
	Q _{cem}	is the cement content $[kg/m^3]$ ,
	$Q_{sf}$	is the silica fume content $[kg/m^3]$ ,
	$Q_{bfs}$	is the blast-furnace slag content $[kg/m^3]$ , and
	O _{fa}	is the fly ash content $[kg/m^3]$ .

The model is mainly based on empirical results and rests on the above mentioned concrete technological parameters known to affect frost durability. It is also used by the Finnish Road Authorities to predict service life [18] (Finnish Road Administration, 2008).

### 4.3 Future service life models with 'interaction parameters'

Computer simulation offers a possibility to better manage the effects of constantly changing climatic conditions and simultaneous degradation processes. Computer simulation utilizes theoretical/analytic degradation models. However they are applied as differential approximations in order to take into account the momentary condition and exposure stresses and the momentary progress of various degradation mechanisms in the structure.

There are theoretical methods of coupling degradation mechanisms. Examples of interaction include influences such as frost action likely accelerating the processes of carbonation and chloride penetration in concrete by increasing the permeability of concrete. Also frost-salt attack likely accelerates the penetration of carbonation or critical chloride content by removing material from the surface of concrete. [4]

### 4.4 Service life models in the Finnish national codes

### Simple models

The Finnish national codes presents simple models for service life design of concrete structures based on carbonation and frost attack [11]. The design life span is calculated by Equation (12):

	$t_L = t_{Lr}$	$\cdot \mathbf{A} \cdot \mathbf{B} \cdot \mathbf{C} \cdot \mathbf{D} \cdot \mathbf{E} \cdot \mathbf{F} \cdot \mathbf{G},$	(12)
where	t _L t _{Lr} A to G	is design life span [a], is comparison life span of 50 years [a], and are coefficients taking into consideration affecting the design life span.	nd all of the variables

The design parameters affecting the life span with respect to frost attack are presented in Table 11.

Coefficient	Affecting feature	Design parameters
А	Material properties, porosity	Water-cement ratio, air content
В	Design, structural features	Structural member, possible surface treatment
С	Workmanship	Curing measures
D	Internal climate	-
E	External climate	Freeze-thaw class, geographical direction and location
F	Design loads	-
G	Maintenance	Inspection and service sequence

Table 11 - Coefficients affecting the freeze-thaw life span of a concrete structure.

Every coefficient, A to G, can be determined from a respective table. As an example the first coefficient A can be selected as a function of the maximum aggregate size and concrete air content measured from fresh concrete.

In the case of life span assessment for carbonation, a similar formula as Eq. (12) is used and the coefficients A to G are obtained in a similar manner.

The service life design according to the Finnish national codes so far does not present any model for structures in a chloride environment and the interaction between degradation modes is not considered satisfactorily. In DuraInt-project these matters are under development.

### Computer simulation in specification of factors of service life

After calibration with field and laboratory tests, computer simulation can be used for quantification of factors in the service life models with respect to various degradation mechanisms. Considering the models in the Finnish national codes (Eq. (12)), the simulation method is especially suitable for quantification of factors A, B, D and E.

The simulation program of VTT allows a possibility for easy quantification of the environmental factor E. The climatic data have been gathered from different observation sites so that the factors for coastal Finland, Middle Finland and Northern Finland can be determined using actual weather parameters in these regions. The weather models are based on the data gathered by the Finnish Meteorological Institute and they consist of data on temperature, relative humidity, velocity and direction of wind, amount of rain and intensity of solar radiation.

The effects of other degradation modes on the service life can be studied using the interaction parameters in the degradation models. As the service life can be determined with and without parallel degradation modes the "interaction factors" can be determined.

The incremental time in the computer simulation is normally one hour. The total calculation may cover some months, some years or even some hundreds of years from the lifetime of the structure although the time of calculation by itself takes only some hours.

### 5.0 CONCLUSIONS

Over the past 10 years, two major projects have been completed in Finland and a third is underway where field stations have been established to obtain real-time data on concrete durability. Three field stations exist and have numerous samples under evaluation: two for frost resistance both in southern and northern Finland, one for frost-salt attack and chloride exposure, and all three stations can include carbonation evaluation. The goal of the projects has been to evaluate the correlations between laboratory and field testing, with the aim of improving the service life modelling of concrete.

The tested concretes have included a range of mixture designs and binding materials, including blast furnace slag, fly ash and silica fume. Both air entrained and non-air entrained mixtures have been tested in the lab and exposed in field studies in hopes of seeing a wide range of durability performance. Additional concrete will still be produced in the current DuraInt project, which is scheduled to be completed in 2011.

The results obtained in these projects will be summarized and maintained in a public database, to be used for future durability modelling and improvement of service life prediction codes. The existing Finnish codes for service life account for carbonation and frost attack, yet more data will help improve the models. It is important in future development to account for the interaction of durability factors like frost, carbonation, and chloride, which is the main goal of the newest DuraInt project. The existing Finnish codes do not account for concretes subjected to chloride and therefore need improvement.

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# 14 years experience with Øresund Link concrete



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### ABSTRACT

Nine concretes with different mix designs have been stored for 14 years at DTI's exposure site. The mix designs used were all allowed for the Øresund Link project. Generally all concretes have perfomed well during the years of exposure; however, concrete with only PC and the concrete with Great Belt mix design appears, based on a microstructural appearance, as having the best quality. Contrary concrete with BFC has become very porous and carbonated in the surface. This structure is believed to decrease the durability of BFC concrete.

**Key words:** Fly ash, silica fume, blast furnace slag, durability, petrography

# 1. INTRODUCTION

DTI has since 1994 had a large number of concrete blocks and cores in storage. The concrete was originally mixed and cast with the purpose of testing different concrete compositions for their fire resistance. The mix designs used for the concrete were all mix designs allowed for the Øresund Link construction. One of the mixes was the Great Belt mix design. None of these mix designs were, however, used in Øresund Link construction.

The concrete in storage consists of 9 different concrete mixed with various combinations of fly ash, silica fume, blast furnace slag, and air void content. The concrete were vibrated with poker vibrator and covered with plastic immediately after casting. They were hereafter stored in outside water tanks for 8 years and then placed outside at DTI's storage facilities for the last 6 years.

During the spring 2008, a number of cores were taken from the blocks. Since the originally numbering during the outside storage time were lost the initially purpose of the coring was to identify which concrete were which. The second purpose of the coring was to analyse the concrete by means of all sorts of high technology analysis methods in order to get information about the paste structure and mineralogical composition of known concrete stored for years. This study deal with the initial analysis performed on the samples using only optical microscopy and scanning electron microscopy (SEM-EDX).

### 2. SAMPLES AND METHODS

The materials used for the concrete is as follows:

Cement:	Low-alkali sulphate-resistant Portland Cement (Aalborg Portland) [CEM I 42,5],
	Blast-furnace cement (Robur Hoogovencement class A) [CEM III/A]
Fly ash:	Avedøreværket (Danish)
Silica Fume:	Unicon Beton (Danish); added as slurry
Fine Agg.:	Sea Sand 0/4 class A
Coarse Agg.:	Rønne Granite, 2/8, 8/16, 16/32
Plasticizer:	Plastiment A-40, Sika
Super Plast.:	Sikament 110ü
Air:	Sika Fro V5

The mix designs of the 9 concrete samples are given in Table 1.

Content kg/m ³	Great Belt	ØSK- PC	ØSK- PCFA	ØSK- PCMS	ØSK- PCFA- MS	ØSK- PCMS Air	ØSK- PCFA- MS Air	ØSK- BFC	ØSK- BFC Air
Cement	330	348	332	337	302	337	305	352	346
Fly ash	42	0	55	0	55	0	56	0	0
Micro silica	21	0	0	14	15	14	15	0	0
Water	134	131	136	134	137	135	134	129	134
Air entraining	0	0	0	0	0	0.04	0.05	0	0.04
Plasticizer	0.78	0.55	0.75	0.50	0.60	0.57	0.52	0.47	0.38
Superplasticizer	2.25	1.79	2.23	2.03	2.46	2.04	2.26	2.01	1.43
Fine agg	523	776	732	774	722	666	627	755	646
Coarse agg. 2/8	0	364	362	359	354	359	358	352	335
Coarse agg. 8/16	1411	347	355	345	367	367	370	370	397
Coarse agg. 16/32	0	484	459	480	469	462	460	479	467
w/c-ratio	0.348	0.384	0.383	0.380	0.387	0.371	0.372	0.372	0.392
Air cont. fresh, %	1.5	2.9	4.6	2.8	2.2	5.4	5.4	2.6	6.3
Slump, mm	25	100	140	90	90	120	110	95	130
Density, kg/m ³	2410	2433	2384	2424	2451	2383	2353	2410	2294
Temp. fresh, °C	26.7	24.7	25.0	24.5	24.9	25.0	25.2	25.0	25.0
28 days comp. strength, MPa	75.6	56.0	53.2	52.1	61.4	64.2	57.5	58.6	43.3

Table 1 – Mix design of the 9 concretes kept in DTI's storage area

A total of 12 cores were taken from 12 blocks, giving doublets of three concrete compositions. The cores were all cored from the side of the blocks facing up meaning the core surfaces are form side surfaces.

In order to perform the analyses DTI performed different specimens of each cores such as plane sections, fluorescent impregnated thin sections, polished thin sections and polish sections. The methods used for specimen preparation follows the description found in [1].

A limited number of analyses have at present been performed of the samples namely macroscopic analysis, optical fluorescent microscopic analysis and SEM-EDX analysis. The SEM-EDX analyses were performed using a Quanta 400 ESEM from FEI, at DTI. The samples were carbon coated.

In order to 100 % identify the blocks of different composition the resistance was measured using a Wenner-probe. Three measurements (kOhm) were taken of each block after the blocks were surface dried by the sun.

# 3. CONCRETE CONDITIONS

The concrete blocks were initially inspected at the storage place (Figure 1). Generally the surfaces of all blocks appeared in good shape. Minor scaling of paste and chipping of corners where observed. The degree of damaged was though minor and more cosmetic. Table 2 shows a summary of the field inspection as well as results of the resistance measurements. The resistance measurements seem to reflect the composition and conditions of the different concretes. The highest resistance was observed in concrete having blast furnace slag cement (BFC) and in concretes having a composition of Portland cement (PC), fly ash (FA) and silica fume (MS). The lowest resistance was found in concrete with only PC and PC and MS. The concrete containing BFC was observed to have red and yellowish algae growth on the surface.



*Figure 1 – DTI's storage place with the tested concrete blocks.* 

Concrete ID	Block	Resistance	Inspection
	ID	kOhm	
		Average	
GB PCFAMS	5B	365	Bug holes towards cast surface
PC	3B	73	Scaling of corner
PCFA	4B	313	Bug holes towards cast surface
PCMS	6A	67	Scaling of surface
PCFAMS	7A	384	Scaling and cracks along corner, bug holes towards cast surface
PCFAMS	8D	470	Few cracks along corner
PCMS air	9A	86	Scaling of surface towards cast surface
PCFAMS air	2A	611	Weak scaling towards cast surface
PCFAMS air	7D	720	Scaling of corner
BFC	8B	781	Scaling of corner
BFC air	9B	777	Scaling and bug holes towards cast surface, weak red coloration of surface
BFC air	9D	1106	Scaling of corners, intense red coloration of surface, yellow algae growth

*Table 2 – Summary of field inspection and resistance measurements* 

### 4. PETROGRAPHIC ANALYSIS

### 4.1 Macroscopic Analysis

All concretes appeared sound with a dense dark paste. The air content of the samples were visually estimated and all in all seemed to correlate with the mix design except for the concrete with PCFA. This concrete was not air entrained but visually it had a high air content which correlated with the measured air performed in 1995.

Table 3 – Macroscopic observations. The air void parameters are from an air void study of cores examined in 1995. Abbreviations: SSA specific surface area, SF spacing factor.

Туре	Block	Visual air	Air	SSA	SF	Macro Observations
	ID	(2008)	vol %	$mm^{-1}$	mm	
GB(FAMS)	5B	Low air	1.1	34	0.27	Dark brownish paste
PC	3B	Low air	3.4	29	0.19	Dark brownish paste
PCFA	4B	High air	6.6	34	0.12	Dark brownish paste
PCMS	6A	High air	3.3	27	0.21	Dark brownish paste
PCFAMS	7A, 8D	Low air	2.4	24	0.28	Dark brownish paste
PCMSair	9A	High air	4.2	29	0.17	Dark brownish paste, weak scaling
PCFAMSair	2A, 7D	High air	5.0	20	0.24	Dark brownish paste
BFC	8B	Low air	1.8	31	0.24	Green paste, surface scaling during coring
BFCair	9B, 9D	High air	4.1	29	0.17	Green paste (9B), weakly green paste (9D)

### 4.2 Microscopic Analysis – Optical and SEM

A total of 12 thin sections representing the 9 different concretes have been examined using optical fluorescent microscopy. The micro-structural appearances of the concrete are summarized in the following.

#### Great Belt Mix Design (GB)

Generally this concrete appears in very good shape (Figures 2 and 3). Very minor carbonation to max. 0.2mm occurs in the surface. Only few cracks are present in the concrete (Figure 3). These

cracks are formed before the concrete was hardened. Ettringite filling voids are observed near the surface. Low air content. Fly ash particles and few silica fume agglomerates are observed.



Figure 2 – General structure of the GB concrete. Low air content.4.3x3.4mm



*Figure 3 – Surface area of the GB concrete. No cracks are present. 1.7x1.3mm* 

# ØSK PC Mix Design

The concrete having only PC is in very good shape (Figure 4). Very minor carbonation to max. 0.2mm occurs in the surface. Only few cracks are present within the concrete (Figure 5). Ettringite and calcium hydroxide (CH) is observed in voids throughout the concrete. The air content is relatively low.



Figure 4 – General structure of the PC concrete. Relative low air content, large voids. Ettringite needles in voids. 4.3x3.4mm



Figure 5 – Surface area of the PC concrete. No cracks are present. 1.7x1.3mm

### ØKS PCFA

The concrete with the combination PC and fly ash (FA) is generally in good shape (Figure 6). Despite the concrete is not air entrained it contains a relative high air content with many small voids. The concrete has a large number of small fines cracks and plastic defects near the surface, which all are formed in the non-harden concrete (Figure 7). The surface of the concrete is carbonated to depths of 0.2mm. Beneath the carbonated surface the paste is leached for CH to a depth of 6mm. A crust of calcite appears on the surface. FA particles are observed in the paste.



Figure 6 – General structure of the PCFA concrete (6A). Relative high air content, small voids. 4.3x3.4mm



Figure 7 – Surface area of the PCFA concrete (6A). Many small plastic cracks and defect present. 1.7x1.3mm

### PCMS

The concrete with the combination PC and silica fume (MS) is generally in good shape (Figure 8), but contains a relatively high number small, fines cracks and plastic defects near the surface. Some surface scaling is observed. These defects all seem to have formed in the non-harden concrete (Figure 9). The surface of the concrete is carbonated to depths of 0.2mm. Beneath the carbonated surface a 4mm thick zone without CH occurs. SEM analysis shows that the cement grains of this zone have a limited degree of hydration. The concrete contain a moderate air content with relatively large voids. A crust of calcite appears on the surface. Small ettringite needles are present in air voids throughout the concrete.



Figure 8 – General structure of the PCMS concrete. Moderate air content, relatively large voids. 4.3x3.4mm



Figure 9 – Surface area of the PCMS concrete. Small plastic cracks and defect present. 1.7x1.3mm



Figure 10 – BSE image of the upper 2mm of the PCMS concrete (6A). A high number of unhydrated cement grains appears just under a porous and well hydrated surface.



Figure 11 – BSE image of the near surface area of the PCMSair concrete (9A). Few fine cracks are observed. The surface is somewhat scaled.

### ØSK PCMS air

The concrete with the combination PC, MS and air is generally in good shape (Figure 12) but contains some small fines cracks near the surface (Figure 11) as well as one coarse surface parallel crack in a depth of 0.8mm (Figure 13). Minor surface scaling is observed. The cracks are all formed in the non-harden concrete. The surface of the concrete is carbonated to depths of 0.1mm. Carbonated paste occurs along the coarse crack. The concrete contains a moderate air content with relatively large voids Small air voids are filled with CH throughout the concrete.



Figure 12 - General structure of the PCMS air concrete. Moderate air content, relatively large voids. 4.3x3.4mm



Figure 13 – Surface area of the PCMS air concrete. A coarse crack is observed under the surface. 1.7x1.3mm

### ØSK PCFAMS Mix Design

Concrete from 2 blocks with the mix design of PC, FA and MS are examined (Figures 14 and 15). The structure of the concrete is not 100 % alike. The concrete of block 7A are generally in generally in a better shape than 8D. Both concretes have low air content and contain small fines cracks near the surface. The number of cracks are, however, much higher in core 8D (Figure 16

and 17). All the cracks observed are formed the non-harden concrete. The surface of the concretes is carbonated to different depths; core 7A to depths of 0.2mm (Figure 18) and core 8D from 0.12 to 10mm. The depth of carbonation correlates with the number of cracks in the surface. A crust of calcite is seen on the surface of block 7A but not on block 8D. Small ettringite needles are present in air voids near the surface of the concrete of block 7A but not 8D. Fly ash particles are observed in both concretes but silica fume agglomerates only in block 7A.



Figure 14 – General structure of the PCFAMS concrete (7A). Low air content, relatively large voids. 4.3x3.4mm



Figure 16 - Surface area of the PCFAMS concrete (7A). Few small plastic cracks present. 1.7x1.3mm



Figure 15 - General structure of the PCFAMS concrete (8D). Low air content, relatively large voids. 4.3x3.4mm



Figure 17 – Surface area of the PCFAMS concrete (8D). Many small plastic cracks/defects present. 1.7x1.3mm

In front of the carbonated surface a zone rich in ettringite is observed (Figure 19). In this zone ettringite is observed within relicts of former cement grains. Minor micro-cracking of the paste occurs in this zone.



Figure 18 – BSE image of the surface area of PCFAMS concrete (7A). Cement grains are decalcified and the pasta partially carbonated.



Figure 19 – BSE image of the pasta below carbonated surface of the PCFAMS concrete (7A). Ettringite is observed in relicts after former cement grains.

### ØSK PCFAMS air Mix Design

Concrete from 2 blocks with the mix design of PC, FA, MS and air are examined (Figures 20 and 21). The structure of the concrete is not 100 % alike, but both are generally in good shape. Some fine cracks are found in the surface of both blocks to depths of 12mm in 2A and 6mm in 7D (Figures 22 and 23). The cracks observed are formed the harden concrete. The surface of the concretes is carbonated to different depths; block 2A to depths of 0.12mm and block 7D from 4 to 8mm (Figure 25). A crust of calcite is seen on the surface of block 2A but not on block 7D. Both concretes have high air content with well distributed voids. Ettringite needles are present in air voids near the surface of both concretes. Fly ash particles but not MS are observed in both concretes.



Figure 20 – General structure of the PCFAMS air concrete (2A). Moderate air content, well distributed. 4.3x3.4mm



Figure 21 - General structure of the PCFAMS air concrete (7D). Moderate air content, well distributed. 4.3x3.4mm



Figure 22 - Surface area of the PCFAMS air concrete (2A). Few fine cracks present. 1.7x1.3mm



*Figure 23 – Surface area of the PCFAMS air concrete (7D). Few cracks present. 1.7x1.3mm* 

In front of the carbonated surface of concrete 2A a zone rich in ettringite is observed (Figure 24). In this zone ettringite is observed within relicts of former cement grains. Minor micro-cracking of the paste occurs in this zone.



Figure 24 – BSE image of PCFAMSair concrete (2A). Ettringite is observed in relicts after former cement grains. Positioned under carbonated surface at a depth of 132 µm.



Figure 25 – BSE image of the surface of PCFAMSair concrete (7D). The surface area partially carbonated.

### ØSK BFC Mix Design

The concrete containing slag cement is generally in good shape (Figures 26 and 27), but contains some small fine cracks near the surface as well as a high number of adhesion cracks along sand grains. The porosity of the surface is highly increased (Figure 28). The depth of porosity correlates with the depth of carbonation which is 4mm (Figure 29). The concrete contains a low air content.



*Figure 26 - General structure of the BFC concrete. Low air content. 4.3x3.4mm* 



Figure 27 – Surface area of the PCMS air concrete. The porosity is increased near the surface. 1.7x1.3mm

In front of the carbonated surface a distinct zone rich in ettringite is observed (Figure 30). In this zone ettringite is observed within relicts of former cement grains. Some micro-cracking of the paste occurs in this zone.

Throughout the pasta the slag grains have reacted. The small grains appear fully reacted leaving a grey imprint (Figure 30). A thin reacted zone is present around most of the larger slag grains. Monosulphate is occasionally observed in voids of the concrete.



Figure 28 – BSE image of the surface area of BFC concrete (8B). The surface is porous.



Figure 29 – BSE image of the porous surface of BFC concrete (8B). Angular slag grains are abundant. The paste is partially carbonated.



Figure 30 – BSE image of pasta in front of porous carbonated surface of BFC concrete (8B). Many of the slag grains are fully reacted. Ettringite appears in air voids and in relicts after former cement grains



Figure 31 – BSE image of BFC concrete (8B). Small slag grains are fully reacted (grey), larger grains (white) are surrounded by a grey reaction zone. An monosulphate-like phase is present in the paste

# ØSK BFC air Mix Design

Concrete from 2 blocks with the slag cement (BCF) and air are examined and is generally in good shape (Figures 32 and 33). Both concretes contain relatively many fine cracks in paste as well as many adhesion cracks. Few fine cracks are observed in the surface of block 9D; one extending to a depth of 10mm. The porosity of the surface is increased (Figures 34 and 35) and carbonated to depths of 6 to 10mm. The upper max. 1.2mm of the paste on top of carbonated paste appears black as if it was attached by acid. Both concretes contain a moderate air content. Calcite precipitation occurs in air voids near the surface of block 9B.



Figure 32 – General structure of the BFCair concrete (9B). Moderate air content, well distributed. 4.3x3.4mm



Figure 33 - General structure of the BFC air concrete (9D). Moderate air content, well distributed. 4.3x3.4mm



Figure 34 - Surface area of the BFC air concrete (9B). Few small defects present. 1.7x1.3mm



Figure 35 – Surface area of the BFC air concrete (9D). Few small defects present. 1.7x1.3mm

# 4.3 Microanalysis – SEM

The SEM-EDX analyses have primarily been performed in order to analyse the composition of the inner and outer hydration product in order to see if the different mix designs could be distinguished from each based on the Ca/Si ratio. The data shows a clear difference of the Ca/Si ratio between the different mixes especially when looking at the inner hydration product (Figure 36, Table 4).



Figure 36 – Ca/Si ratio of the inner hydration product of the different mixes

Based on the Ca/Si ratio the examined mix designs can be divided into three groups (Figure 26). The mix with only PC and mixes with PC and MS plot as one group; in general with the highest Ca content. The silica fume does not seem to influence on the total Si content as it can not be distinguished from concrete's without silica fume. The mixes containing FA plot as a second

group, with the highest Si content. The FA content, which also is about twice as high as the MS content of the mix design, seems to play a major influence on the Ca/Si ratio. The BFC mixes has the lowest Ca/Si ratio as well as having a rather a low content of both elements. Instead the inner hydration zone, "reaction zone", around the slag grains is rich in Mg and Al (Table 4).

Table 4 – Average numbers obtained on the inner hydration product of the different mixes. Mixes are arranged in increasing Ca/Si order.

		Atomi	ic %										
Sample ID	Phase	Na	Mg	Al	Si	S	Cl	K	Ca	Fe	0	<u>Ca</u> Si	(Si+Al)
BFC	"IH"	0.14	12.32	8.25	8.27	0.92	0.23	0.07	11.57	0.18	58.06	1.40	0.70
BFCair	"IH"	0.13	11.27	8.94	8.32	0.94	0.41	0.07	11.81	0.23	57.90	1.42	0.68
GB	IH	0.19	0.71	1.39	15.38	0.21	0.09	0.07	23.22	0.49	58.27	1.51	1.39
PCFAMS	IH	0.26	0.66	1.62	15.01	0.25	0.45	0.06	23.02	0.60	58.09	1.53	1.38
PCFAMSair	IH	0.23	0.48	1.60	15.13	0.19	0.08	0.07	23.48	0.52	58.24	1.55	1.40
PCFAMS	IH	0.37	0.54	1.32	15.04	0.21	0.05	0.14	23.80	0.51	58.03	1.58	1.46
PCFA	IH	0.10	0.68	1.32	15.09	0.27	0.07	0.02	23.90	0.39	58.18	1.58	1.46
PCFAMSair	IH	0.25	0.79	1.20	14.99	0.22	0.07	0.04	23.97	0.47	58.02	1.60	1.48
PCMS	IH	0.19	0.55	0.65	13.96	0.44	0.11	0.03	26.11	0.39	57.57	1.87	1.79
PCMSair	IH	0.17	0.54	0.83	13.84	0.49	0.04	0.05	26.03	0.40	57.64	1.88	1.77
PC	IH	0.19	0.57	0.75	13.60	0.56	0.19	0.08	26.17	0.41	57.49	1.92	1.82

Contrary the difference between the chemistry of the outer hydration product of the different mixes is less obvious (Figure 37, Table 5), except for the BFC mix which are slightly different.



Figure 37 - Ca/Si ratio of the outer hydration product of the different mixes

		Atomi	c %										
Sample ID	Dhasa	No	Ma	A 1	C:	c	Cl	V	Ca	Ea	0	$\frac{Ca}{S}$	$\frac{Ca}{(Si+A1)}$
Sample ID	Fliase	Ina	Mg	AI	31	3	CI	Λ	Ca	ге	0	51	(SI+AI)
BFC	OH	0.54	0.85	2.89	13.78	1.88	0.09	0.14	20.06	0.40	59.37	1.46	1.20
PCFAMSair	OH	0.52	0.44	1.69	14.05	0.87	0.10	0.09	23.33	0.65	58.27	1.66	1.48
PCFAMSair	OH	0.32	0.30	1.54	14.31	0.75	0.14	0.17	23.81	0.47	58.21	1.66	1.50
BFCair	OH	0.90	1.23	3.40	12.30	2.09	0.13	0.25	20.48	0.39	58.85	1.66	1.30
PCFAMS	OH	0.63	0.25	1.65	13.88	0.91	0.16	0.22	23.89	0.38	58.06	1.72	1.54
PCFAMS	OH	0.58	0.20	1.80	13.36	1.42	0.39	0.12	23.38	0.47	58.30	1.75	1.54
PCFA	OH	0.60	0.35	1.68	13.33	1.07	0.11	0.15	24.09	0.58	58.06	1.81	1.60
PCMS	OH	0.72	0.30	1.34	13.47	0.96	0.12	0.37	24.36	0.54	57.83	1.81	1.65
GB	OH	0.52	0.30	2.45	12.73	1.16	0.13	0.14	23.57	0.79	58.20	1.85	1.55
PCMAair	OH	0.46	0.28	1.18	12.93	0.97	0.06	0.18	25.60	0.60	57.76	1.98	1.82
PC	OH	0.48	0.20	1.16	12.07	1.10	0.19	0.17	26.95	0.42	57.27	2.23	2.04
PCFA PCFA PCMS GB PCMAair PC	OH OH OH OH OH	0.38 0.60 0.72 0.52 0.46 0.48	0.20 0.35 0.30 0.30 0.28 0.20	1.80 1.68 1.34 2.45 1.18 1.16	13.30 13.33 13.47 12.73 12.93 12.07	1.42 1.07 0.96 1.16 0.97 1.10	0.39 0.11 0.12 0.13 0.06 0.19	0.12 0.15 0.37 0.14 0.18 0.17	23.38 24.09 24.36 23.57 25.60 26.95	0.47 0.58 0.54 0.79 0.60 0.42	58.06 57.83 58.20 57.76 57.27	1.73 1.81 1.81 1.85 1.98 2.23	1.34 1.60 1.65 1.55 1.82 2.04

Table 5 – Average numbers obtained on the outer hydration product of the different mixes. Mixes are arranged in increasing Ca/Si order.

### 5. CONCLUSIONS

The present study using only optical microscopy and SEM have showed that all concretes, on a macroscopic level, have performed well to the exposure to normal inland climate.

The concretes having only PC and the concrete with the Great Belt mix design are almost looking perfect. There are no signs of defects from casting and no signs of deteriorations from the exposure. The porosity of the paste is similar throughout the concrete.

Concretes having combinations of FA and MS all show signs of casting defects in the form of small oriented cracks near the surface. Small micro-cracks are also observed in the surface of most of these concretes resulting in minor scaling of the surfaces. The porosity of the paste is similar throughout the concrete.

The BFC concretes all show a remarkable deep carbonation of the surface. Looking at the carbonated zone it appears that this zone is highly porous. The surface of the concrete is only minor damaged by scaling but it is believed that the highly porous zone with affect the generally durability of the BFC concrete. In front of the carbonated zone of the BFC concrete a thin zone containing ettringite in paste appears. This is, however, also observed in some of the other concrete but not as distinct as observed in the BFC concretes. Whether or not the presence of this zone means anything for the durability of the BFC concrete is at present not known.

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# **Experiences from Norwegian Marine Exposure Sites** - How does the Experiences comply with the Standards?





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### ABSTRACT

The Norwegian Public Roads Administration (NPRA) has over a long period tested a large variety of concrete compositions in marine exposure sites located along the coast of Norway. Being a public owner of many coastal bridges enables us to practice relate the results from such exposure sites based on experience. This paper gives an introduction to some of the marine exposure projects and experiences in relation to the upcoming revision of EN 206-1.

**Key words:** Concrete, reinforcement corrosion, marine exposure, chloride penetration, standards

# 1. INTRODUCTION

It is an inconvenient truth that concrete coastal bridges, especially those situated in harsh climatic conditions, suffer from various deteriorating mechanisms. The most severe is considered to be steel reinforcement corrosion due to chloride ingress from sea-water.

Most research on durability properties for concrete concern the materials performance on "labcrete" specimens and do not take into account site execution and production issues. Experience shows that the execution of concrete structures is also critical for the future durability properties. Rising of formwork, assembly of the reinforcement, concrete casting and curing is, of course, done on site, and thereby strongly depends on weather conditions. Building in a harsh and rough climate (strong winds, low temperatures and rough sea) is very challenging and demanding and experience show that execution conditions affect the quality of the structure.

Another point of concern is when damaged areas (corroded steel and delaminated concrete) are to be repaired on existing structures. This is a very complex task since the concrete has to be removed to a depth where the chloride content is lower than a critical level. A level that in many cases is difficult to establish with any degree of certainty. The NPRA has several ongoing projects to map sustainable materials and applicable concrete compositions, concrete protection systems (mainly surface treatments) and alternative repair strategies. In this paper two projects will be introduced and briefly presented:

- Chloride Resistant Concrete
- Gimsøystraumen Bridge

# 2. CHLORIDE RESISTANT CONCRETE:

"Development of chloride resistant concretes", initiated in 1993. The project, still running today, involved extensive laboratory investigations in addition to field exposure of various types of reinforced concrete elements made from 31 different concrete types. Phase I (started in 1993) of the project involved 17 different concretes, whereas Phase II (started in 1997) involved another 14 different concretes.

In Phase II monitoring of some corrosion parameters were initiated in 1999. This involved automated measurements of the electrical resistance (converted to resistivity by calculation) of concrete and the rate of oxygen reduction (cathodic effectiveness) for the various concrete types.

# 2.1 Concrete types and elements

The concretes in Phase II were designed to represent both past, present and future concretes used in Norwegian coastal bridges. Note that all concretes were full scale mixed at a concrete factory and cast under realistic conditions to represent "real-crete" and not "lab-crete". Also note that the experimental set-up does not involve a systematic variation of parameters, as it is more based on a qualified estimate of the durability properties of the various concretes.

The concrete qualities span from a pure OPC concrete with w/c-ratio 0.56 to an OPC concrete with 9% silica fume and a w/b-ratio of 0.32. There are six concretes with various three-powder mixes (OPC/PFA/CSF; OPC/BFS/CSF). Table 1 shows the mix proportions of all 14 concretes without additives from part II. All concretes were mixed using the same plastizers, super-plastizers and air-entraining additives in varying amounts in order to produce concretes with good workability.

					, ,		· O:		
Concrete	Cement	PFA	BFS	CSF	Water #	Sand	Gravel	w/b	Cement type
1	326	_	_	_	181	939	886	0.56	CEM I
2	365	-	-	15	180	932	880	0.47	CEM I
3	374	-	-	34	192	861	881	0.47	CEM I
4	378	-	-	34	181	872	898	0.44	CEM I
5	312 *	79	-	16	175	885	918	0.43	CEM II/A-V
6	389 **	-	-	35	186	827	932	0.44	CEM I
7	139 ***	-	254	16	165	889	920	0.40	CEM III/A
8	338	84	-	17	186	795	926	0.42	CEM I
9	443	-	-	40	153	823	913	0.32	CEM I
10	360 *	90	-	40	165	814	928	0.34	CEM II/A-V
11	406	101	-	20	173	742	922	0.33	CEM I
12	384	95	-	19	169	798	926	0.34	CEM I
13	435	-	-	25	185	738	587 ¤	0.40	CEM I
14	406	-	-	34	188	707	591 ¤	0.43	CEM I

Table 1 Mix proportion of the concretes, constituents in  $kg/m^3$ 

# Free water content (total water minus absorbed water in the aggregate)

* PFA blended OPC; 20% PFA/80% OPC

** Sulphate resistant OPC

*** BFS blended OPC; 65% BFS/35% OPC

x Light weight aggregate (high strength)

For each concrete type, three different types of reinforced elements were made: 3m long beams, 15 x 30 cm in cross-section; 1m high pillars, 30 x 30 cm in cross-section; and 1.5 x 0.75 m big slabs, 20 cm thick. Six beams were made of each concrete type: two beams were exposed uncracked, cover depth 25mm; two were cracked in the middle section and exposed to sea water with the cracks left open (tensioned by bending), cover depth 25mm; and two uncracked beams with cover depth 60mm. One beam of each type was produced with cast-in electrodes, i.e. three beams of each concrete type have cast-in electrodes. The electrodes are simply reinforcement steel bars 40cm long with diameter 12mm. The placement of both electrodes and beams is such that the three electrodes in each beam are situated in a) purely atmospheric; b) tidal; and c) purely submerged conditions, respectively. Figure 1 shows the details of beams and electrodes.



*Figure 1 Reinforced beams with electrodes at three positions* 

### 2.2 Measurements

The electrical resistance was measured between the electrode at each of the three positions (a-c) and the main reinforcement in two different ways:

- a) Continuously, with an automatic monitoring system with data logging capabilities
- b) Manually, with a hand-held device from time to time over the exposure period

The data logging equipment has capacity to continuously monitor all three beams (i.e. nine resistances) of one concrete type at the same time. This means that over the same period of time only one concrete type is monitored, i.e. a direct comparison between different concrete types over the same period of time is not possible. However, with this setup we get a lot of good data to compare different exposure conditions and the influence of cover depth. To measure all concretes almost at the same time, one has to switch from one concrete to the other consecutively. This has to be done by physically un-plugging and re-plugging the measurement cable from the DSUBs that connects the nine electrodes in each concrete type (one DSUB for each concrete type). This has been done several times in different seasons with different temperatures to get comparable data from all concrete types. The monitoring system is measuring the resistance at 100Hz ("ResMes" device from Protector AS in Norway), and the logging interval can be adjusted to any given interval down to one measurement every second ("Camur" data logger from Protector AS). Normally the logging interval is one measurement

every hour. Data is downloaded to a desktop computer in the office using a modem and lately via Internet.

Results from this project will be analysed and published in the near future. Some preliminary results on electrical resistivity are published by Larsen et al. [1]

# **3. GIMSØYSTRAUMEN BRIDGE:**

Gimsøystraumen Bridge is a cantilever bridge finished in 1981, situated in Lofoten in Northern Norway. It is 840 meters long with 5 long spans (126-148 m) and 4 short spans (25-65 m). The post tensioned box girder has a height that varies from 2.2 to 7.4 m. The highest point of the bridge is 36.5 m above the sea level.

In order to gain experience with the complicated task of maintaining and repairing costal concrete bridges, the NPRA initiated the "OFU Gimsøystraumen Bridge Repair project" in 1993. The project ended in 1997, and was concluded in the international conference "Repair of Concrete Structures. From Theory to Practice", held in Svolvær, Norway, May 1997. Numerous reports and publications (in Norwegian) give background, accomplishment, results and recommendations (conclusions) from this 4 million €project. In 1997 a new project was started to assess the effect of the various measures that were undertaken during three years with trial repairs and maintenance work on the bridge.

# 3.1 Environmental load

In 1992 a large field investigation was conducted to assess the condition of the bridge. About  $9000 \text{ m}^2$  of the concrete surface was visually inspected for any signs of deterioration. In this area a lot of measurements were made: about 17,000 single measurements of the concrete cover thickness; more than 900 chloride profiles were established; the reinforcement was visually inspected in 110 locations after chiselling off the concrete cover; and mapping of the electro chemical potential (ECP) of the reinforcement was done on about 3000 m² of the concrete surface.

To briefly summarise the results from this rather large investigation, one can say:

- there is a significant windward/leeward effect on the microclimate, both for the pillars and the box girder
- the chloride load is significantly higher on the leeward side of the box girder and the pillars, compared to the windward side, as shown in Figure 2
- the chloride load is significantly affected by the height above sea-level (being highest for the parts closest to the sea-level)
- there was significant corrosion on the reinforcement on the lower parts of the leeward side of the box girder
- the thickness of the concrete cover was on average 29 mm (std. deviation 5.2 mm) for the box girder
- the depth of carbonation varied from 1 to 6mm



Figure 2 – Chloride content in % of concrete mass, measured at depth 0-10 mm from the concrete surface (Fluge 2001, [2])

### **3.2** Trial repairs and preventive measures

In 1993, 1994 and 1995, trial repairs were done on different parts of the Gimsøystraumen Bridge. All trial repairs were conducted in the same manner:

- 1 A thorough condition survey was carried out first
- 2 A large-scale monitoring system was then installed
- 3 The trial repair work was done
- 4 Finally a detailed documentation was established

Results from the following up project, i.e. data from 1997 up to present day, will be published when all the data are systemised and analysed. The project is now being shut down since the bridge will undergo a full refurbishment in the near future. Some preliminary results from the following up project are published by Larsen and Østvik, [3].

### 4. DISCUSSION AND RELATION TO STANDARDS

As indicated in the previous parts the NPRA has ongoing and interesting field exposure programmes both from real structures and various exposure sites. All in all our experience agree with the conclusions of Helland, Maage and Aarstein [4] that well compacted and sound concrete surfaces are performing well even in severe marine climate. However, it is clear that structures actually build in harsh environments probably will contain defects of some sort given the rough execution conditions during the building period. Helland, Maage and Aarstein's results are mainly based on offshore concrete base structures, which were manufactured either in fjords or docks under relatively controlled conditions. For several of the Norwegian coastal bridges this was not the case. Occasionally, reports from actual building sites tell stories about undesirable incidents, i.e. that formwork and assembled reinforcement is exposed to chloride splash and spray during storms. Mobile concrete plants being exposed to sea water spray etc. If

this is not dealt with properly, which even in many cases are impossible, the structure contains a life span reducing defect from even before it is completed.

The standards define limits for the maximum chloride content of the concrete constituents in addition to the exposure class, however, the standards does not take into account such extreme situations as in the examples given above. This is an important issue, and the actual building environment should be taken into account when deciding the exposure class for the structure.

It is no use building with the best materials if the execution conditions do not allow the contractor to perform a sufficiently good job in transforming the best materials into the best structures. It is the authors' opinion that the execution conditions should be one of the factors involved when deciding the exposure class and if preventive measures are needed. Examples on preventive measures may be a more rigid regulation of building period to avoid for example winter storms, or alternatively a requirement for non corrosive reinforcement or an immediate surface treatment if continuous building is chosen.

# 5. CONCLUDING REMARK

In the authors' point of view the current best knowledge on obtaining sufficient durability performance on marine concrete structures are mainly based on experience. Few research programmes has been dedicated on execution related issues. Efforts should be taken to optimise execution methods in order to obtain the best possible durability performance for concrete structures in severe climates or environments. Based on this the standards should focus on subjects like how to obtain and secure concrete cover, avoid chloride exposure on reinforcement steels before casting etc.

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# Field exposure on Gotland. Carbonation rate of concrete.



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Key words: Cement, concrete, carbonation, field exposure,

# 1. INTRODUCTION

### 1.1 General

Since the beginning of the 1980:s Cementa has exposed concrete samples at Smöjen on the east coast of Gotland. Manufacture and set out of concrete samples was done for the first time in 1982 with the main cements then produced by Cementa, and since then every time that a new bulk cement was introduced.

Different W/C-ratios and curing conditions have been applied. Storage conditions are outdoors unsheltered from rain (main series), but samples stored outdoors, but sheltered from rain, also exist.

# 1.2 Concrete

The standard W/C-ratio is 0.60. There are some samples with W/C = 0.45 and 0.80

Different cements are included in the investigations.

CEM I –cements:

Slite Std P and Skövde Std P Two old Portland cements, designation according to present standard EN 197-1 is CEM I 42,5 R

Slite SH and Skövde SH Rapid hardening Portland cement, designation CEM I 52,5 R

Degerhamn Anläggning Sufate resisting, low alkali Portland cement with moderate heat development, designation CEM I 42,5 N MH LA SR. CEM II-cements:

Skövde St<br/>d M. An old blended cement with 24 % of fly ash, designation according to present standard is<br/> CEM II/B-V 42,5 N

Slite and Skövde Byggcement Portland-limestone cement, designation CEM II/A-LL 42,5 R

Also some other special cements have been used but are not presented in this report.

# 1.3 Curing

The test specimens were subject to two different curing methods, A and B.

The A-method samples were demoulded after one day, followed by wet curing for six days (wet sacking covered with plastic sheets) and then storage in laboratory air until transport to the exposure site, normally at the age of one month.

The B-method samples were demoulded after one day, then stored in laboratory air for one week, then wet cured for six days and finally stored in laboratory air again until transport to the exposure site.

# 1.4 Samples stored outdoors, unsheltered from rain

The unsheltered samples are in the shape of small walls, dimensions  $800 \times 800 \times 150$  mm. The walls are stored vertically on concrete foundations at the exposure field, see photo below.



*Figure 1 – Photo from Smöjen, Gotland. Wall samples.* 

Regular tests of strength development and carbonation depths by the phenolphthalein method have been done on drilled out cores from the unsheltered samples.

### 1.5 Samples stored outdoors, sheltered from rain

The sheltered samples are in the shape of prisms, dimensions  $400 \ge 250 \ge 150$  mm The four small sides are sealed with bitumen, the two large ones are uncoated. The prisms are stored on wooden bars under roof in an open shelter.

The sheltered ones are provided with reinforcement with different concrete cover and the intention is to follow corrosion rate after that the carbonation front has reached the bar. No observation of beginning corrosion has been done so far so the samples have rested unused. During 2008 some of them have however been analyzed for carbonation depth with the phenolphthalein method in connection with the  $CO_2$ -uptake project, se below, clause 4. The uncoated under and upper sides have been studied.

# 2. **RESULTS**

Results from measurements of carbonation depths on the unsheltered walls are presented in table 1.

				Carbonatio	n dep	ths (m	ım, me	ean va	lue)						
			Year		21	1	3	5	7	8	10	11	15	20	25
Curing	w/c	Cementtype	casting	Direction	d	У	У	У	У	У	У	У	У	У	У
A	0.60	Slite STD P	82	mean.	0.1	1.0	2.2		2.2			2.3	3.4	2.4	3.1
				East (A)	0.0	1.0	2.3		2.3			5.0	3.5	3.2	3.8
				West (B)	0.0	1.0	2.0		2.0			1.0	3.3	1.7	2.5
В	0.60	Slite STD P	82	mean.	0.0	2.7	2.5		4.7			2.2	3.3	2.3	2.8
				East (A)	0.0	2.7	3.0		4.7			2.0	3.0	3.7	2.8
				West (B)	0.0	2.7	2.0		4.7			2.3	3.7	0.8	2.8
А	0.60	Slite STD P	82	mean.	0.0	3.2	2.2		4.4			1.6	4.3	2.5	1.9
				East (A)	0.0	3.3	2.7		4.3			2.0	3.3	2.5	2.0
				West (B)	0.0	3.3	2.0		4.3			1.7	6.0	2.5	1.5
В	0.60	Slite STD P	82	mean.	0.0	3.0	2.2		4.0			2.0	4.8	3.7	2.4
				East (A)	0.0	3.0	2.7		4.0			2.7	4.3	4.7	2.4
				West (B)	0.0	3.0	1.7		4.0			1.3	5.3	2.7	2.5
А	0.60	Skövde STD P	82	mean.	0.0	2.7	3.5		5.3			2.7	4.3	2.4	4.0
				East (A)	0.0	2.7	4.3		5.3			3.3	4.3	2.7	4.5
				West (B)	0.0	2.7	2.7		5.3			2.0		2.2	3.5
В	0.60	Skövde STD P	82	mean.	0.0	2.0	2.3		4.5			3.2	5.0	4.0	2.9
				East (A)	0.0	2.0	2.3		4.7			3.3	4.0	4.3	2.3
				West (B)	0.0	2.0	2.3		4.3			3.0	7.0	3.7	3.5
А	0.60	Skövde STD M	82	mean.	0.0	3.7	4.8		6.0			3.8	5.5	5.3	3.8
				East (A)	0.0	3.7	6.0		6.0			4.0	5.3	6.2	4.5
				West (B)	0.0	3.7	3.7		6.0			3.7	5.7		3.0
В	0.60	Skövde STD M	82	mean.	0.0	3.7	4.7		6.0			3.7	3.8	3.7	5.8
				East (A)	0.0	3.7	5.3		6.0			3.7	2.7	4.2	4.9
				West (B)	0.0	3.7	4.0		6.0			3.7	5.0		6.6

Table 1 – Results from measurements of carbonation depths of unsheltered walls.

А	0.60	Slite STD P	84	Mean	0.0	2.3	3.0	2.5		3.7	2.5	1.5
				East (A)	0.0	2.7	3.0	2.3		3.7	2.7	1.7
				West (B)	0.0	2.0	3.0	2.7		3.7	2.3	1.3
В	0.60	Slite STD P	84	Mean	0.0	3.2	3.0	2.2		3.2	1.7	3.0
				East (A)	0.0	3.3	3.0	2.3		3.3	1.7	3.0
				West (B)	0.0	3.0	3.0	2.0		3.0	1.7	3.0
А	0.60	Deg Anl	84	Mean	0.0	2.5	3.0	2.3		3.0	2.2	1.3
				East (A)	0.0	2.7	3.0	2.3		3.3	2.0	1.7
				West (B)	0.0	2.3	3.0	2.3		2.7	2.3	1.0
В	0.60	Deg Anl	84	Mean	0.0	2.3	3.0	2.3		2.5	1.4	3.0
				East (A)	0.0	2.3	3.0	2.2		3.0	1.5	3.7
				West (B)	0.0	2.3	3.0	2.3		2.0	1.2	2.3
А	0.60	Skövde STD M	84	Mean	0.0	1.7	3.0	3.8		4.3	4.3	4.5
				East (A)	0.0	2.0	3.0	4.3		5.3	4.6	5.0
				West (B)	0.0	1.3	3.0	3.3		3.3	4.0	4.0
В	0.60	Skövde STD M	84	Mean	0.0	1.8	3.5	3.5		4.3	3.4	4.8
				East (A)	0.0	2.0	4.0	4.0		5.7	3.9	6.0
				West (B)	0.0	1.7	3.0	3.0		3.0	2.9	3.7
А	0.60	*Slite STD	85	Mean	0.0	0.0	3.7		2.3	4.8	5.8	5.3
				East (A)	0.0	0.0	4.3		4.0	6.0	6.9	6.0
				West (B)	0.0	0.0	3.0		0.7	3.7	4.7	4.7
В	0.60	*Slite STD	85	Mean	0.0	0.0	4.3		1.5	5.2	4.7	5.0
				East (A)	0.0	0.0	4.7		3.0	5.7	5.1	5.3
				West (B)	0.0	0.0	4.0		0.0	4.7	4.3	4.7
А	0.80	Slite STD	85	Mean	0.0	2.0	7.5		9.3	9.5	9.0	8.2
				East (A)	0.0	2.0	7.7		8.7	9.0	7.7	7.7
				West (B)	0.0	2.0	7.3		10.0	10.0	10.4	8.7
В	0.80	Slite STD	85	Mean	0.0	3.0	13.2		9.3	10.0	8.3	9.0
				East (A)	0.0	3.0	13.3		9.3	9.3	7.7	12.3
				West (B)	0.0	3.0	13.0		9.3	10.7	8.9	5.7
А	0.40	Deg Anl	85	Mean	0.0	0.0	1.2		0.0	0.3	2.3	0.8
				East (A)	0.0	0.0	1.0		0.0	0.3	2.0	0.7
				West (B)	0.0	0.0	1.3		0.0	0.3	2.7	1.0
В	0.45	Deg Anl	85	Mean	0.0	0.0	1.3		0.3	0.0	4.5	0.9
				East (A)	0.0	0.0	0.5		0.5	0.0	4.0	0.4
				West (B)	0.0	0.0	1.7		0.0	0.0	4.9	0.8
А	0.45	Slite STD	85	Mean	0.0	0.0	1.0		1.0	1.0	1.3	1.5
				East (A)	0.0	0.0	1.0		1.0	1.3	1.3	1.7
				West (B)	0.0	0.0	1.0		1.0	0.7	1.3	1.3
В	0.45	Slite STD	85	Mean	0.0	0.0	2.5		1.0	1.0	1.9	0.7
				East (A)	0.0	0.0	2.3		1.0	1.0	2.1	0.3
				West (B)	0.0	0.0	2.7		1.0	1.0	1.7	1.0
А	0.60	Skövde SH	88	Mean	0.0	2.3	2.0	2.2		3.5	2.5	
				East (A)	0.0	2.7	2.0	3.0		4.3	2.7	
				West (B)	0.0	2.0	2.0	1.3		2.7	2.3	
В	0.60	Skövde SH	88	Mean	0.0	2.0	1.8	1.7		1.5	2.5	
				East (A)	0.0	2.0	1.7	2.0		2.0	3.0	
				West (B)	0.0	2.0	2.0	1.3		1.0	2.0	
А	0.60	Slite SH	88	Mean	0.0	1.3	1.3	1.3		1.2	2.0	
				East (A)	0.0	1.3	1.3	1.7		1.3	2.0	
				West (B)	0.0	1.3	1.3	1.0		1.0	2.0	
В	0.60	Slite SH	88	Mean	0.0	1.7	1.7	1.3		2.2	2.2	
				East (A)	0.0	1.7	1.7	1.7		2.3	2.3	

				West (B)	0.0	1.7	1.7		1.0	2.0	)	2.0	
А	0.60	Slite Byggc.	2000	Mean	0.0	0.9		2.5	3.2				
				East (A)	0.0	0.7		2.7	3.0				
				West (B)	0.0	1.2		2.3	3.3				
В	0.60	Slite Byggc.	2000	Mean	0.0	2.9		3.0	4.8				
				East (A)	0.0	2.5		3.0	4.2				
				West (B)	0.0	3.3		3.0	5.3				
А	0.60	Skövde Byggc.	2000	Mean	0.0	1.8		2.7	3.4				
				East (A)	0.0	2.0		2.5	3.3				
				West (B)	0.0	1.7		2.8	3.5				
В	0.60	Skövde Byggc.	2000	Mean	0.0	2.6		2.8	3.8				
				East (A)	0.0	2.3		2.7	3.8				
				West (B)	0.0	2.8		3.0	3.8				

* Concrete with 10 % silica fume.

In figure 2 the carbonation depths after 25 years are shown for surfaces facing to the west (dominating wind) and to the east. No systematic difference can be found. In the following presentation the mean values have been used.

# Carbonation depths after 25 years, surface faced to the east (dashed) and west (filled)



Figure 2 – Carbonation depths, influence of wall surface orientation

The carbonation depths are presented as function of time for some concretes in figure 3 and 4. The two different curing methods are of no importance at actual age 8-25 years. Results from method A have been chosen.



Carbonation depths after 8 years. Different cements and W/C-ratio. Outdoors, unsheltered from rain.

Figure 3 – Carbonation depths as function of time, first 8 years.



Carbonation depths first 25 years. Different cements and W/C-ratio. Outdoors, unsheltered from rain

Figure 4 – Carbonation depths as function of time, first 25 years.

### **3 DISCUSSION**

The carbonation depth is, as expected, strongly depending on W/C ratio. It is also depending to some extent on cement type. Concrete with CEM II/A-LL and even more concrete with CEM II/B-V shows larger carbonation depths than the CEM I concrete.

In all the cases however carbonation depths seems to reach a constant value after 3 - 7 years. Even in the case with W/C = 0.80 and CEM I the depth is not larger than about 10 mm after 25 years and have not increased in the period 8 - 25 years.

The results for the walls exposed to rain are thus not in line with the commonly anticipated square root dependency of time.

Following Swedish praxis the exposure class according to EN 206-1 for the walls would be XC 4, the minimum requirement on W/C is 0.55 according to the Swedish SS 13 70 03. The minimum cover to reinforcement is 20 mm (SS 13 70 10). If the tendency with constant depth of carbonation is stable during coming years it means that the safety margin against corrosion is large. This is also true for the CEM II cements even if the depth of carbonation is larger.

There is of course a statistical uncertainty in the measured values. If one assumes that the carbonation depth of concrete with Skövde Std M, W/C = 0.60, is constant in the period of 7 – 25 years we get five values to use for a simple statistical calculation. The values are 6.0 3.8 5.5 5.3 and 3.8 mm.

Mean value is 4.9 mm. Standard deviation is 1.0 mm (Assume normal distribution.)

The 5 % fraction is 4.9 + 1.64 * 1.0 = 6.5 mm.

Also with a larger constant (due to the low number of results) the fraction value is still very small. For instance, k = 3 corresponds to not more than 8 mm carbonation depth.

# **4 RESULTS FROM THE CO₂ CYCLE PROJECT**

Lately a great interest has been shown for carbonation as a way to take up  $CO_2$  from the atmosphere and bind it to the concrete. A Nordic project " $CO_2$  Uptake during Concrete Life Cycle" was carried out in 2003 – 2005. At present a Swedish project " $CO_2$  Cycle in Cement and Concrete" is ongoing.

In the latter project carbonation measurements on concrete surfaces with different exposure conditions shall be carried out. So far measurements have been done in 2008 on the Smöjen prism samples, stored outdoors but sheltered from rain. The table below show results from these measurements for three different cements, one with two different W/C-ratios.

Determination of the carbonation depths with thin section analyse was done in order to verify the phenolphthalein method results.

Cement, year	Phenolphtha	lein method	Thin section method			
W/C-ratio	Upper side	Under side	Upper side	Under side		
Byggc, 2000 W/C = 0.60	2.2	5.3	To be tested	To be tested		
Std P, 1982 W/C = 0.60	1.3	12.3	1.5	8		
Std M, 1982 W/C = 0.60	7.5	12.7	10	12		
Std P, 1985 W/C = 0.80	11.6	23.2	5	23		

Table 2 Carbonation depths in mm for samples stored outdoors but sheltered from rain.

Designations according to EN 197-1:

Byggc = CEM IIA/LL-42,5 R Std P = CEM I 42,5 R

Std M = CEM II 42,5 RStd M = CEM II B/V 42,5 N

The carbonation depth is in all the cases substantially greater on the under side of the prism than on the upper side. This result is unexpected since the prisms were stored on wooden bars to let the air freely circulate around the prisms and they were not exposed to rain.

The carbonation depths on the under sides are larger than the corresponding values of the samples exposed to rain, which is expected. The upper sides are however not only less carbonated than the under sides but also sometimes less than the corresponding samples exposed to rain.

Suspicions arose that the prisms under some weather condition might be exposed to condensed water on the surface, leading to less carbonation on the upper side. A measurement of the relative humidity in the concrete close to the upper and lower surface for three prisms revealed however no difference. They were all in the range of 68 - 73 %.

The observed difference is therefore still to be explained. Difference due to concreting procedure or curing can be ruled out. It might be that humidity conditions were more even at the measuring (done in the early autumn) than otherwise, but it seems difficult to explain the large difference that was observed.

In the ongoing study,  $CO_2$  Cycle in Cement and Concrete, carbonation depths will be measured on a lot of structures, especially indoor, were lack of information is rather big due to absence of corrosion risk. The results will be available at the end of 2009.
# Does the EN 206-1 Exposure Classification Apply to Tunnel Concrete?



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## ABSTRACT

This paper gives an outline of chemical deterioration mechanisms affecting sprayed concrete used for rock support, and provides a discussion of the consequences. The most severe degradations, found in subsea tunnels, were due to a composite saline ground water attack in close association with acidification caused by reactions within Mn-Fe layered biofilms. Thaumasite Sulfate Attack associated with Alum Shale still represents a concern, whilst degradations within the freshwater environment are uncommon. The NS-EN 206-1 Exposure Classes did not predict the extent and rate of severe degradations in high quality mixes. Tunnel concrete is also influenced by effects of hydraulic gradients, bacterial activity and evaporation. As yet the standard does not mention sprayed concrete. Revised Exposure Classes should preferably include bio-degradation, bicarbonate and hydrogeological context, emphasising the importance of "Life Time Exposure Classification" for dynamic water environments.

**Key words:** Sprayed concrete, freshwater, Alum Shale environment, saline ground water, biodegradation.

# **1. INTRODUCTION**

Sprayed concrete used for rock support in tunnels differs from cast concrete in many respects. This material is special in that the main quality parameters are sensitive to the execution process and also because sprayed concrete is directly exposed to variable ground water and rock mass conditions. However, quality routines and good workmanship involving certification of nozzlemen has developed the technology. Modern sprayed concretes therefore usually display a quite uniform quality. Yet sprayed concrete layers are potentially influenced by instabilities within the surrounding rock masses as well as ground waters of variable chemical composition.

Steel fibre reinforced sprayed concrete is used as a final tunnel rock support method together with rock bolts within a wide range of rock mass conditions. Spray thicknesses vary from about 5 to 30 cm, and are usually designed on the basis of rock mass classification systems such as the Q-system (e.g. [1], and later updates). When the rock support has been established according to

rock mechanical principals there is still a possibility that chemical deteriorations may cause thinning and weakening of the sprayed concrete in the long run. Loss of bearing capacity might hence lead to destabilisation of the rock mass. Therefore relevant exposure classification is necessary to ensure long term safety of tunnels.

# **1.1** Background, motivation and objectives

The life time expectancy of sprayed concrete linings in Norwegian tunnels is fifty years, which corresponds to the designed life time. Durability investigations up to 1997 indicated that the situation was not alarming. Ca-leaching, carbonation and steel fibre corrosion was apparently restricted to very thin sprays (< 5 cm) and freeze-thaw damage was regarded as unimportant. The concrete strengths and mixes were in most cases found to comply with the design criteria [2]. However, the aggressive subsea environment was regarded as an important topic for future investigations. The experience up to 1997 is embedded in NB Publication 7 [3], which is the Norwegian specification for sprayed concrete for rock reinforcement. The specification was introduced already in 1979 and has been revised several times. This reflects the leading role Norwegian engineers have had in the development of modern sprayed concrete technology. Presently NB Publication 7 is under revision, mainly due to the need for harmonisation with the recent EN- 14487, EN 14488 and EN 14489 standards for sprayed concrete. Further details may be found in [4].

However, investigations by the present author from 2000 throughout 2007 have demonstrated that several deterioration mechanisms are affecting tunnel concrete. The mechanisms and structural impact summarised by [5,6], showed that lifetime of sprayed concretes located within certain chemically aggressive environments is much shorter than fifty years. Hence, it was necessary to compare these findings with the NS-EN 206-1 and establish whether the extent and rate of deteriorations was predictable in view of the Exposure Classes. If not predictable, it would seem logical to address the status of the standard and provide a basis for future revisions. The objectives of this paper were to:

- Summarise the characteristics of Norwegian sprayed concrete used for rock support, and establish the main environmental variables affecting concrete within tunnel space.
- Present the main deteriorations in sprayed concrete and the characteristics of associated aggressive groundwaters.
- Undertake exposure classification according to the NS-EN 206-1 Exposure Classes [7] and compare the actual Durability Classes with observed degradations.
- Discuss the findings vs. status of (NS-)EN 206-1 and propose improvements.

# 2 NORWEGIAN SPRAYED CONCRETE IN TUNNELS

# 2.1 Norwegian specifications for sprayed concrete versus investigated concrete

According to NB Publication No. 7 [3], Norwegian sprayed concrete for rock support should be made according to the following range of specifications:

- Wet method
- CEM I with added 4-15 % silica fume (SF) by cement weight or other pozzolana with a proven effect similar to SF

- Setting accelerators, usually Al-sulfate; previously water glass
- Strength Classes B25, 30, B35, B40 and B45. <u>B40</u> is not a NS-EN 206-1 Strength Class.
- Durability Classes M40, M45 and M60.
- Binder contents 400-530 kg/m³
- w/c = 0.60-0.40: mostly w/c = 0.40 0.45
- Steel fibre reinforcement for ductility (40-50 kg/m3)

NB Publication No. 7 and NS-EN 14487 utilises the NS-EN 206-1 Exposure Classes as a normative reference.

The studied sprayed concretes were all made according to the wet method, using a rapid setting Portland cement (PC rapid) or Sulfate Resisting Portland Cement (SRPC) with (1)- 5-16 % SF. Strength Classes and Durability Classes were B40 or B45 and M40 or M45, respectively. The binder contents varied from 485 to 580 kg/m³ and the w/c -ratios were ranging from 0.38 to 0.47 (0.50). Steel fibre contents were within the 30 to 60 kg/m³ range. In general it was concluded that the investigated sprayed concretes were complying with NB Publication 7 and NS-EN 206-1. They represent "real life" sprayed concretes made according to a quality scheme, and there was no indication that deteriorations were due to poor workmanship. A more specific relationship between Exposure Class, Durability Class and state of deterioration for individual cases is given in Table 2.

# 2.2 Context of sprayed concrete in tunnels

Steel fibre reinforced sprayed concrete usually takes the shape of the blasted rock mass behind and is characterised by a tortuous and rough outer surface. The surface of sprayed concrete frequently accumulates particles and biota and attracts moisture. This is a favourable environment for chemical reaction and growth of microbes [5,6]. The principal aspects of environmental loads acting upon sprayed concrete linings in tunnels are illustrated in Figure 1. The tunnel environment is characterised by differential mechanical loads from the rock mass, variably aggressive waters and variable hydraulic gradients. Drought in tunnels might also cause evaporation and lead to increased ionic strength of leakage waters within the tunnel space. Moreover, the effects from exhaust fumes cannot be excluded, although this was not studied.



Figure 1 – Environmental loads acting on sprayed concrete in tunnels. In addition the effects of evaporation within tunnel space should be considered.



Figure 2 – Surface deposits (rated) of calcite (leached Ca from cement paste) and iron  $\pm$  manganese oxides (Fe) in the Flekkerøy subsea tunnel, showing a positive correlation with hydraulic gradient (depth beneath sea level) (cf.[5,6]).

Investigations in a subsea tunnel have shown that Ca-leaching of sprayed concrete and Mn-Fe surface deposition increased significantly with the depth beneath sea level (Figure 2). Hence there can be no doubt that the magnitude of the <u>hydraulic gradient</u> represents a very important environmental variable affecting the sprayed concrete lining. In view of the rather thin concrete layers involved (frequently 5-10 cm), aggressives should enter more easily into the pore space of sprayed concrete than into thick layers of cast concrete [5,6]. This is mainly due to the differences in concrete water conductance, which increases with thickness of the concrete layer. In contrast to subsea tunnel sections, tunnels under land are influenced by a variable water table with a less constant water pressure. Shifting of ground water level also leads to variable redox conditions within the rock mass which may effect temporal changes in the water chemistry: It is well established that lowering of the water table leads to sulfide oxidation and formation of aqueous sulfate within Alum Shale [8].

Volumetrically large water leakages are rare in tunnels due to pre-grouting. Instead volumetrically small leakages with low flow rates are representative of the typical water loads on tunnel concrete. Such leakages may be spread over large concrete surface areas or be more focused. <u>Evaporation</u> has been found near tunnel fans [5,6] and the effect is most efficient in thin water films, corresponding to volumetrically small and spread water leakages. The evaporation rate is suppressed at high relative humidity (RH). However, RH in tunnels commonly range from 60 to 70 % [9], suggesting there is a significant potential for increased aggressiveness by evaporation.

## 2.3 An outline of the structural deterioration phenomena in sprayed concrete

The investigations indicated that, although most sprayed concretes still appear to be intact there are many examples of deeply weathered concretes which are in the process of loosing their integrity. Structural deterioration of sprayed concrete is characterised by cracking, spalling, leaching and degradation of the cement paste matrix. These features are almost exclusively related to the presence of water leakages acting from behind the sprayed layers and from the outer surface. Destructive steel fibre corrosion may take locally along cracks or more generally associated with secondary minerals (see chapter 3).

Chemical degradations may lead to 1) fallout of strong thick concrete slabs or 2) focused or widespread weakening and thinning due to leaching, spalling and formation of concrete debris (Figure 3). These mechanisms have caused structural damage at early age (< 5 years), although they mainly should be regarded as somewhat "long term processes". The first process is facilitated by cracking across the sprayed concrete layers leading to local transformations of the cement paste along cracks and the interface with the rock mass. Destructive steel fibre corrosion occurs, notably in the subsea environment, causing loss of sprayed concrete ductility. It is significant that the adhesion zone quite commonly is weak and sometimes permeable [11]. The second process involves bulk diffusion of aggressive waters from both sides of the sprayed concrete layers. This process has caused significant thinning of the sprayed concrete layers, typically occurring within the saline ground waters environment.



*Figure 3 – Two principal types of structural deterioration in sprayed concrete linings.* 

## **3** DETERIORATIONS RELATED TO THREE MAIN EXPOSURE CATEGORIES

#### 3.1 General

The exposure conditions reported by [5,6] were categorised into three main groups; a) freshwater in land tunnels and land sections of subsea tunnels, b) Alum Shale ground water with sulfate, and c) saline ground waters within subsea tunnel sections. The investigation strategy was very similar to the recommendations by the International Tunnelling Association (ITA), Working Group 12 focusing on four durability aspects, namely 1) Complete information about the exposure conditions, 2) Sprayed concrete material information, 3) Duration of exposure and 4) Design basis and lifetime expectancy compared to specifications and work execution [10]. Water chemical analyses were performed and samples of variably deteriorated sprayed concretes and concrete spalls and debris were investigated in thin section by a polarising microscopy, X-ray diffraction (XRD) and Scanning Electron Microscopy (SEM). Selected samples were also investigated by Electron Microprobe Analysis (EMPA) and stable isotopes. The detailed documentation is given in [11,12,13,14,15].

The present short summary focuses on the main diagnosis and related water analyses given in Table 1, serving as background for exposure classification and systematic comparison with degraded concretes of known composition (Chapter 4).

Table 1 – Chemical & stable isotope data for waters. Subsea tunnel samples in <u>italics</u> were collected from fresh waters under land. Water chemical analyses from the previous Blindtarmen test site from [16] (I to IV) and [17]. * = waters with some interaction with concrete; ** = waters with extensive interaction with concrete (upstream from sample stations). Ammonium concentrations were generally low (max 8 mg/L).

Samples	Characteristics	δ ¹⁸ Ο	δ ² H	рН	Cl	Na ⁺	$\mathbf{K}^{+}$	Ca ²⁺	Mg ⁺	SO4 ²⁻	HCO ₃ ²⁻
		‰	‰		mg/L	mg/L	mg/L	mg/L	mg/L	mg/L	mg/L
Subsea tunnels		SMC	OW								
Flekkerøy 1**	Water shield w/Mn (?) bacteria	-4.4	-37	7.39	27900	12800	294	3100	1420	2240	43
Flekkerøy 2	Water shield, large leakage	-0.8	-4.5	8.07	18700	10200	396	429	1310	2600	146
Flekkerøy 3*	Water shield w/Fe bacteria	-1.2	-10.8	7.98	18300	9530	297	1020	1180	2500	119
Flekkerøy 4*	Water shield w/Mn bacteria	-3.7	-28.3	8.02	11100	5080	136	1060	754	1380	117
Flekkerøy 5*	Interface rock/concr w/ Fe bact	-2.6	-23.4	7.81	17500	8300	193	1920	1010	2300	84
Byfjord 1*	Water shield; w/possible Fe bact	-0.4	-15.9	7.55	50200	27800	507	1660	3280	3830	137
Byfjord 2**	Via degraded concrete surface	-6.2	-47.4	7.56	11400	5720	50.7	1280	571	1250	41
Byfjord 3*	Water shield	0.1	-6.1	7.75	25400	14500	483	596	1790	3300	151
Byfjord 4*	Water shield	-6.7	-48.8	8.33	260	193	14	15	15.4	51.2	141
Oslofjord V1*	Via degraded surf.;Mn-Fe bact	-0.1	-1.8	7.91	19100	9170	307	473	1260	2640	132
Oslofjord V2*	Via degraded surf.;Mn-Fe bact	n.a	n.a	7.89	19300	9300	308	472	1210	2670	137
Oslofjord V3	Crack in concrete, large leakage	-0.3	-0.4	7.88	19100	9180	313	460	1270	2640	146
Oslofjord V4*	Crack in concrete w/Mn-Fe-bact	0.6	1.9	7.93	18900	9140	316	458	1260	2610	146
Oslofjord V5	From rock joint	-0.4	-5	7.93	18600	9060	318	442	1240	2580	150
Oslofjord V6**	Ditch under degraded; Mn-Fe-b.	0.8	2.6	7.71	18200	9480	340	484	1321	2550	179
Oslofjord 1**	Degraded surface w/Mn-Fe bact	0.4	-4.2	6.60	n.a	8940	163	1310	1320	2630	n.a
Oslofjord 2**	Degraded surface w/Mn-Fe bact	-1.7	-10.1	6.64	18300	8700	160	1360	1320	2604	161
Oslofjord 3**	Degraded surface w/Mn-Fe bact	-1.1	-5.6	6.69	18300	8640	184	1110	1340	2720	123
Oslofjord F1*	From rock mass w/ Fe-bacteria	n.a	n.a	7.80	18500	8990	318	435	1240	2550	139
Oslofjord F1-2005*	Ditch below F1 w/ Fe-bacteria	-0.8	-13.7	7.30	17800	9740	316	419	1230	2440	179
Oslofjord V10	Average water at pump station	-5.1	-32.9	7.67	9990	5110	161	370	725	1410	135
Oslofjord V10II	Average water at pump station	-4.8	-40.5	7.60	10600	5690	155	391	763	1450	144
Oslofjord 17450**	Below degr. surf. w/Mn-Fe-bact	-1.0	-14.3	7.49	17800	9640	262	547	1270	2410	154
Oslofjord T4-1	Crack in concrete	-0.4	-4.3	7.83	16900	9280	348	394	1180	2740	135
Oslofjord T4-2	Ditch below T4-1 w/ Fe-bact	-0.6	-4.3	7.82	17500	9320	349	398	1190	2840	135
Oslofjord SOS31	Ditch water	-10.2	-74.7	8.03	9.4	12.8	2	40	4	13.6	139
Oslofjord SOS 8	Rock water, slow	-10.5	-74.2	8.16	59.6	63	2	31	6	21.1	165
Oslofjord B1	Rock water large basin	-11.1	-81.5	7.43	123	76.3	3	21	3	13.3	64

2	1	7
7	4	/

Oslofjord A2*	Stalactite water in saline zone	n.a	n.a	7.00	1910	1040	16	131	57	334	n.a
Freifjord 6600**	Drain w/Mn-Fe bacteria	n.a	n.a	7.00	19900	8550	37	1680	1310	2710	101
Freifjord 6650**	Dropping on c surface/Mn-Fe b.	n.a	n.a	6.50	19200	6700	33	2950	1250	3130	n.a
Freifjord 3135*	Ditch	n.a	n.a	7.00	24	168	6	13	1	146	n.a
Freifjord 5910**	Water shield w/Fe-Mn bacteria	n.a	n.a	6.00	17100	7300	34	2480	1270	1930	n.a
Freifjord 4930**	Water shield under Mn-Fe bact	n.a	n.a	5.50	5680	2050	11	1610	205	410	n.a
Freifjord 5700**	On surf. w/Mn-Fe-bact	n.a	n.a	6.00	17100	8080	23	1980	1220	2230	n.a
Freifjord 4890*	Via degraded surf. w/Mn-bact	n.a	n.a	6.00	5110	1680	14	1340	237	424	n.a
Freifjord 6915*	Drain under Mn-Fe bact	n.a	n.a	5.50	15700	6580	34	2420	1170	1780	n.a
Freifjord 4930-1**	Water shield under Mn-Fe bact	-15.5	-115.1	6.88	6500	2100	10.2	1600	213	510	23
Oslofjord seawater	60 m depth NIVA Solbergstrand	0.2	-0.77	7.74	18600	10800	390	413	1370	2630	144
<u>Freshwater</u>											
Lier 44265	Drain in railroad tunnel	-11.8	-82.1	8.18	2.8	15.0	0.8	44.5	12.1	22.3	173
Harpefoss 1	Crack in concrete	n.a	n.a	5.70	16.9	3.2	7.8	59.9	2.3	111.0	n.a
Harpefoss 2	Crack in concrete	n.a	n.a	5.67	13.2	3.5	4.4	38.5	2.8	106.0	n.a
Harpefoss 3	Crack in concrete	n.a	n.a	5.66	15.8	11.0	5.0	58.5	1.5	102.0	n.a
Harpefoss 4	Crack in concrete	n.a	n.a	6.06	12.8	3.4	n.d.	38.9	3.9	105.0	n.a
Alum Shale water, O	<u>Islo</u>										
Åkeberg 1*	Drainage hole, TSA aff. concr.	n.a	n.a	7.60	29	26.0	22.0	615.0	110.0	1841	274.0
Ekeberg 1**	Stalactite water, TSA aff. concr.	n.a	n.a	7.00	10	25.0	16.0	106.0	20.0	592	100.0
Ekeberg 2**	Crack in TSA affected concrete	n.a	n.a	7.00	10	43.0	22.0	574.0	74.0	2031	56.0
Svartdal 320-06	Pond below TSA aff. concrete	-11.3	n.a	6.84	18.7	44.4	9.1	172	21.4	541	66.8
Alum Shale water, B	lindtarmen Test Site - Oslo 1952 - 19	<u>73</u>									
I	Ground water (isolated from air)	n.a	n.a	5.38	421	n.a	n.a	n.a	n.a	2772	n.a
II	Ground water (isolated from air)	n.a	n.a	6.14	491	n.a	n.a	n.a	n.a	653	n.a
III	Ground water (isolated from air)	n.a	n.a	5.00	343	n.a	n.a	n.a	n.a	2530	n.a
IV	Ground water (isolated from air)	n.a	n.a	4.89	347	n.a	n.a	n.a	n.a	2523	n.a
Sept 55 B	Bottom of tunnel	n.a	n.a	6.22	420	n.a	n.a	n.a	n.a	2772	n.a
22/5/63 B	Bottom of tunnel, oxidised	n.a	n.a	3.91	210	37	7	760	110	4116	n.a
22/5/63 H1	Hole I oxidised ground water	n.a	n.a	2.95	190	37	10	740	150	4284	n.a

#### **3.2** Freshwater environment

The concretes in contact with freshwater loads were visually mainly intact. Local Ca-leaching and calcite stalactite formation was observed. The water analyses reported in Table 1 indicate somewhat elevated sulfate concentrations < 150 mg/L, which is less than Exposure Class XA1. However there is some concern related to impact of sulfate attack. This is due to the fact that secondary ettringite and thaumasite can be stabilised at rather low sulfate concentrations: In fact a local thaumasite attack at Harpefoss, detected when this sprayed concrete was 16 years old, [12] had formed at sulfate concentrations about 110 mg/L. In the Lier railway tunnel there was evidence of an old sulfate attack (ettringite-thaumasite) which took place at a time when leakages were abundant. Presently the tunnel concrete is typically dry due to a permanent lowering of the water table. The single water chemical analysis from this tunnel shows a very low sulfate content, which if representative, might also indicate that the sulfate source within the rock mass has been exhausted [9]. The present problem is due to extensive spalling and falloff, related to very extensive outer carbonation. The Lier sprayed concrete was made in 1965-1971, representing old technology of no direct relevance to a study of the durability of modern concrete. The investigations within the freshwater environment were not very comprehensive because other environments were obviously more important.

## 3.3 Alum Shale environment

The Norwegian Alum Shale Committee (1947-1973) concluded that the well known quick cement paste degradation in the Oslo region was due to secondary ettringite and variable impact from acid attack [8]. However, this has recently been refuted. Petrography of young sprayed concrete used for rock support and old test samples from the previous "Blindtarmen" test site (1952-1982) has demonstrated that this classic "ettringite case" in fact was caused by Thaumasite Sulfate Attack (TSA) in close association with decalcification and internal detrimental carbonation in the form of Popcorn calcite and other relatively coarse calcite. There was also a variable impact of acid attack. Non-deleterious Thaumasite Formation (TF) was quite common, suggesting TSA is under way several places. Modern cases are represented by the Åkebergveien road cut and the Ekeberg and Svartdal road tunnels (Table 1) [11,12,14].

Thaumasite growth requires a source of sulfate and carbonate, usually a cool and moist environment and otherwise utilises Ca and Si in the cement paste matrix. TSA leads to partial or complete degradation of the cement paste matrix, in contrast to expansion and map cracking caused by ettringite. Recent investigations have proven that extensive Ca-leaching and calcite formation greatly facilitates the development of full scale TSA [14]. Oxidation of Alum Shale sulfides (pyrrhotite and pyrite) was previously regarded as the main source of sulfate ions, whilst research by the present author has demonstrated that also primary anhydrite in the shale may contribute significantly to the aqueous sulfate concentration. The sulfide oxidation mechanisms are complex, and bacterial oxidation assisted by *Acidithiobacillus species* appears to play a significant role [18]: These investigations have also shown that the source of thaumasite carbonate is both bicarbonate derived from calcite dissolution and atmospheric CO₂.

Water chemistry implies that sulfate concentrations within the 500-4000 mg/L range, coupled with ordinary bicarbonate contents of around 100-275 mg/L, are responsible for the overall <u>TF-TSA-carbonation process</u>. Associated ground water pH ranges from circum neutral to strongly acidic (pH < 3). Lower pH causes efficient leaching of the outer cement past matrix and an increased porosity, making the overall concrete more open for sulfate and bicarbonate ions. The

overall rate of the TF-TSA-carbonation increases in presence of acidic water where thaumasite and Popcorn calcite form in an intermediate position between pristine paste and the acid leached domains [14].

In reality the chemical composition and acidity of Alum Shale ground water varies at each site, as shown by the analyses from the Blindtarmen test site (Table 1). The important lesson is that ground water compositions under land are not constant. They vary over time reflecting the complex chemical interaction between jointed rock mass and a shifting water table, likely responding to variations in precipitation and drying. Alum Shale represents Exposure Class XAS (NS-EN 206-1; NA.11) and sprayed concretes are now usually made with SRPC and silica fume in line with the findings of the previous Alum Shale Committee. These concrete mixes have countered the severe attacks. However, Hagelia et al.[11,12] reported on the occurrence of TF and TSA in a modern steel fibre reinforced concrete less than 2 years old (Svartdal highway tunnel) and the overall TF-TSA-carbonation process had caused spalling, thaumasite-calcite mush formation and destructive steel fibre corrosion after less than 13 years at the Åkeberg road cut (Figure 4). There was no indication of internal sources related to the concrete aggregate. For this reason TSA still represents a concern within the Alum Shale provinces.



Figure 4 – Spalling after less than 13 years in a 1987 steel fibre reinforced concrete (SRPC and SF), due to TSA and carbonation due to PCD. A previous spray had been severely affected and was replaced after 17 years in service. Alum Shale environment, Åkebergveien Oslo.

Popcorn Calcite Deposition (PCD) is another concern where bicarbonate bearing ground waters enter deep into concrete in a through solution process. This sort of carbonation is different from ordinary surface carbonation in that it forms relatively coarse calcite grains as a direct consequence of decalcification of CSH, hence influencing the strength. In contrast, ordinary surface carbonation only reaches a few mm in and is essentially harmless unless it reaches steel reinforcement. Steel fibres in sprayed concretes are always corroded within the surface carbonated zone [2], whilst effects of PCD can have detrimental effects even in absence of TSA [12,14].



Figure 5 – Lifetime of sprayed concrete under a hydraulic gradient is potentially shorter than concrete immersed under hydrostatic conditions, such as the Blindtarmen test samples. Thick continuous line represents 10 cm thick sprayed concrete (SRPC and 5 % SF) the thick stippled line 10 cm thick Blindtarmen test prisms (PC and 15 % SF)[14].

Comparison of deteriorated zones ("affected thickness") with initial thicknesses can be used as a proxy to life time studies (Figure 5). Sprayed concrete on Alum Shale at near neutral pH was somewhat more affected by the TF-TSA-carbonation process than the Blindtarmen test specimens that had suffered events of acid attack. Modern sprayed concrete in contact with Alum Shale and additional sulfuric acid attack can be expected to remain in service for less than 20 years, in contrast to some 20 to 30 years at pH neutral conditions [14]. The combined effects of acid attack and TSA on sprayed concrete influenced by a hydraulic gradient remains to be studied directly. Yet the effect is less efficient when spray thicknesses exceed 10-15 cm [12].

# 3.4 Saline ground water environment with localised development of hypersalinity

#### A novel mechanism involving bacterial degradation

A novel sprayed concrete deterioration process has been discovered in Norwegian subsea tunnels (e.g. [13]). The process ultimately resulted in complete disintegration of the cement paste matrix and destructive steel fibre corrosion (Figure 6), even in the strongest concrete (Strength Class B45) [5,6]. The composite reaction mechanism comprised 1) acid production due to redox reactions within layered Mn-Fe biofilms inhabited by *Leptothrix* and *Gallionella* species forming biominerals, and 2) infiltration of saline Cl⁻, Mg²⁺, SO₄²⁻ and HCO₃⁻ enriched ground water, leading to formation of magnesium silicate hydrate, Thaumasite Sulfate Attack, Popcorn Calcite Deposition (PCD) and associated precipitation of brucite, Mg-calcite, aragonite and gypsum. The development of these secondary minerals generally decreased the acid resistance of the concrete. The bacterial acid attack initially acted from the outside surface, whilst the saline infiltration acted from both sides of the spray. Eventually the acid attack also entered the interface against the rock mass.

In contrast to static conditions, the hydraulic gradient facilitated magnesium attack several cm inside sprayed concrete, causing shrinkage, increased inflow of aggressives and consumption of hydroxyl ions. Constant replenishment of saline Mn and Fe bearing waters resulted in continuous formation of layered biofilms and a sustained attack. The deterioration had caused

outer debris formation and thinning of sprayed concrete layers at rates varying between < 0.5 up to 5-10 mm/year, leading to deep and complete disintegration of the cement paste matrix after less than five years at several places. These rates were estimated on the basis of visual observation where an approximate control level could be established. However, in absence of the Mn-Fe biofilms the attack by saline infiltration was mostly slower. At an advanced stage gypsum had formed extensively at the expense of cement paste, building up crusts on the outer sprayed concrete surfaces. Gypsum crust thicknesses have been found to exceed 5 cm, forming either within/underneath rusty bacterial slimes or as precipitates. Such crust material should be removed prior to repair sprays in order to avoid sulfate attack soon afterwards.

Steel fibres delivered  $Fe^{2+}$  to the acid producing reactions, and should therefore be abandoned in subsea tunnel concrete. The steel fibre corrosion leads to a loss of sprayed concrete ductility which is a very important property when used for rock support. In general steel fibre corrosion is also triggered by more familiar mechanisms, such as chloride penetration, carbonation, and Mg-attack [5,6,13,15]. An interesting "pre-rusting" effect was found in most subsea sprayed concretes: Steel fibres in freshly cut and washed core material from sound concrete were mostly intact and shiny but exhibited pronounced surface corrosion after a few hours. In contrast steel fibres in concrete from the freshwater environment remained unaffected.



Figure 6 – Old core locations in the Freifjord subsea tunnel showing extensive breakdown of the sprayed concrete 16 years later. Rusty and black areas (right) consist of iron oxyhydroxides from Gallionella ferruginea and manganese oxides derived from Leptothrix sp. Grey deposits consist of gypsum formed from Ca in concrete and sulfate from seawater.

#### Significance of salinity, hypersalinity and evaporation

The ground waters in the subsea tunnel sections appear to represent mixtures of fresh water and seawater like saline ground waters in different proportions, with some effects from interaction with rock mass. However some drain waters collected from aluminium water panels in the Flekkerøy and Byfjord tunnels were much more saline than seawater (hypersaline). These waters were exposed to drought near busy tunnel fans and represent evaporation, as is also indicated by their Na/Cl ratios which are similar to seawater or any mixture of seawater and freshwater (Figure 7). Apparently some sodium at some localities has been bound within the rock mass, possibly due to exchange with clay minerals. Also sample 6650 (Freifjord) leaking over concrete indicated some degree of hypersalinity in direct contact with concrete (Table 1). Most waters on water panels had drained over concrete surfaces before they entered the panels. In contrast to aluminium sheaths, cement pastes certainly also had absorbed Cl, Na, Mg and sulfate, as shown by SEM analysis [15]. Leakages showing extensive interaction with concretes influenced by the composite bacterial and saline water attack (indicated with ** in Table 1) had been strongly enribed in Ca relative to background water (e.g. seawater, rock water and fast leakages through concrete cracks with Ca = 394-460 mg/L). Ca concentrations also increased downstream: Water sample "Oslofjord 3", from about 1 m downstream from a leakage entrance point, had Ca = 1110 mg/L whilst "Oslofjord 2"; 2 m further below had reached 1360 mg/L. This excess Ca must be be derived from from the cement paste, since this particular location showed extensive cement paste degradation due to Mn-Fe biofilms and saline water attack.



Figure 7 – Chloride and sodium variation in waters collected in subsea tunnels and land tunnels. Circles = Byfjord; triangles = Freifjord; diamonds = Flekkerøy; small diamonds = Oslofjord, and square = seawater. Big arrow trend of evaporation.

#### 3.5 Characterisation of waters by hydrogen and oxygen stable isotopes

Stable isotopes are extensively applied within environmental fields. Hydrogen and oxygen isotopes were in this case used in order to distinguish between origins of waters, with focus on hypersaline waters. The main issue related to the EN 206-1 exposure classification is to find out

the potential for development of "excess" aggressiveness due to evaporation within tunnel space. In water the heavy ²H (deuterium) and ¹⁸O isotopes make up small fractions, whilst ¹H and ¹⁶O are far more abundant. Due to the differences in weight (e.g. molecular bonding energies) isotopic fractionation takes place, reflecting different processes and origins. The isotopic composition of H and O is expressed as the ratio of the heavy to light isotopes relative to a standard composition, referred to as "Standard Mean Ocean Water" or SMOW. A further introduction to the basics of stable isotope systematics may be found in [19]. Figure 8 shows a plot of oxygen isotopes versus hydrogen isotopes, presented as  $\delta$  values in % of the deviation from SMOW. According to convention SMOW plots at (0,0) and the real seawaters are not very different (see Table 1). All analysed samples plot on a trend which indicates an overall mixture of seawater like ground water (near 0,0) and freshwaters (at very negative values). This trend nearly coincides with seawater, falling below the Meteoric Water Line (MWL), which represents river water, rain and snow from all over the world. The seawater like ground waters (cf. chemical compositions in Table 1) with  $\delta^{18}$ O and  $\delta^{2}$ H within 0 to - 1 ‰ and 0 to - 5 ‰ ranges, respectively, are strongly predominated by the seawater component, whilst the most negative signatures mainly represent freshwaters. Freshwaters always have quite negative H and O isotopic compositions and melt waters derived from snow and ice are most depleted in the heavy isotopes.



Figure 8 – Stable H and O isotopes of waters collected in subsea tunnels and freshwater tunnels. The open squares represent strongly evaporated waters on the lower side of the main trend. Arrows corresponds to the slopes of evaporating waters from two arbitrary initial compositions along the freshwater – seawater mixing line (thick line and regression). Open squares; evaporated hypersaline waters. Thin stippled line is the Meteoric Water Line.

Evaporation of any water composition will lead to enrichment in their heavy isotopic compositions, because the light isotopes prefer the vapour. Accordingly the H and O compositions in remaining waters become progressively more positive as evaporation proceeds. Evaporated waters derived from different starting compositions fall below the actual freshwater – seawater mixing line on separate trends with similar nearly constant slopes, as illustrated in Figure 8. Initial compositions prior to evaporation are somewhere along or near this mixing line. In general all of the hypersaline waters fell below the main seawater – freshwater mixing line in

keeping with evaporative effects. The hypersaline water from the Flekkerøy subsea tunnel ("Flekkerøy 1") had a very negative composition in comparison with seawater, and originated from a water mixture with a significant portion of freshwater. This means that the initial water composition before evaporation most likely was brackish water. Similarly the most evaporated water from the Byfjord subsea tunnel ("Byfjord 1") extrapolated backwards to the main mixing line show it was derived from an isotopic composition which is characteristic of brackish water. "Byfjord 3" had the largest seawater component among the hypersaline waters. Thus, undoubtedly the effects of evaporation within tunnel space can increase the waters agressivity against concrete very extensively. This will be elaborated further on in the next chapter dealing specifically with EN 206-1 Exposure classification.

# 4 EXPOSURE CLASSIFICATION ACCORDING TO NS EN 206-1

# 4.1 Introduction

Table F.1 in EN 206-1 is not used in Norway. Instead Table NA.11 has been introduced in National Annex NA.F, showing the relationships between Exposure Class and Durability Class. Table NA.11 also introduces Exposure Class XSA nationally for "Especially aggressive environment", such as Alum Shale ground water, and XA4 for manure cellars. According to [3] sprayed concrete for rock support does not require classification of frost environment. NS-EN 206-1 states that if a relevant environmental classification has been made and the mix design complies with recommendations in this standard, then the concrete life time should be 50-100 years. Obviously this was not always the case.

# 4.2 Classification and comparison with real life tunnel concrete

The classification was based on water chemistry reported in Table 1 and documentation of initial concrete quality [2,9]. The durability status of several sprayed concretes up to 2008 was compared to Strength Classes and Durability Classed as derived from the concrete quality documentation. Table 2 gives a summary of the results. Most samples were cores from 2 to 16 years old concrete. Judgement about the durability status was based on detailed petrography as well as visual inspections. It should be noticed that although the degradations are typical, they are overrepresented in the summary. In general the choice of Exposure Classes for corrosion caused by carbonation and seawater was a bit tricky, because no verbal statement in the standard refers explicitly to sprayed concrete and tunnel environments.

#### Freshwater environment

In the few included examples corrosion class XC2 by carbonation was considered as the most close to the verbal statement in the standard. Much of the concrete was dry and X0 is then relevant classification. Concretes from Harpefoss, Freifjord, Byfjord and Oslofjord were considered, mainly representing M45. Only the concrete at Harpefoss was marginally affected by TSA with only small structural effects after 16 years in service. According to [12] early leaching with surface efflorescence of calcite and thenardite occurred. However there are some uncertainties regarding the initial quality, and the Durability Class of the concrete was M45-M60. Yet it might be concluded that the durability of sprayed concrete in the freshwater environment is in agreement with the predictions implied by NS-EN 206-1. These concretes had a higher designed quality than strictly demanded by the chemical exposure conditions.

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Table 2 – Comparison of NS-EN 206-1 classes and real life durability of sprayed concrete used for rock support. Concrete sample numbers from [15]. Deterioration mechanisms: Mn-Fe & saline = bacterial acid attack and saline gr. water load; Saline = saline gr. water load without acid; TF-TSA-PCD = Thaumasite Formation-Thamasite Sulfate Attack-internal carbonation. Destructive steel fibre corrosion in extensive attacks. Ordinary surface carbonation was always present.

Location (age) &	Water samples	Exposure	Strength	Durability	Real life durability & influence of	Deterioration
concrete samples	(Table I)	Class	Class	Class	mechanism	mechanism
Subsea sections						
Flekkerøv (1988)						
1373 1374	1 (hypersaline) 5	XC2 XS2 XA3	B45	M45	Extensive thinning: $> 5$ cm gypsum crust	Mn-Fe& saline/ovnsum
1070, 1074	3	XC2, XS2, XA3	B45	M45	Thinning?, weak paste, 4-6 cm deep Mg	Mn-Fe & saline
Byfjord (1990-1991)						
1-2 at 2868 m	1&3 (hypersaline)	XC2, XS2, XA3	B45	M45	Good performance, local Mg, 14 yrs	Saline (minor Mn-Fe)
Visual at 2862 m	2	XC2, XS2, XA2	B45	M45	Good performance, leached surface, 14 yr	Saline (minor Mn-Fe)
Oslofiord (1998)						
Slab H2004	123	XC2 XS2 XA3	B45	M40	Replaced after 5 years service: >5 cm Mg	Saline
Ki I. II. II. IV. V	1.2.3	XC2, XS2, XA3	B45	M40	0.5-5 cm loss of thickness: age $< 5$ vrs	Mn-Fe & saline
Ki 1. 2. 3. 4. 5	V1 trough V6	XC2, XS2, XA3	B45	M40	Possible loss, some paste transformation	Mn-Fe & saline
Ki 6, 7, 8, 9, 10	V1 trough V6	XC2, XS2, XA3	B45	M40	No loss, weak transf. paste: Mg deep	Saline
Spall V5	V1 trough V6	XC2, XS2, XA3	B45	M40	Extr. friable, > 6cm compl. transformed.	Mn-Fe & saline
Freifjord (1990-1991)						
Visual at 4930	4890, 4930-1	XC2, XS2, <u>XA1</u>	B45	M45	Extensive paste degradation, ages 5-16 yrs	Mn-Fe bact + saline
5700, 5800	5700, 5910	XC2, XS2, XA3	B45	M45	Some thinning and internal degradation	" – w/ gypsum crust
6080	5910	XC2, XS2, XA3	B45	M45	Some thinning and internal degradation	" – w/ gypsum crust
6150	5910, 6650 (hyp)	XC2, XS2, XA3	B45	M45	<u>4 cm deep Mg attack (thinning?), 13 years</u>	Mn-Fe & saline
Alum Shale						
Structures in Oslo						
Åkebergveien (1987)	1	XC2 XAS	$\approx B45$	$\approx$ M45	Spalling and localised mush formation	TF-TSA-PCD
Cores 1 2 3 4	1	1102, 1110	DIS	10115	after < 13 years Still in service: 21 years	11 1511100
Ekeberg (1992): C 1 2	1.2	XC2. XAS	$\approx$ B45	M45	Leaching, stalactites, some friable paste	TF-TSA-PCD
Svartdal (1998): Cores	320-06	XC2. XAS	B45	M40	Leaching, small structural effects after 8	TF-TSA-carbonation
320A,B; 370A,B;		- , -~	-	-	vears, process started at $< 2$ yrs. some	
3200,N; 370H,V					friable paste	
· · · ·	Table continued o	verleaf				

Location & concrete samples	Water samples (Table 1)	Water samplesExposureStrengthDurabilityReal life durability & influence(Table 1)ClassClassClassmechanism		Real life durability & influence of mechanism	Deterioration mechanism	
Freshwater						
Harpefoss (1984); 1,2,3	1.2.3 &4	XC2, (X0)	No docum.	M45-M60	Small local effects after 16 years	TF-TSA-carbonation
Freifjord (1990); 3132	3135	XC2, (X0)	B45	M45	High quality, no effect	None
Byfjord (1990); 3	4	XC2, (X0)	B45	M45	High quality, no effect	None
at 4410 m						
Oslo (visual inspections)	SOS8, SOS31,B1	XC2, (X0)	B45	M40	High quality, no effect	None

#### Alum Shale environment

This environment is by definition XSA and due to frequent moist conditions XC2 seemed relevant. The Durability Class of sprayed concrete at the Åkebergveien road cut and Ekeberg tunnels was around M45, whilst the concrete in the Svartdal tunnel classifies as M40. NS-EN 206-1 gives a relevant environmental classification but since the effects of the TF-TSA-carbonation process still results in some structural disintegration after quite a few years this represents a concern. Yet the XSA Exposure Class requires Durability Class M40, which was not fulfilled for the most affected concrete at Åkebergveien. However, the effect of early sulfate attack (< 2 years) in the Svartdal M40 concrete suggests some problems related to formulation of an optimal mix still remains. As stated in Figure 5, the effects of acidic Alum Shale ground water should be more aggressive, and have not yet been studied.

#### Saline environment

The saline environment was mainly characterised by Exposure Classes XC2, XS2(3?), XA3. The Durability Classes of sprayed concrete were M45 except for M40 in the Oslofjord subsea tunnel. Table 2 shows that extensive deteriorations occur at places. Sample H2004 represents sprayed concrete within the service tunnel down to the pump station. This concrete was strongly weathered and influenced by saline ground water attack with extensive Mg penetration (brucite and MSH) and development of TSA and PCD. No acid attack related to Mn-Fe biofilm was involved here. The life time of this approximately 10-12 cm thick spray was only 5 years, as it was demolished and replaced in 2004. Presently the new concrete is characterised by extensive water penetration, being very well developed already by the end of 2005.

Although Mg attack with or without TF-TSA and PCD has influenced subsea concrete deep inside, additional acid attack related to bacterial activity definitely had the most severe effects. This composite attack has led to severe cement paste deterioration with extensive debris formation in concretes belonging to Durability Classes M45 and M40, even in waters belonging to Exposure Class XA1 (Freifjord tunnel location 4930). The late stage outer gypsum deposits related to the acid production had formed in thicknesses reaching at least 6 cm in the Flekkerøy subsea tunnel at one location, but were uncommon in the young Oslofjord subsea tunnel. Previous reports [2] suggest that the capillary suction of the Freifjord sprayed concrete is somewhat elevated, which might be one of the factors assisting degradation here. The composite bacterial – saline attack appears to be present in most subsea tunnels. However, sprayed concrete in the Byfjorden subsea tunnel performs well. This is most likely due to the overall thicker spray here (10-15 cm) and much lesser abundance of Mn-Fe biofilms.

At one location in the Oslofjord subsea tunnel (Spall V5) sprayed concrete influenced by Mn-Fe biofilms and saline ground waters was extremely friable: This big spall (5-6 cm thick) was collected in wither 2004 and could easily be broken off, leaving a visible "scar". When returning to the location after two months degradation in adjacent concrete had been so fast there was no sign of the spalling any longer. Petrographic investigations showed that quite coarse calcite, Mn-oxides and ferric iron compounds had been deposited throughout the spall, which in fact disintegrated extensively while drying in the laboratory. This concrete has as yet not been replaced.

The most severe attacks outlined in Table 2 had occurred in concrete at least 100 m below sea level. Thus there is a complex relationship between the chemical loads and the "driving force" represented by the hydraulic gradient.

Preliminary data on compressive strength (P. Hagelia in prep) have shown that as long as the saline water attack involving Mg substitution, thaumasite and internal carbonation is rather

scattered the strength is not very significantly influenced (yielding about 40 -55 MPa). However when these secondary minerals exceed some 40-70 % of the cement paste matrix the strength drops by about 50 % or more.

Based on the compiled data in Table 2 it can be stated that, despite that initial concrete properties largely complied with the specifications given in NS-EN 206-1, the extent of the attacks were by no means predicted by this standard. It should be emphasised that the investigated tunnels are still (in 2009) only 11 to 21 years old.

## 5 DISCUSSION OF NS-EN 206-1

#### 5.1 Evaporation and classification

Evaporation in tunnel space leads to increased salinities and aggressiveness of waters with implications for exposure classification according to NS-EN 206-1. Jørgensen [20] established experimentally the relationship between chloride and stable isotope enrichment during evaporation. Using these results is was possible to estimate the initial salinities of evaporated hypersaline waters assuming that the initial water composition fall close to the seawater – freshwater mixing line (Figure 9). Although this is a strait forward and conventional application of H isotopes, it seems possible that additional processes might have contributed to the scatter near the mixing line. The uncertainty may reach about 2500 mg/L on the Cl scale. Yet this does not alter the main conclusion: In fact the most evaporated sample appears to have increased the chloride contents from about 5000 mg/L to 50000 mg/L upon evaporation.



Figure 9 – Chlorine concentrations versus H isotopes of waters collected in subsea tunnels. Stippled arrows represent effects of evaporation. Thin stippled line is the mixing line of seawater and freshwater and thick arrows indicate initial salinities prior to evaporation.



*Figure 10 – Chlorine vs. magnesium showing trends of evaporations projected onto the EN-206-1 Exposure Classes.* 



*Figure 11 – Chlorine vs. sulfate showing trends of evaporations projected onto the EN-206-1 Exposure Classes.* 

Thus, having established the approximate chloride contents in the hypersaline waters prior to evaporation, trajectories of evaporative effects were then plotted on Cl vs. Mg and Cl vs.  $SO_4^{2^-}$  diagrams and compared to the Exposure Classes (Figures 10 & 11). Evidently evaporation can cause a 100 % to 1000 % increase in concentrations. Judging from the length of the trajectories it can be envisaged that low saline waters less aggressive than XA1 can develop into highly aggressive waters belonging to Exposure Class XA3 at very high ionic strengths.

For obvious reasons evaporation influences waters on concrete surfaces as well as metal water panels to a similar extent. Therefore, wherever possible, tunnel fans should be installed at a sufficient distance from widespread aggressive leakages. Undoubtedly unfavourable location of tunnel fans may directly contribute to unnecessary aggressive loads and a shorter service life of sprayed concrete.

## 5.2 Significance of dynamic water environments

The present investigation has clearly demonstrated that the tunnel environment is characterised by a dynamic water system, implying that the water loads change character from location to location and from time to time. The main features can be summarised as follows:

#### Natural background

1) Hydrogeological changes under the sea; frequent lowering of volumetric flow rate due to clogging or mineral deposition on rock joints [9].

2) Hydrogeological changes under land; lowering or fluctuations of ground water level with effect on hydraulic gradients (i.e. changing driving force for aggressives).

3) Hydro-chemical changes: Aggressive reservoirs may become depleted or activated (Alum Shale and others).

Dynamics in tunnels

Tunnel drought, due to tunnel fans and others: small spread leakages are most prone to evaporative effects and development of "excess" aggressiveness. Salinities may increase by 100 % – 1000 % from initial.

Tunnel air: exhaust fumes, dust and particles, bringing external components into contact with concrete surfaces (not investigated).

In general the deterioration rate of sprayed concrete = f(w/c, spray thickness, rock mass stability, hydraulic gradient, water chemistry, bacteria). The concrete standards and other specifications should preferably focus on this broad perspective.

# 5.3 What the standard does not tell: some recommendations

NS-EN 206-1 considers mainly static water and does not really account for mobile waters, typical of the tunnel environment and many other contexts. In the United Kingdom the classification for concrete in aggressive ground covers a wider range of applications than (NS)EN-206-1 [21]. Mobile and highly mobile waters are accounted for due to the experience that such waters generally are more aggressive than static waters. For example mildly acidic waters (pH = 5.5-6) may have very small structural effects when static but cause deteriorations when mobile. It would be a great advantage to incorporate this reality in EN-206-1.

The present investigation has unveiled practical problems: NS-EN 206-1 does not provide any example related to sprayed concrete and tunnel environments, despite that the EN- 14487, EN 14488 and EN 14489 standards for sprayed concrete refer to EN-206-1. In particular the problem is that the standard does not cover the entire durability problem.

In the first place there is no clear-cut definition of what seawater is (is the brackish Black Sea, a seawater or not?). It was not very clear what the differences between XS2 and XS3 represents when applied to tunnel concrete. The standard considers various contact relations between

"seawater" and concrete without taking notice to the different salinities encountered. Preferably variable salinity (chloride) should be included in EN-206-1, because degradation rates are always sensitive to concentration levels of all aggressive ions. Moreover there was some uncertainty as regards carbonation. The examples given by the standard seem "coloured" by one of the carbonation mechanism, namely the effect of atmospheric carbon dioxide. There is no mention of the role of bicarbonate, which is very important for the development of internal detrimental carbonation in the form of Popcorn calcite. Amendments should therefore make a clear distinction between the two different types and contexts of carbonation, by including bicarbonate (alkalinity) on the list of parameters to be analysed for. This would also provide information on the buffering capacity for acids, as well as capacity for TSA when sulfate also is present.

Also the effects of biological material should be included in EN-206-1. Recently RILEM summarised the results of microbial impact on concrete and other building materials, and stated that a multidisciplinary approach to concrete durability is necessary [22]. As a first approach the present author would suggest that EN-206-1 includes XSA (Especially aggressive) and splits into subclasses with bacterial attack and acid rock drainage (similar to some Alum Shale environments). Subsea tunnel sections should be integrated in the existing XSA Exposure Class and the potential effect of evaporation should be accounted for.

Finally the hydrogeological context is important to understand. Temporal changes in water pressure and water chemistry are governed by the interaction of water and rock mass. The durability development of buried concrete depends on the time integrated exposure conditions. Thus classification of hydrogeological environments should help unveiling the changing exposure conditions during the service life of concretes. There is hence a direct relationship between geological characterisation/classification and water exposure classification for tunnel concrete. For these reasons the concept of <u>"Life time exposure classification"</u> is introduced herein. The concept should be developed for various applications of concrete in different environments and geological settings based on hydro-geochemical and hydrological interpretation.

# 6 CONCLUSIONS

The NS-EN 206-1 Environmental Classes did not predict the extent and rate of the more severe attacks found in sprayed concrete used for rock support in tunnels. Tunnel concrete is also influenced by effects of hydraulic gradients, bacterial activity and evaporation. Ground water chemical loads can be very aggressive and may vary over time. Revision of the standard should involve biodegradation, bicarbonate contents and hydro-geological classification. There is also a need to differentiate better between the most aggressive exposure conditions involving "Life Time Exposure Classification" based on hydro-geochemical and hydrological interpretation.

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# **Properties of Concrete Exposed to Running Fresh Water for 24 years.**



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# ABSTRACT

A total of nine concretes, comprising three cement types, incorporation of fly ash, superplasticized high strength concrete and high performance concrete with microsilica, have been monitored during 24 years of exposure to running fresh water under Danish outdoor climatic conditions. The compressive strength development has been measured, and durability aspects have been assessed after 6 and 21 years of exposure, with very positive results.

**Key words:** Blended cement, fly ash, microsilica, high performance concrete, long-term exposure, compressive strength development, long-term compressive strength, cementing efficiency factor, durability.

# **1. INTRODUCTION**

The project described in the present paper was initiated by Aalborg Portland A/S in 1979. The aim of the project was to investigate the long term performance of a number of concrete compositions which were newly developed and consequently were untried at the time.

It was decided to produce a number of full scale precast units using the different types of concrete, and incorporate these units as "steps" in a fish-ladder for a local lake. In that way the new concretes would be easily accessible for periodic inspection over the following many years.

Inspection results up to the age of 14 years were reported in [1], and durability aspects were reported briefly at 21 years age in [2]. The present paper presents and incorporates results after 24 years, obtained in connection with the decommissioning of the fish-ladder.

# 2. EXPERIMENTAL

#### 2.1 Materials

The cements used were a normal portland cement (CEM I 42.5), a low- alkali sulphate resistant cement (CEM I 42.5-SR), and a blended portland-flyash cement (CEM II /B-V 42.5).

The CEM I 42.5-SR cement was the first Danish cement produced without clay as a raw material. Instead, ground quartz sand and fly ash were used to contribute silicon and aluminium

to the raw mix. It was produced as a sulphate resisting cement with a low  $C_3A$  content, and with a low alkali content, too.

Blended cement containing fly ash was new in Denmark as well. The cement for the present project was produced by separately grinding normal Portland cement to a Blaine-fineness of 388  $m^2/kg$  and fly ash to a Blaine-fineness of 515  $m^2/kg$ , and subsequently mixing 70 wt% ground cement with 30 wt% ground fly ash.

The chemical and physical data for the cements, fly ash and microsilica (condensed silica fume) are shown in Table 1.

		CEM I 42.5	CEM I 42.5	CEM II / B-V	Fly ash	Micro silica
			SR	42.5		
Used in concrete mixture No.		2, 5, 6, 7, 8, 9, 10	3	4	5	8, 9, 10
Chemical analysis						
SiO ₂	[%]	21.38	25.75	29.74	50.58	>90
Al ₂ O ₃	[%]	4.93	1.46	12.06	28.71	
Fe ₂ O ₃	[%]	2.97	1.72	3.7	4.69	
CaO	[%]	64.51	67.2	47.76	6.05	
MgO	[%]	1.24	0.56	1.36	1.35	
SO ₃	[%]	2.37	1.82	2,00	0.68	
Loss on ignition	[%]	0.9	1.19	3.18	7.83	
K ₂ O	[%]	0.63	0.04	0.59	0.73	
Na ₂ O	[%]	0.32	0.17	0.29	0.23	
Eq. Na ₂ O	[%]	0.74	0.2	0.68	0.71	
C ₃ A (calculated)	[%]	8.0	1.0	-		
Physical tests						
Fineness						
Passing 45 µm	[%]	83.5	96.8	96.1	82.9	100
Blaine	$[m^2/kg]$	363	299	436	367	20000
Density	[kg/m ³ ]	3150	3170	2890	2230	2200
Compressive strength (EN 19	6-1)					
1 day	[MPa]	11.7	8.8	8.9		
2 days	[MPa]	21.5	17.6	20.9		
7 days	[MPa]	40.5	30.3	35.5		
28 days	[MPa]	53.6	49.9	49.0		
56 days	[MPa]	59.1	61.7	60.8		
90 days	[MPa]	60.8	66.1	65.9		

*Table 1 – Chemical and physical data for cement, fly ash and microsilica.* 

* Consists of 70 wt% ground portland cement (Blaine: 388 m²/kg) and 30 wt% ground fly ash (Blaine: 515 m²/kg)

The chemical admixtures used were an air entraining admixture, a lignosulphonate based plasticizing admixture, and a naphthalene sulphonate based superplasticizing admixture.

The fine aggregate was from an inland deposit, and the coarse aggregate consisted of 8-12 mm and 12-16 mm sea dredged gravel.

#### 2.2 Mix design

The project comprised a total of 9 concrete mixtures. The mix designs are shown in Table 2.

No.		2	3	4	5	6	7	8	9	10
Designation		Reference	CEM I 42.5	CEM II/B-V	FA-	HS	SC		HPC	
		CEM I 42.5	SR	42.5	addition	no air entr.	air entr.	10 v.% MS	30 v.% MS	50 v.% MS
Mix design (nominal)										
Cement (C) Ely ash (EA)	[kg/m3]	330	330	310	290 125	400	330	530	415	294
Microsilica (MS)	[kg/m3]				125			42	125	209
Water (W)	[kg/m3]	160	160	150	160	115	102	120	113	105
Fine aggregate	[kg/m3]	710	800	722	664	772	772	698	705	714
Coarse aggr. 8-12 mm	[kg/m3]	535	533	541	498	579	579	524	530	536
Coarse aggr. 12-16mm	[kg/m3]	535	445	541	498	579	579	523	530	536
Admixture type*		AE	AE	AE	AE+P	SP	AE+SP	SP	SP	SP
W/C		0.48	0.48	0.48		0.29	0.31			
W/(C+0.4FA+2MS)		0.48	0.48	0.48	0.47	0.29	0.31	0.20	0.17	0.15
W/(C+FA+MS)					0.39			0.21	0.21	0.21
Paste vol. fraction incl. air		0.31	0.30	0.29	0.35	0.26	0.24	0.32	0.32	0.31
Fresh concrete properties										
Slump	[mm]	30	20	20	10	20	10	150	60	110
Vebe	[s]	2.6	7.0	4.5	6.3	9.8	15.0	0.0	5.3	-
Density	[kg/m3]	2277	2330	2302	2280	2450	2418	2472	2424	2445
Air content	[%]	4.9	3.9	3.8	4.0	2.0	3.6	1.5	2.2	1.9
Compressive strength (cast	test cylin	ders, water cu	red at 20°C	)						
1 day	[MPa]	12	14	10	11	30	31	51	50	36
7 days	[MPa]	30	35	30	28	58	55	75	85	82
28 days	[MPa]	38	44	40	45	72	66	95	115	114
90 days	[MPa]	42	50	52	56	82	78	115	135	133
1000 days (2.7 yrs.)	[MPa]	48	60	59	65	94	83	116	137	147
2280 days (6.2 yrs.)	[MPa]	52	65	60	71	102	94	125	143	158
Compressive strength (drilled cores)										
5000 days (13.7 yrs.)	[MPa]	46	62	52	51	76	80	100	118	117
8760 days (24.0 yrs)	[MPa]	61	78	61	75	104	107	131	131	158

Table 2 – Mix design, fresh concrete properties, and compressive strength

* AE: Air entraining admixture, P: Plasticizing admixture, SP: Superplasticizing admixture

Concrete No. 2 served as the reference concrete and was designed to serve in an aggressive environment according to the Danish code. It was prepared at a water/cement ratio of 0.48 and with an air content of 4.9 %, using a traditional Danish portland cement (Concrete No. 1 which was used for ladder unit No. 1 was unsuccessful and was therefore not included in the project).

Concrete No. 3 had a mix design essentially similar to that of the reference, but was prepared with the new low alkali, sulphate resisting cement.

Concrete No. 4 also had a mix design essentially similar to that of the reference but used blended cement (fly ash cement). The fineness of the cement was chosen to provide the same 28 day compressive strength as the reference.

Concrete No. 5 was prepared with separately added fly ash and was designed to yield a 28 day compressive strength equal to that of the reference, assuming a cementing efficiency factor of the fly ash equal to 0.3, i.e. about 40 kg/m³ of cement was replaced by 125 kg/m³ of fly ash.

Concrete No. 6 was a "high strength concrete" (HSC) without air entrainment, having the very low water/cement ratio 0.29. Using a high dosage of superplasticizing admixture led to an acceptable workability of the concrete despite the fact that the cement paste volume fraction of the concrete was significantly lower than that of the reference. At the time concrete of this type

had been utilised in truss concrete girder bridge structures in Japan and were expected to attain some interest in Denmark as well. The concrete had no air entrainment, in expectation of the low water/cement ratio securing its frost resistance.

Concrete No. 7 was similar to No. 6 except for the air content. This concrete was intended to have an air content of 4-6 %, allowing for a lower cement content, and securing frost resistance.

Concrete Nos. 8, 9, and 10 were all high performance concretes, containing microsilica, efficiently dispersed by a very high dosage of superplasticizing admixture. The concretes had an extremely low water/(cement + microsilica) ratio - 0.21 – and no air entrainment. Nos. 8, 9, and 10 had increasing content of microsilica, corresponding to 10, 30, and 50 % by volume of cement + microsilica. These mixtures were precursors of the later Densit[®] line of cement based high performance products.

#### 2.3 Precast concrete units

Nine precast concrete units were produced, each using one of the experimental concretes, and each eventually forming one step of the fish ladder. The individual unit was formed as an "inclined box", 2.9 m long, 0.9 m wide, and 1.15 m high, as shown in Figure 1. The units were produced at a precast concrete plant in the summer of 1979 and installed on site in September 1979. The appearance of the fish ladder in July 2001 can be seen in Figure 2.



Figure 1 – Sketch of the precast concrete unit, forming one step of the fish ladder



Figure 2 - Photograph of the fish ladder (July 2001) [2]

## 2.4 Exposure conditions

The concrete surface was differently exposed in the various parts of each unit. Part of the surface was permanently submerged in the running fresh water, part of it was exposed to the ambient climatic conditions, including freezing and thawing, while either in a predominantly dry state or located in a "splash zone".

# 2.5 Compressive strength

Test cylinders  $\emptyset$  100 mm x 200 mm for compressive strength measurement were cast in connection with production of the box units and were water cured at 20°C until testing up to the age of 6 years. At 14 and 24 years age cores ( $\emptyset$  95 mm x 140-150 mm) were drilled from the units and used for compressive strength determination.

# 2.6 Evaluation of durability

Ultrasonic velocity was measured at selected locations of the box units immediately after demoulding, then after transport and installation, and periodically up to the age of  $1\frac{1}{2}$  years. The results showed that no damages had been incurred during handling, transport and assembly of the units.

Durability was evaluated by microscopic analysis of thin sections prepared from cores taken from the bottom plate of the box units at 6 years age, and from cores taken from the overflow area at 21 years age.

#### 3. **RESULTS AND DISCUSSION**

#### **3.2** Compressive strength

The compressive strength of concrete Nos. 2-4 is shown in Figure 3.



Figure 3 – Compressive strength development of concrete Nos. 2-5

The data points up to 6 years age originate from testing based on the cast and water cured specimens, and it is seen that the compressive strength systematically increases over that period, as expected. The 14 and 24 year strengths have been measured on drilled cores. At 14 years age the compressive strength result is apparently lower than at 6 years, where after the strength increases from 14 years to 24 years and reaches a level at 24 years significantly above that at 6 years. This pattern suggests that it is the nature of the specimens that is responsible for this behaviour. It has been reported [3] that cast specimens can yield as much as 25-55 % higher compressive strength than cored specimens. On the other hand, part of the "discrepancy" might be explained by the maturity of the cored samples being significantly lower than that of the cast specimens which have been cured at a constant temperature of 20°C. However, since we do not have a reliable tool to convert core results to cast cylinder results, we are left with the observation that at least there seems to be a significant strength increase in the period from 6 years to 24 years.

The reference concrete exhibits a strength gain of 37 % from 28 days to 6 years of age, and another 17 % increase from 6 to 24 years. The sand/chalk cement leads to significantly higher concrete strengths than the ordinary portland cement, perhaps because it has a higher content of both  $C_3S$  and  $C_2S$ . The blended cement meets its goal of producing a 28 day concrete strength similar to that of the reference, and gives significant further strength gain up to 6 years. From 6 years to 24 years, however, there does not seem to be any noticeable strength increase. The fly ash addition in concrete No. 5 apparently has a higher cementing efficiency factor than expected, and leads to a large concrete strength gain during the entire period up to 24 years.



Figure 4 – Compressive strength development of concrete Nos. 6-7

The compressive strength development of the two high strength concretes is shown in Figure 4. Again, significant strength gain is noted beyond 28 days and during the entire period up to 24 years.

Figure 5 shows the compressive strength development of the three high performance concretes.



Figure 5 – Compressive strength development of concrete Nos. 8-10

The 28 day strengths range from 95 to 114 MPa and the compressive strengths further increase to 125-158 MPa at 6 years age. The 24 year strength with a microsilica content of 10 % by volume is distinctly higher than the 6 year strength, with 30 % microsilica the compressive strength apparently drops somewhat from 6 to 24 years, while it remains constant with 50 % microsilica. However, due to the non-systematic behaviour, and due to the fact that the cores probably underestimate the compressive strength it appears likely that at least there is no significant strength decrease at later ages in the dense concretes with microsilica, contrary to what has been sometimes suggested in the technical literature.

## 3.3 Durability

The results of thin section analysis by microscopy on samples taken at 6 years age [1] from the bottom plates and 21 years age [4] from the overflow area are summarised in Table 3.

A more or less continuous carbonate layer was observed outermost on all samples at 21 years age. It had a sharp border to the paste, indicating that the layer had been formed as a result of efflorescence. This was not seen on the 6 year samples which were taken from the water covered bottom plates.

The entrained air seemed to be well distributed, some minor depositions were found in the voids, and carbonation depths were found to be modest. Microcracks in the hardened paste were particularly found in the dense concretes containing microsilica, as expected. The observations after 21 years were generally not much different from those at 6 years, and no durability problems were observed.

	No.	Age (years)	Air entrainment*	Substance in voids*	Paste microcracks	Carbonation depth (mm)	Thickness of carbonate
REF		6	Non-hom	Some ettr	Some	04-10	-
KLI.	2	21	Fairly hom.	Some ettr.	A few	0.1 - 1.2	$\approx 0.6$
S/C-	2	6	Hom.	Ettr., CH, CĈ	Some	0.1 - 0.7	-
cement	3	21	Fairly hom.	Ettr., CH, CĈ	Some	0.1 - 0.2	Up to 2.0
FA-	4	6	Hom.	Little ettr. & CĈ	A few	0.1 - 0.7	-
cement	4	21	Hom.	Little ettr. & CĈ	A few	0.3 - 1.0	Up to 2.0
FA-	5	6	Hom.	Little ettr.	A few	0.1 - 0.7	-
addition	5	21	-	-	-	-	-
HSC,	6	6	None	CH, CĈ	Some	0.1 - 0.7	-
2.0% air	0	21	-	-	-	-	-
HSC,	7	6	Hom.	Little ettr.	Many	0.4 - 1.0	-
3.6% air	/	21	Hom.	Little ettr. & CH	Some	-	Up to 2.5
HPC,	0	6	None	None	Many	0.1 - 0.4	-
10% MS	0	21	None	None	Some	Up to 0.3	Up to 2.5
HPC,	0	6	None	None	Many	0.1 - 0.3	-
30% MS	9	21	None	Little CĈ	Many	Up to 0.2	Up to 2.0
HPC,	10	6	None	Little CĈ	Many	Up to 0.1	-
50% MS	10	21	None	None	Many	0	Up to 0.5

Table 3 – Results of thin section analysis.

* Hom.: homogeneous, Ettr.: Ettringite, CH: Calcium hydroxide, CĈ: Calcium Carbonate

In connection with dismantling the fish-ladder cores were drilled from the concrete units, cut in half and epoxy impregnated. Visual and microscopic inspection of these samples revealed no sign whatsoever of deterioration of any of the cores.

# 4. CONCLUSIONS

A total of nine concretes, comprising special cements, incorporation of fly ash, superplasticized high strength concrete and high performance concrete with microsilica, have been investigated during 24 years of exposure to running fresh water under Danish outdoor climatic conditions. The results lead to the following conclusions:

- None of the concretes investigated show any sign of durability problems after 24 years of exposure to running fresh water
- A large number of microcracks were observed in the hardened paste of dense non-airentrained concrete with microsilica. However, the microcracks had no adverse effect on frost resistance after 24 years of exposure in a wet/humid environment
- All concretes showed a 30-50% strength increase from 28 days to 6 years, and a significant strength increase from 14 to 24 years.
- No sign of late strength loss of dense microsilica containing concrete was observed, despite the presence of microcracks

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# Durability of field concretes made of portland and silica fume cements under sea water exposure for 25 years









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#### ABSTRACT

The durability of structures made of blended cements lacks of field data (especially sulfate attack), which makes it difficult to fit these new concretes with existing test methods and performance criteria. Most results on sulfate resistance are derived macroproperties (expansion) to the detriment of micro-characteristics (phase assemblage).

This paper concerns the microstructural study of field samples exposed to the North Sea for 25 years. Complementary techniques such as micro-XRF, PIXE, SEM-EDS and XRD are used to evaluate the microstructural performance and stability of the phase assemblage of a silica fume blend and an SRPC along the sulfate (and chloride) ingress profile, compared to pure portland concrete.

**Key words:** sulfate attack, sea water, silica fume, SEM, phase assemblage, ettringite, brucite, Friedel's salts.

#### 1. INTRODUCTION

The understanding of concrete degradation mechanisms is the key to optimize concrete and its components for durability and reliability, thus to maximize the durability of structures. This study will focus on sulfate attack which can be important in subterranean environments and to a certain degree under fresh and sea water conditions. Sulfate attack manifests in the field through decreased performances of the exposed structure, which is characterised by a loss in strength due to cracking, decohesion, softening of the cementitious matrix [1]. Most results dealing with sulfate resistance of cement are derived from accelerated laboratory tests on mortars bars. Data from sulfate induced damages of structures under field conditions are fewer and highly fragmented. At the moment there is no real link between data from accelerated laboratory experiments and the performance of field structures under sulfate attack, making life time predictions of concrete constructions difficult.

#### 1.2 From chemical to thermodynamic problem

Sulfate attack is driven by reactions between sulfate ions and cement hydrates (Fig. 1) [1]. Since ions are concerned, presence of moisture is necessary. The reaction products are crystalline and mainly of three types:

- Ettringite (Ca₆(Al,Fe)₂(SO₄)₃(OH)₁₂.26H₂O; hexagonal)
- Gypsum (CaSO₄.2H₂O; monoclinic)
- Thaumasite (3CaO.SiO₂.SO₄.CO₃.15H₂O; hexagonal)



*Figure 1 – Scheme of the reactions between sulfate ions and cement hydrates.* 

Ettringite formation makes the object of most papers because of its potential expansive behaviour. This compound forms from aluminate-bearing sources in the cement paste for instance,  $C_3A$  and its hydration products (AFm, monosulfoaluminate) or even aluminate-bearing glass in fly ash. Gypsum can form from CH; while thaumasite needs more specific conditions to form from ettringite and C-S-H: low temperature and presence of carbonate. Carbon carbonate can be introduced into cement from two main sources: carbon dioxide dissolution from the environment and limestone fillers (CEM II/A-LL and CEM II/B-LL). Carbonate presence in cement can lead among others to the formation from monosulfoaluminate of monocarboaluminate (3CaO.(Al,Fe)₂O₃.CaCO₃.11-12H₂O).
The relative amounts of  $CaCO_3$ ,  $C_3A$ , and  $CaSO_4$  control the equilibriums of the phases (e.g., calcite, monocarboaluminate, monosulfoaluminate, ettringite, thaumasite and gypsum) regarding the cement hydrates and the pore solution (Fig. 2). At this stage, it is worth to note that sulfate attack can be approached by rigorous thermodynamic.



Figure 2 – Ternary diagram C₃A-CaSO₄-CaCO₃.

Thermodynamic modelling describes the problem according to the Gibb's energy minimisation (i.e., phase stability according to the phase rule). Such models only concern CEM I, CEM II/A-LL and CEM II/B-LL types cements [2-3]. Also numerical models have been developed [4], which can be extended to other cementitious systems (CEM II/A-D, CEM V).

The main problem remains the choice of the more realistic system; field data are therefore a considerable input for such theoretical approaches.

# **1.1** From prescriptive towards performance approach

### Prescriptive approach

The traditional approach to prevent sulfate attack from occurring aims at establishing prescriptions on cement composition. This approach was developed in the early 30's with the limitation of alumina availability to avoid ettringite formation: together with a decrease of the water to cement ratio, the  $C_3A$  content of cement was reduced and sulfate resisting Portland cements (SRPCs) were introduced on the market with high  $C_4AF$  content to balance  $C_3A$ .

More recently, the increased use of mineral admixtures has lead to the introduction of new norms on cement composition to fulfil durability criteria: 5 types of cement were introduced, with 27 different possible compositions e.g.:

• CEM II / A&B-V&W types should concern high alumina fly ash although it is not established whether an increased Al₂O₃ content would stabilize monosulfoaluminate with respect to ettringite or not if sulfate is supplied [5].

• CEM III types should have a slag replacement higher than 65wt.-% to reduce sulfate attack since there are a lot of disagreements on the effects lower replacement levels and their consequences in the case of sulfate attack [6].

Nevertheless, owing to different habits and raw materials availability, most of the countries came to national prescriptions concerning the type of cement to be used and the w/c-ratio according to the type of exposure.

Furthermore, recent trends tend to integrate physical effect considerations in addition to chemical degradation and since expansion can occur even with low  $C_3A$  cements [7], sulfate attack is indeed more complicated than purely related to ettringite formation. Compared to field results, such prescriptive approaches are therefore not flexible enough to allow preventing sulfate degradation.

### Performance approach

Efficient resistance to sulfate attack implies the setting of fast, reproducible and reliable laboratory tests. So far, no harmonized CEN standard could be established: the draft for an EN test method failed because of a too large dispersion of results between test laboratories, mainly because of non-representative testing conditions [8]:

- too high sulfate concentrations compared to real conditions
- uncontrolled high pH, even if solution regularly changed according to ASTM C1012
- ITZ influence not taken into account
- non-adapted and non-comparable curing conditions

Furthermore, the microstructural changes and degradation induced by field sulfate attack need to be accurately investigated for evaluation of accelerated lab-tests. This has been done in the case of pure portland materials but the effects of SCMs are poorly documented so far.

The aim of the present work is to start to fill the lack of knowledge on this topic with generating qualitative and quantitative microstructural data of concrete from real structures and field exposures involving sulfate attack under different environmental regimes, ranging from Southern to Northern Europe (Spain, Germany, United Kingdom, Denmark, Norway). This implied the careful collection of selected specimens for which the nature of the cement used, mix design, cure and field exposure history are known. This paper focuses on the results from Hirtshals harbour exposure site (North Sea, DK) and concerns the comparison of sulfate resistance of a CEM I, CEM II/A-D and an SRPC.

# 2. MATERIALS & METHODS

### 2.1 Concretes

Aalborg Portland S/A disposes of a field trial site in Hirtshals harbour (DK), where large concrete panels  $(2 \times 1 \times 0.2 \text{ m}^3)$  are semi-immersed into the sea (Fig. 3). The present project takes part into the frame of the European Consortium NanoCem and as partner of this, Aalborg Portland A/S provided the concrete specimens with their subsequent up to 28 days data. The specimens were delivered under the form of cylinders of 100mm diameter and length, which have been wet cored in the submerged part of the concrete panels. Their binder content ranges from 340 to 390kg.m⁻³ and their maximal aggregate size is of 16mm. The CEM I, CEM II/A-D and CEM I SR mixes have respectively a mass water-to-binder ratio (W/B) of 0.43, 0.51 (0.39, considering the *k*-factor of efficacy for silica fume) and 0.42. The concretes studied are made of a CEM I, a CEM II/A-D made on the basis of the CEM I and an SRPC (Tables 1 & 2) with

2wt.-% C₃A. The specimens were cast on site and semi-immersed immediately after demoulding.



*Figure 3 – Photographs of the field trial exposure site in Hirtshals harbour (DK).* 

Table 1 – Clinker Bogue composition of the cements.

	Bogue composition of the clinker (wt%)						
	$C_3S$	$C_2S$	C ₃ A	C ₄ AF			
CEM I	55.0	20.0	10.0	9.0			
CEM I SR	SR 60.0		2.0	10.0			

*Table 2 – Mix-design and micro-XRF oxides composition of the binders.* 

	Μ	Oxides composition of the binder (wt%)								
	Mineral admixture	Substitution level (wt%)	SiO ₂	Al ₂ O ₃	Fe ₂ O ₃	CaO	Eq. Na ₂ O	LOI	Total	SO ₃ in the bead after fusion
CEM I	-	-	21.0	5.2	3.0	63.0	0.6	1.5	95.3	2.9
CEM II/A-D	Silica fume	10	28.1	4.9	2.7	56.7	0.8	1.7	95.9	2.7
CEM I SR	-	÷	24.0	2.5	3.1	66.0	0.3	0.9	97.5	1.8

Besides the provided up to 28 days properties, densities and porosities of the received specimens have been measured with a picnometer and by mercury intrusion porosimetry (MIP). The three concretes studied present comparable macroscopic properties at early age, 28 days and 25 years (Table 3).

	Fresh properties		28 days property	25 years properties					
	Slump (mm)	Air (%)	Strength (MPa)	Absolute density (kg.m ⁻³ )	Bulk density j (kg.m ⁻³ )	Total porosity (%)	Total porosity _{MIP} (%)		
CEM I (42.5)	$50\pm20$	5.8	54.00	$2495\pm1$	2292 ± 2	8.1	6.1 ± 0.1		
CEM II/A-D	$70\pm20$	5.8	54.00	$2543\pm1$	$2364 \pm 23$	3 7.0	$5.4\pm0.1$		
CEM I SR	$60 \pm 20$	6.0	50.00	-	2480	-	-		

Table 3 – Macroscopic properties of the studied concretes at early age, 28 days and 25 years.

### 2.2 Exposure conditions

The concrete panels are semi-immersed in the North Sea of Hirtshals harbour (DK). No data were available concerning the exact composition of the water in Hirtshals harbour; nevertheless, it is assumed that the average ocean composition (Table 4) can be taken as reference for the exposure conditions to which the concretes were subjected to. The main species are sulfate and chloride, which can penetrated inside and interact with the cementitious matrix of concrete. It is worth to note that the sulfate  $(SO_4^{2^-})$  content of the ocean corresponds to an equivalent concentration of 3-4g.l⁻¹ in sodium sulfate  $(Na_2SO_4)$ , which is 10 times lower than the usual concentration applied in quick lab tests for sulfate resistance.

Table 4 – Mean ocean water characteristics.

		Mea	in water	compos	ition (w	t%)		Water	Water
	SO42-	Na ⁺	$Mg^{2+}$	$Ca^{2+}$	Cl	$\mathbf{K}^+$	HCO ₃ -	(°C)	pН
Ocean	2.65 ± 0.5	10.56 ± 0.15	1.27 ± 0.15	0.40 ± 0.15	18.98 ± 0.15	0.38 ± 0.15	0.15 ± 0.15	6-17	8

### 2.3 Investigation strategy

### Physical changes statement

The first damage state evaluations were done on a macroscopic scale through visual observations, ultra-sound measurements and optical microscopy.

Damages caused by sulfate attack lead in most of the cases to a loss in strength of the exposed structure. Macroscopically, a reduction in the material density can be observed. Impulse velocity measurement is based upon the principle that ultra sound waves propagate with a reduced velocity in materials of reduced density. An emitter and a receptor transducer are placed on each surface of the core, facing each others on the core length direction. The concerned transducers are for longitudinal waves, propagating in a direction parallel to the drill core axis and of a frequency of 50 kHz. Measurements have been carried out on a USPC 3041 portable device with VS-150-M sensors from Vallen Systeme, after the European standard prEN 13296.

The cores were then cut and transversal thin sections  $(5 \times 5 \text{ cm}^2)$  prepared. The 25µm thickness of the thin sections allows them to be observed under the optical microscope in polarizing light.

Crystals other than those of the cubic system have the property to separate a beam of polarized light into two rays travelling at different speeds and right angle to one another. The magnitude of this double refraction (or birefringence) is defined as the difference of these two refractive indices and results in specific interference colours for a given crystal. These interference colours can be of different orders according to their intensity; all these interference colours and orders have been referenced into the Michel Lévy colour chart. Therefore sulfate-bearing phases such as monosulfoaluminate, ettringite, thaumasite, gypsum, mirabilite, thenardite can be identified according to their interference colour [9]. The petrographic study has been carried out on an Axiskop 40 Pol from Zeiss.

### Ion ingress profiling

One major interest of the field samples study is the real ion ingress profiles assessment. Elemental mappings can be drawn on a cm-scale by micro-X-ray fluorescence (micro-XRF) spectrometry. The micro-XRF spectrometer used in this study is an Eagle III  $\mu$ -Probe from Röntgenanalytik. X-rays are emitted from a rhodium anode and focussed through a series of capillary lens with a bias voltage of 40kV on the sample surface. The applied spot size is of 150 $\mu$ m and the resolution of 512×400 pixels². X-rays excite sample atoms whose specific relaxation energy is captured by a Si(Li) semi-conductor detector.

As comparison technique proton-induced X-ray emission (PIXE) has been carried out. Principle of PIXE is the same than micro-XRF, where accelerated protons ¹H are used as excitation ray. Measurements were conducted at the University of Surrey on the External Beamline. Specific X-rays are collected by a lithium drifted silicon detector (Si(Li) semi-conductor) with a scan area diameter of 3mm (215pixels). The output data are in form of a mapping from the scanned surface. The data treatment is done by image surface analysis and therefore expressed in pixel².

Additionally, the ionic profiles were measured by analytical chemistry. Quantitative sulfate profiles have been determined using coulometric titration analysis, while chloride profiles were obtained from conductivity measurement. 5mm slices of the cores were cut in the depth from the contact surface and ground to powder, from which 200mg and 2g were analysed to extract sulfate and chloride content in each slice. A behr C30 HT/S 30 HT/CS HT from behr Labor-Technik was used for sulfate quantification: sulfate was evaporated from the powdered sample by heating at 1350°C; the gaseous sulfate SO₂ enters into a pH-controlled solution of sodium sulfate and hydrogen peroxide (Na₂SO₄, H₂O₂)) and its subsequent pH modification due to sulphuric acid H₂SO₄ formation is balanced by OH⁻ release through electric potential measurement. Chloride content is deduced from conductivity measurements in AgNO₃ + NaCl solution using a silver electrode to precipitate chloride in AgCl form.

#### Phases assemblage zonation

In a first step, representative locations along the sulfate penetration profiles (e.g.; contact surface, highest peak of the profile, decreasing slope and background plateau) were chosen for phases analysis by scanning electron microscopy (SEM) on polished sections. For the phases identification into a cementitious matrix, SEM can be used in back-scattered electron (BSE) imaging modus or in energy-dispersive spectroscopy (EDS) modus. In BSE-modus on polished sections phases can be distinguished through their atomic number responsible of their grey level (the heaviest phase, the highest atomic number, the highest -brightest- grey level). 2D-morphologies can qualitatively discriminate phases. EDS-modus can assess a semi-quantification of the phases in equilibrium. EDS-modus is based on the same principle than micro-XRF or PIXE but accelerated electrons are used as excitation source for the specific X-rays emission collected by a Si(Li) detector. Hundreds of EDS spot analysis of the cementitious

matrix were carried out from which atomic ratios between selected elements allows identifying phases in equilibrium, knowing reference phases stoichiometry. A big advantage of this technique is the assessment of phases finely intermixed into the C-S-H matrix, which cannot be identified in BSE-modus. The SEM used is a Quanta 200 from FEI microscope with a tungsten cathode for electron emission at 15 kV and monitored by Spirit analysis software.

Those results were complimented with X-ray diffraction analysis. Crystalline phases possess the ability to diffract emitted X-rays according to specific angles owing to Bragg's law. If C-S-H is XRD-amorphous, portlandite and especially sulfate attack products gypsum, ettringite and thaumasite are highly crystalline: they can be identified by XRD. Using an electrical steel plane from BOSCH 1mm slices were planed along the core depth and sieved at 100 $\mu$ m. XRD patterns of the powders were acquired during 20 min with a step size of 0.017° from 7 to 70 2 $\theta$  and from 6 to 20.5 2 $\theta$  in order to increase the amplitude resolution at low angles, where cementitious hydrated phases and especially gypsum, ettringite and thaumasite presents specific peaks. The device used is a by a Philips X'Pert Pro diffraction system with a CuK $\alpha$  anti-cathode, in Bragg-Bentano position and assisted by the software X'Pert HighScore Plus.

As cross-check, some of the powders studied in XRD were investigated under solid state aluminium nuclear magnetic resonance (^{Al}NMR) as well using a Bruker Spectrometer from the ESPCI in Paris.

### 3. **RESULTS & DISCUSSION**

### **3.1** Physical changes

#### Visual observations

After 25 years immersed in the North Sea, the CEM I concrete is cracked along the contact surface while the CEM II/A-D and CEM I SR do not have any significant damage (Fig. 4).

#### *Ultra-sound measurements*

The ultra sound impulse velocity inside a material can be related to its dynamic E-modulus (Eq. 1.), knowing the bulk density of the concerned material [10].

(1)

$$E_{dyn} = \frac{l_s^2}{\left(t_s - t_k\right)^2} \cdot \rho_s \cdot k$$

 $E_{dyn}$ : dynamic elastic modulus (N.mm⁻² # MPa)

*l_s:* sample length (mm)

 $t_s$ : ultra sound impulse delay time in the sample ( $\mu s$ )

 $t_k$ : correction factor: intrinsic delay time of the transducers (3.7 $\mu$ s)

 $\rho_s$ : sample bulk density (kg.m⁻³)

 $c_l$ : correction factor considering the hindrance of lateral strain (= 0.9)

The correlation between the dynamic E-modulus and the bulk density underlines the reduced physical performance of the CEM I concrete versus the CEM II/A-D and the CEM I SR, with especially a dynamic E-modulus in the range of 10 times lower (Fig. 4).



Figure 4 – Macroscopic state: visual observations (left), dynamic elastic moduli (right).

### Optical microscopy

The main application of optical microscopy in this study consists in the identification of the carbonation depth. In the presence of  $CO_2$  and sufficient moisture, portlandite (Ca(OH)₂) can turn into calcite (CaCO₃). Calcite is highly crystalline compared to the cementitious matrix: its birefringence magnitude is of -0.172, which confers it a yellow-brown interference colour of the  $6^{\text{th}}$  order, easily identifiable on the black-greyish C-S-H background. This method precised at  $\pm$ 0.1mm is preferred to the standard method with the coloured indicator phenolphthalein because of the tiny range of the carbonation zones. The CEM I concrete presents a carbonation front of 2.1mm while the two other concretes made of CEM II/A-D and CEM I SR are only carbonated up to respectively 0.7 and 1.2mm depth. Two main parameters can influence the carbonation of concrete: the quantity of the portlandite available to turn into calcite and the connected porosity of the microstructure to allow carbonate to penetrate in the depth of the sample. Considering the low dilution effect due to the low silica fume replacement (10wt.-%) and the expected increased amount of portlandite in a sulfate resistant cement (higher  $C_3S$  and  $C_2S$  contents compared to a CEM I), the difference of factor 2 in the carbonation depths between the CEM I concrete and the two other concretes is unlikely due to chemical characteristics of the mixes. The filler effect of silica fume is especially high due to its high specific surface ( $\sim 20\ 000\ \text{m}^2\text{.kg}^{-1}$ ) which enhances C-S-H nucleation; additionally, the pozzolanic activity of silica fume leads to an increased C-S-H formation. The higher  $C_3S$  and  $C_2S$  contents of the sulfate resistant cement facing an ordinary portland cement ensure a larger amount of C-S-H formed, responsible for the dense binder property of the mix. The increased performance against carbonation of the blend and of the sulfate resistant cements over the CEM I is therefore attributed to their denser microstructures, reducing the pore network connectivity. It is worth to note that  $CO_3^{2-}$  ions are relatively big  $(CO_3^{2^2})$  ion size parameter of 5Å): the pore network of the CEM II/A-D and the CEM I SR must be correspondingly fine or tortuous enough to limit  $CO_3^{2-}$  intrusion.

# 3.2 Ion ingress profiling

### Micro-XRF

The 40×60mm² cross sections scanned with micro-XRF reveal a deeper penetration of chlorine than sulfate for each of the mixes (Fig. 5). This can be due to the smaller size of Cl⁻ compared to  $SO_4^{2^-}$  (ion size parameter of 3Å for Cl⁻ compared to 4Å for  $SO_4^{2^-}$ ), which favours Cl⁻ transport over  $SO_4^{2^-}$ . Additionally to this physical effect, chemical aspects must play a role, notably the higher chlorine electronegativity (3.16) compared to sulfur (2.58), and also the higher activity coefficient of chloride over sulfate, which difference increases with increasing ionic strength.

Those chemical aspects can be at the origin of an enhanced chloride binding of the C-S-H over sulfate binding. The ions ingresses are reduced in the CEM II/A-D and CEM I SR concretes compared to the CEM I, but this reduction is not obviously of the same range. Considering the CEM I SR, the sulfate and chloride penetrations are reduced of a factor 4 regarding the CEM I (Fig. 5). This can be attributed to the denser microstructure of the cementitious matrix of the sulfate resistant cement over the ordinary portland cement, which, as explained in the case of the reduced carbonation (above section), reduces the pore network connectivity and thus limits transport-based phenomena. On a chemical point of view, AFm phases have a higher Cl-binding capacity than C-S-H [11]. In consequence, the deficit of the low alumina containing sulfate resistant cement in AFm phases would promote chloride penetration through the relative inert C-S-H, which is the opposite than the observations in micro-XRF if compared with the portland cement. In the case of the CEM II/A-D the 10wt.-% of silica fume replacement seems to significantly limit sulfate penetration while its effect on chloride penetration is much negligible compared to the CEM I (Fig. 5). Silica fume addition can lead to a decrease in the pore network connectivity as explained in the case of carbonation (above section). Nevertheless, since chlorides penetrate in the CEM II/A-D cementitious matrix up to a comparable depth as for the CEM I, while sulfate penetration is reduced of a factor 10, the pore network of the CEM II/A-D mix must be connected enough to allow ion transport but fine enough to avoid ions bigger than sulfate from penetrating. The difference in alumina content between the two mixes would let think to a deeper penetration of sulfate for the CEM II/A-D due to its subsequent lower AFm availability, which would fix sulfate for ettringite formation. However, silica fume has a pozzolanic activity that could in principle cause a pH drop of the pore solution due to portlandite consumption. Ettringite is highly unstable at low pH, especially at pH below 11.



Figure 5 – Micro-XRF qualitative sulfur and chlorine ingress profiles and penetration depths.

#### PIXE

The concrete cut cross sections have also been investigated under PIXE. Regarding the large spot size of this method (3mm), aggregates cannot be excluded from the analysis zone. Ions from the surrounding environment penetrate inside concrete through the capillary pore network of the cementitious matrix. The total sulfur and chlorine data (surface in pixel²) collected from the analysed surface must be calibrated over data from a specific element of the cementitious matrix. Calcium is present only in the cement paste since aggregates are mainly of quartz. Therefore, the calibration is done calculating the pixel² ratios S/Ca and Cl/Ca. Comparing with the counts S/Ca and Cl/Ca ratios from micro-XRF results (Fig. 6), the profile shape and the penetration depths are confirming the reliability of the micro-XRF technique for ion penetration analysis.



Figure 6 – PIXE and micro-XRF S/Ca and Cl/Ca profiles in the CEM I cementitious.

#### Analytical chemistry

Analytical chemistry is used to quantify the amount of  $SO_3$  in the concrete at different depths. According to the aggregate/binder ratio, the amount of  $SO_3$  in the binder can be approximated. If the shape of the profile of  $SO_3$  content in the binder calculated from analytical chemistry is close to the shape of the S profile from micro-XRF (Fig. 7), its resolution in depth is too poor compared to the depth range of the ion ingress phenomena. On top of this the quantification from analytical chemistry results is too subjected to the aggregate/binder ratio, which varies highly considering the few quantity of material analysed: the high standard deviation of the results shows the poor reliability of this method for this application.



Figure 7 – Titration SO₃ and micro-XRF S profiles in the CEM I cementitious matrix.

#### 3.3 Phases assemblage zonation

#### SEM: BSE and EDS modus

Phase assemblage is part of microstructure concepts. The first approach of microstructure consists in SEM observations in BSE modus. A first idea of the phase modifications due to ions ingresses from the surrounding environment can be done through BSE observations of the microstructure in surface compared to the bulk (Fig. 8). The CEM I presents cracks parallel to the surface filled up with a crystalline product, which has been identified by EDS analysis to be brucite (Mg(OH)₂). Air voids close to surface present fibrous crystals in radial growth, especially in the ordinary portland and sulfate resistant cements: EDS analysis reveals elemental distribution typical for ettringite. Additionally, all the three samples present local precipitations of ettringite in large pores (>  $10\mu m$ ). The ordinary portland and sulfate resistant cement pastes reveal ettringite precipitations in air voids and pores also in the bulk of the sample, even after the sulfate penetration depth. The CEM I SR concrete did not reveal any cracking. This confirms the conclusion from the crystal growth theory (see Laplace's equation), according to which only crystal growth inside a pore of radius smaller than 50nm can cause sufficient stress to exceed the tensile strength of a concrete (few MPa) and thus be at the origin of cracking phenomena. Another interesting observation is the absence of cracks around aggregates, which are in the literature identified as characteristic of sulfate attack on mortars exposed to sulfate in laboratory conditions; i.e., generally Na₂SO₄ solution at 50g.L⁻¹, as for instance in the ASTM C1012 prescription. Even if the equivalent Na2SO4 concentration of the North Sea water is 10 times lower than the ASTM prescription (3-4g.L⁻¹), this non cracking effect is especially surprising considering the advanced ages of the concrete samples (25 years) and their strain free storage conditions. Some tiny spaces are despite observable around few aggregates, where in the CEM II/A-D portlandite precipitation is recognised by EDS.



Figure 8 - SEM micrographs in BSE modus at the surface (top) and in the bulk (bottom) of the three concretes.

Systematic EDS point analyses have been carried out in the outer C-S-H of the samples cementitious matrix at characteristic depths along the ions ( $SO_4^{2-}$  and  $Cl^-$ ) ingress profiles in order to identify finer intermix of phases. Plotting the atomic ratios S/Ca versus Al/Ca for each

of the three concretes allows to split typical sulfate attack involved phases such as gypsum, ettringite, thaumasite, monosulfo- and monocarboaluminate (Fig. 9).

The CEM I shows a strong tendency of the C-S-H points cloud towards ettringite at 3mm depth; i.e., at the highest peak of the sulfate ingress profile as determined by micro-XRF. This enrichment in ettringite concerns this specific part of the sulfate profile since only disparate points in the direction of ettringite are to be seen at 10 and 40mm depth, which ones are in fact from local ettringite precipitation in pores. However, the first 0.5mm is depleted in ettringite as the sulfate profile at this depth shows a strong decrease. 0.5mm depth is inside the carbonation zone, which is characterised by a denser microstructure (calcite is denser than portlandite) and a lower pH around 9 compared to the 12.6-13 value of a concrete bulk. The densification of the microstructure during carbonation could have physically pressed the sulfate further in the bulk of the sample. However, the low pH of the close to surface zone due to carbonation can be enhanced by the low pH of the surrounding sea water. It is known that ettringite is highly unstable at low pH and especially at pH lower than 11, which could explain its depletion in this surface zone. If ettringite is unstable in the surface, and if no other phase can bind sulfate, ions must penetrate deeper in the sample to be stabilised. A third hypothesis could be a leaching of sulfates, which had accumulated at the surface of the sample, towards the surrounding environment.

The CEM II/A-D and the CEM I SR present less dispersed EDS points between C-S-H and ettringite: they are much arranged in a line joining the two phases. This can be an indication that ettringite is not finely mixed to the outer C-S-H but rather crystallised in local areas such as tiny capillary pores. Nevertheless, ettringite depletion at 0.5mm depth is observable in the case of the CEM II/A-D as well, while this phenomenon seems to not occur in the CEM I SR. This is still unclear why the sulfate resistant cement behaves differently and further investigations must be done.

In all the three cementitious matrices, EDS points are situated between the C-S-H and ettringite at 0.5, 3 and 10mm depths but in the bulk of the samples, after the sulfate penetration depth (at 40mm), the points split between C-S-H cloud on one side and theoretical ettringite composition on the other side. This puts in evidence that ettringite firstly precipitates in free spaces and then intermixes with the C-S-H.

Additionally, for all of the three samples, the EDS points, if not in the C-S-H cloud, are divided between ettringite and monocarboaluminate with the tendency: the most ettringite, the less monocarboaluminate and vice-versa. This trend towards monocarboaluminate was not expected considering the chemistry of the mixes (not particularly high LOI and not particularly low  $SO_3$  content). Indeed, monosulfoaluminate would be expected from the theory at depth, where sulfate has not ingresses enough to turn it into ettringite. It appears the more probably sulfate supply causes monocarboaluminate to turn into ettringite in the three concretes observed. It might be possible that the initial monosulfoaluminate of the cement paste has turned into monocarboaluminate with time. Nevertheless the questions of the source of carbonate and the location of the sulfate delivered by this transformation remain.

The absence of gypsum is notable since this phase is often referred to in the literature as typical sulfate attack product. The presumed low pH conditions at the surface of the concretes should even enhance its stability. This absence can be due to the low sulfate concentration compared to the one generally used in laboratory tests, which is unable to stabilise gypsum.

The plots of the atomic ratios Mg/Ca versus Al/Ca and Cl/CA versus Al/Ca permits to identify respectively brucite and Friedel's salts formation (Fig. 10). Magnesium present in sea water can penetrate inside the cementitious matrix of the exposed concretes and react with portlandite to form brucite. Due to the big size of  $Mg^{2+}$  (ion size parameter of 8Å) and its low electronegativity (1.31), magnesium does not penetrates deep inside concrete and reacts therefore on the very surface with the available portlandite to form brucite. Indeed, brucite is detected by EDS for each of the three concretes only up to 0.5mm depth and even only in the first 0.1mm for the CEM I SR. On the other hand, chloride has the ability to penetrate deeper than sulfate into concrete. Therefore Friedel's salts are only observable deeper than ettringite formation. Nevertheless, sulfate and chloride must be in competition to react with AFm phases such as monosulfo- and monocarboaluminates to respectively form ettringite and Friedel's salts.



Figure 9 – SEM EDS S/Ca vs. Al/Ca atomic ratios plots of the outer C-S-H for the three concretes at 0.5, 3, 10 and 40mm depth (from the top to the bottom).



Figure 10 - SEM EDS Mg/Ca and Cl/Ca vs. Al/Ca atomic ratios plots of the outer C-S-H for the three concretes at characteristic depths.

### XRD

The XRD analysis has been up to now only carried out on the CEM I and the CEM II/A-D concretes. Diffractograms of samples from indoor exposure site (same mix design but not exposed to sea water) were also acquired as references. In principle, XRD results (Fig. 11) confirmed and affined SEM observations and analyses.

The XRD diffractogram of the CEM I concrete underlines the depletion of ettringite in the first 2mm, followed by an enrichment at 3mm with a slight decrease up to the bulk. The apparition of Friedel's salts after the ettringite peak formation (3mm) is very visible and the surface precipitation of brucite is estimated at the first 2mm. Leaching effect is strongly observable on portlandite, which peak starts to appear at 2mm depth.

The CEM II/A-D XRD diffractogram confirms the absence of ettringite in the first mm, and its continuous presence at further depth, probably as precipitate in pores. Friedel's salts appear at 2mm depth, but were detected by SEM EDS analysis more at the surface of the concrete. Portlandite is hardly seen, probably due to its consumption for the pozzolanic reaction of silica

fume. It was also difficult to identify it on SEM BSE micrographs. Brucite is not detected in the slices scanned by XRD.

In both samples no monosulfoaluminate is to be observed, while a peak for hemicarboaluminate is clearly identified. Therefore, the presumed monocarboaluminate from SEM EDS analyses must in fact be hemicarboaluminate. However, a "double peak" of ettringite is in both cases observable at lower angle. This peak could fit with muscovite (phase from aggregates) but its intensity variation with depth is rather suspicious for an aggregate phase. Such a peak has already been observed by S. Lamberet [12], who attributed it to an ettringite phase but was not able to explain it. If this peak is from a form of ettringite, its shifts towards lower angles than the pure ettringite phase indicates a widening of the d-spacing, which can be due to  $Al^{3+}$  substitutions by a bigger cation (Fe³⁺ for instance) or incorporation of elements (e.g., titanium, chromium, chlorine). Nevertheless, no any of these elements have been clearly identified yet by SEM-EDS analysis.

Nevertheless, no quantification can be carried out on these diffractograms, because of the presence of aggregates, which amount in each depth is not constant and cannot be evaluated.



Figure 11 – XRD patterns of the CEM I (left) and CEM II/A-D (right) concretes at low angles.

# ^{Al}NMR

As cross-check technique, ^{Al}NMR was performed on the CEM I and CEM II/A-D samples (Fig. 12). Two peaks have been identified for aluminium of coordination VI in the case of the CEM I concrete: one near +13ppm characteristic of ettringite and one near +10ppm characteristic of AFm phase. Unfortunately, different AFm phases have almost equal chemical shift and relative strong quadrupolar interactions in comparison with ettringite thus the distinction between the monosulfoaluminate hemicarboaluminate and monocarboaluminate is not possible. Nevertheless, XRD results let think that the detected AFm consists in hemicarboaluminate. Near to the contact surface (at 2mm depth) the peak of ettringite is higher than the one measured in the material bulk (at 90mm depth) and on the opposite, the peak of AFm close to the contact surface is smaller than deeper in the sample. This last point let think that ettringite near to the contact surface forms from AFm in sufficient presence of sulfate (3.00wt.-% according to titration results). Considering the CEM II/A-D concrete the peak characteristic for AFm is to be observed, confirming the EDS and XRD observations that no significant ettringite is formed in this sample.



Figure  $12 - {}^{Al}NMR$  spectra of the CEM I versus the CEM II/A-D concretes.

#### Qualitative phase assemblage along ion ingress profile

From the cumulated observations in SEM, XRD and in some instance ^{Al}NMR, qualitative phase assemblage profiles can be drawn as a summary (Fig. 13).



Figure 13 – Qualitative phase assemblages in the cementitious matrices of the CEM I (top left), CEM II/A-D (top right) and CEM I SR (bottom).

#### 4. CONCLUSIONS & PERSPECTIVES

The main ions from the sea water that can possibly penetrate concrete are chloride, sulfate and magnesium. They are firstly selected by their size relative to the cement pore network size range: chloride is expected to penetrate deeper than sulfate, which is in turn expected to penetrate deeper than magnesium. This order corresponds to the order of respective products formed from these ions from the bulk to the surface of the concretes: Friedel's salts, ettringite and then a thin surface layer of brucite. The ions are then in competition inside the cementitious matrix regarding their reactivity. According to their electronegativity, chloride (3.16) should first react, then sulfate (2.58) and finely magnesium (1.31). The relative amount of Friedel's salts formed is in general higher than the one of ettringite, but the chloride ions in sea water are by 10 times more available than the sulfate ions. In each of the three concretes studied, the first reaction zone at the surface consists in the formation of brucite with depletion in Friedel's salts and ettringite. In this zone total depletion of portlandite and decalcification of the C-S-H are also observed. The first reaction zone in surface is indeed the location of leaching due to the low pH of the surrounding sea water compared to the pH of concrete. Less dense decalcified C-S-H lets place for magnesium to penetrate the microstructure. Magnesium is smaller than calcium and stabilizes the hydroxide form brucite  $(Mg(OH)_2)$  over portlandite  $(Ca(OH_2))$ . This phenomenon is increased by the dissolution of portlandite due to leaching. Brucite is extremely stable at low pH and even contributes to the low pH of this first zone. Ettringite and Friedel's salts are AFm, which are not stable at low pH, especially in the case of ettringite at pH lower than 11.

Physical versus chemical effects of the cement composition and especially of silica fume addition have been discussed in terms of impacts on  $SO_4^{2-}$  and  $CI^-$  ions ingress profiles and subsequent phase assemblage changes. At this stage of the work, it is thought that the filler effect of silica fume is responsible of the limited sulfate (and carbonate) ions penetration. Nevertheless the microstructure densification is not high enough to avoid chlorides from ingressing. The small quantity of sulfate that could penetrate in the CEM II/A-D precipitated ettringite in free spaces (air voids, large pores), but was not sufficient to thermodynamically stabilise ettringite as intermixed in the C-S-H. If chlorides penetrate in the CEM II/A-D cementitious matrix as deep as in the CEM I, it is thought that the quantity penetrated is lower: Friedel's salts are only stabilised in the first 10mm in the CEM II/A-D concrete while they are present up to the bulk of the CEM I concrete. Magnesium and carbonate ions penetration is also reduced in the denser microstructure of the CEM II/A-D sample limiting brucite and calcite formation.

However, physical versus chemical effects of the CEM I SR cement on ions ingress and subsequent phase assemblage are rather unclear. It is believed that the low alumina content of such a mix design is responsible for the instability of ettringite and Friedel's salts due to the lack of AFm phases available. Nevertheless, further study on the porosity of the cementitious matrix such as SEM image analysis could discriminate the physical versus chemical effects.

Besides, this study points out the differences between observations in field and in common prescripted laboratory tests. On the contrary to most literature data on laboratory mortar specimens, the field samples studied present no cracking, no gypsum formation, no thaumasite formation, and no magnesium silicate hydrate (M-S-H) formation. The first two effects can be attributed to the low sulfate concentration of sea water compared to the sodium sulfate concentration usually used in laboratory tests: the equivalent Na₂SO₄ concentration of sea water is of 3-4g.L⁻¹, while concentrations in Na₂SO₄ in laboratory tests generally reach 50g.L⁻¹. In parallel to this field study, laboratory samples are stored in 3g.L⁻¹ Na₂SO₄ solution at 20°C in

order to check the effect of low sulfate concentrations on phase assemblage and damage processes. The fact that no thaumasite formation is observed in the field samples despite the low temperature in the North Sea can be attributed to a reduced carbonation compared to laboratory conditions. The absence of M-S-H gel in these field concretes is rather surprising according to previous observations on field samples from the literature. The brucite layer formed in surface might be too dense to allow further magnesium penetration: no enough magnesium would be available to react with C-S-H or with decalcified C-S-H.

In order to better evaluate the results of this study, the penetration profiles must be quantified, this is in progress now using micro-XRF quantification mapping. Additionally, the phase assemblage inside the cementitious matrix should be at least semi-quantified using thermodynamic modelling of the corresponding systems with GEMS modelling program based on free Gibbs energy minimisation.

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# **COIN – and durability of structures**







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### ABSTRACT

COIN is one of 14 Centres for research based innovation (CRI) – which were set up after initiative of the Research Council of Norway. The duration of these centres is 8 years with an evaluation after 4 years. Both SINTEF and NTNU have a history for close contact with the concrete industry in Norway and the building industry in Norway has earlier documented a great potential for added value through innovation. The vision of COIN is creation of more attractive concrete buildings and constructions. This may be fulfilled by developing advanced materials, efficient construction techniques and new design concepts combined with more environmentally friendly material production. COIN shall be staffed from Sintef, NTNU and industrial partners with all together about 25 man-labour years. In addition there shall be 8-10 PhD-students continuously working at as well as many MSc-students. It is further hoped to attract many international guest researchers for COIN.

**Key words:** Concrete innovation, research project, durability, industrial partners

# 1. INTRODUCTION TO COIN

COIN stands for Concrete Innovation Centre and is one of 14 Centres for research based innovation (CRI) – which were set up after initiative of the Research Council of Norway [1]. The purpose of the centres for research based innovation is to build up and strengthen Norwegian research groups that work in close collaboration with partners from innovative industry and innovative public enterprises. It is further to support long-term research that promotes innovation and the competitiveness of Norwegian industry. The duration of these centres is 8 years with an evaluation after 4 years.

Both SINTEF and NTNU have a history for close contact with the concrete industry in Norway and the building industry in Norway has earlier documented a great potential for added value through innovation. It should also be mentioned that concrete work is an important part of construction works and the whole industry has many challenges related to environmental issues. We also see that recruiting is a greater challenge for the building industry than for many other industries.

The objective of COIN is to bring the development a major leap forward by developing advanced materials combined with efficient and sustainable construction techniques and design concepts and to increase the level of competence and strengthen the degree of innovation in the concrete business.

The vision of COIN is creation of more attractive concrete buildings and constructions.

Attractiveness implies aesthetics, functionality, sustainability, energy efficiency, indoor climate, industrialised construction, improved work environment, and cost efficiency during the whole service life. This may be fulfilled by developing advanced materials, efficient construction techniques and new design concepts combined with more environmentally friendly material production.

SINTEF Building and Infrastructure is the host of COIN, and the Norwegian University of Science and Technology is the main research partner. The industrial corporate partners are leading multinational companies in the cement and building industry and the aim of COIN is to increase their value creation and strengthen their research activities in Norway. The industrial partners are: Norcem AS, Rescon Mapei AS, Borregaard LignoTech, maxit Group AB, Unicon AS, Aker Solutions ASA, Spenncon AS, Veidekke Entreprenør AS, Skanska Norge AS and Norwegian Public Roads Administration.

The objectives of COIN are to increase the reputation of concrete to make it the natural and environmentally correct choice, appear as the leader in research and application, to attract leading international companies to increase its research activities in Norway and to contribute to increased education of researchers and masters.

COIN shall be staffed from Sintef, NTNU and industrial partners with all together about 25 man-labour years. In addition there shall be 8-10 PhD-students continuously working at as well as many MSc-students. It is further hoped to attract many international guest researchers for COIN.

# 2. **PROJECTS IN COIN**

There are presently five projects in COIN [1]:

# **1** Advanced cementing materials and admixtures

- Cements with lower CO₂ emission during production
- Admixtures to control hydration development
- Cements and admixtures to prevent cracking
- Alternative pozzolanas
- Cements with lower porosity

### 2 Improved construction technique

- Concretes with high fibre content to be used in load carrying structural parts
- Concretes and production techniques to give good looking surfaces
- Technology for production of optimal crushed aggregate

# **3** Innovative construction concepts

- Design and verification basis for utilization of fibres in load carrying structures
- Development of superlight high performance aggregate and concrete
- Hybrid structures development of new material combinations, e.g. to be used in arctic environment

# 4 Service life design

- Reliable tools for service life design included design of load capacity in structures under reinforcement corrosion or ASR
- Preventive measures to increase service life (surface treatments, inhibitors, low corrosive reinforcement, etc)

### 5 Energy efficiency and comfort

- Utilization of the thermal mass to reduce the need for cooling/heating of buildings

# 3. PHD-STUDENTS IN COIN

At all times it is planned to have 8-10 PhD students [1]. At the moment (October 2008) there are eight and on durability there are two at the moment, Jan Lindgård working with ASR and Ueli Angst on critical chloride content. At the moment one PhD student is lacking in resistivity of concrete. Recently an advertisement for 6 new students has been made public. The advertisement was published in the home pages of NAV (The Norwegian Labour and Welfare Administration), Jobbnorge (Work in Norway), Teknisk Ukeblad (a weekly technical paper) and the NTNU home page. The English version is in the EU portal of the NTNU page. A reference announcement is in the written issue of Teknisk Ukeblad August 8.

# 4. PART PROJECTS ON CONCRETE DURABILITY

- 1 Service life modelling and prediction
  - Develop operational and reliable tools for service life prediction of concrete structures exposed to chloride induced corrosion

Service life modelling and prediction is mainly to identify models for chloride ingress and corrosion and to collect data for relevant model parameters. Other part projects will deliver input parameters to the identified Service life models. It is the intention to have statistical quantification of model parameters and reliability based service life prediction for chloride

induced corrosion. As is seen corrosion of steel in concrete is the main cause (in COIN) for changes of the service life of reinforced concrete structures.

- 2 Critical chloride content and corrosion process
  - Study both in laboratory and in the field new and used methods for obtaining critical chloride content
  - Develop a method for critical chloride content
  - Investigate stainless steels as concrete reinforcement
  - Investigate the relation between concrete resistivity and the corrosion process

A PhD student, Ueli Angst from Switzerland is engaged. He has written most of a state of the art report on Critical Chloride Content and contributed much to a workshop that was held June 5 and 6 this year (2008).

- 3 Electrical resistivity
  - Establish relations between resistivity and reinforcement corrosion, and thereby develop service life models that include resistivity. Standardised methods for measuring resistivity will be suggested

This part project is now seeking a PhD student. The main goals of the project is to model the time dependent behaviour of resistivity, to identify any threshold values of resistivity and establish relations between resistivity and reinforcement corrosion, and thereby develop service life models that include resistivity. It is the intention to do laboratory tests versus field tests and to see whether there is a need for any compensation. It is further the intention to contribute to standardisation of test methods and recommendations.

- 4 ASR mechanisms and performance based concepts
  - To provide improved understanding of the accelerated AAR effects when concrete is subjected to elevated temperature and moisture regimes during various laboratory (performance) testing, compared to field conditions

A PhD student, Jan Lindgård from Norway is engaged. The main goals of the PhD study is to evaluate accelerated ASR effects when concrete is subjected to elevated temperature and moisture regimes during various laboratory "performance" testing, compared to field conditions, to produce a state-of-the art paper covering the key parameters that may influence the speed and extent of alkali aggregate reactivity of a concrete and address the research needs on important parameters. It is further the intention to summarize and evaluate the performance testing performed in Norway during the last ten years and to document and evaluate the influence of the selected issues / parameters for study on the results and outcome from the performance testing with the various test methods selected. Together with the supervisor, Terje Rønning of Norcem, the PhD project shall give input to the RILEM TC ACS-P sub-group as basis for the development of a future international agreed performance testing concept for evaluation of the alkali reactivity of binders/aggregate/mix design combinations.

- 5 Preventive measures
  - The objective is to study the effect of different preventive measures, i.e. surface protection, low-corrosive steel reinforcement and corrosion inhibitors, on the service life of concrete structures exposed to corrosion inducing substances.

The intention with this project is firstly the use of laboratory study to map factors that are contributing to ageing of hydrophobic impregnations (factors that may cause ageing are temperature and temperature fluctuations, moisture and detrimental chemical substances such as alkalis. The mapping will be performed using accelerated test regimes). In this phase chemical and mechanical methods of characterization to will be used to study the test samples. Finally the intention is to study the effect of corrosion inhibitors on the critical chloride content.

- 6 Residual service life & load bearing capacity
  - The objective is to model and predict residual service life and load bearing capacity of concrete structures with on-going reinforcement corrosion for different limits states

The intention of this project is to come up with models for the propagation period (corrosion process) and modelling of corroding structures. The intention is to test the instrumentation and monitoring methods that are used in the assessment of corroding structures. The intention is further to collect data on corroded concrete elements under field exposure and thereby predict the residual service life and capacity of the structure. Finally numerical simulation (FE analysis) will be made of corroded concrete structures

# 5 LABORATORY EXPERIMENTS FOR DETECTING CRITICAL CHLORIDE CONTENT IN REINFORCED CONCRETE

(presented by Ueli Angst at the Workshop in Trondheim June 5 and 6)

The knowledge of chloride threshold values for initiation of reinforcement corrosion in concrete is important for service life predictions and service life design. A lot of research has been devoted to finding such values. The reported results for critical chloride contents scatter over a wide range, e.g. from 0.02 to 3.08% when expressed in the form of total chloride by weight of binder [2]. This is mainly related to the variety of possible measurement techniques, both for field studies and laboratory setups. At present, no standardised or accepted testing method for critical chloride content exists.

A literature review over nearly 40 references reporting chloride threshold values has concluded that many studies were not practice-related [2]. Main pitfalls in laboratory work on critical chloride content have been identified to be the procedure to introduce chlorides and the quality of the steel-concrete interface. The latter is affected by both the rebar characteristics and the properties of the matrix. In many works, smooth rebars have been used instead of ribbed bars and the rebars have been prepared by sandblasting, polishing, cleaning, etc. Experiments have been conducted in alkaline solutions, or in cement paste, mortar or concrete. It is evident that the properties of the steel-concrete interface in the case of, for instance, smooth and polished rebars embedded in cement paste differ from reality. This difference is considered to be important since the steel-concrete interface has been identified to be one of the major influencing factors with regard to critical chloride content [3]. Also, compaction is usually better for laboratory concrete in comparison with real concrete. With regard to the chloride introduction, several techniques are available to accelerate chloride penetration in order to avoid time-consuming experiments as in the case of pure diffusion. However, the situation is different from reality, e.g. in the case of mixed-in chlorides where the steel might not be able to initially passivate.

The literature evaluation [2] also revealed that most critical chloride contents have been measured in terms of total chloride content by weight of binder; free chloride contents have primarily been reported in the case of experiments dealing with solutions or porous cement paste/mortar/concrete. This is mainly due to the limitations of the pore solution expression technique, which is usually the one used for obtaining samples for analysis of free chloride concentrations in concrete pore solution. Up to now, there is a lack of information on the critical chloride content in dense concrete (low w/c ratio, alternative cement types) on the basis of free chloride.

# 5.1 Current PhD project on critical chloride content

The current project aims at measuring the critical chloride content based on both free and total chloride contents in laboratory concrete samples with a dense matrix. Emphasis is put on realistic and practice related conditions with regard to the factors mentioned in the previous section.

For the measurement of the free chloride content, embedded "chloride sensors" are used. These sensors consist of Ag/AgCl electrodes and have been used earlier by other researchers [4].

### 5.2 Chloride sensors – direct potentiometry in concrete

The use of ion selective electrodes (ISE) in direct potentiometry is well established and has long been used in many fields such as analytical chemistry to determine the ionic activity of a certain species in aqueous solutions by a potential measurement. However, the situation in a cement based material such as concrete is more complicated.

### Sensitivity and error sources

Potentiometric measurements are very sensitive to variations in potential. The sensitivity directly follows from the slope of measured calibration curves or from the theoretical slope of 59.2 mV/decade. If only small deviations in potential are considered (in the range of only a few mV), it can be calculated that an error in potential of 1 mV results in a relative error in chloride concentration of ca. 4 percent [5].

Several phenomena have been identified to disturb accurate measurements of sensor potentials when measuring in cement based materials [6]. When an external reference electrode is used by establishing contact to the concrete surface with a wetted sponge the situation can be schematically depicted as in Fig. 1. Several components contribute to the measured potential, namely the *liquid junction potential* at the interface between inner solution of the reference electrode and the wetting agent in the sponge  $E_{RE-S}$ , the *liquid junction potential* at the boundary between the wetting agent in the sponge and the concrete pore solution  $E_{S-C}$ , as well as *membrane potentials*  $E_C$  across the concrete, and eventually *iR drops*. Both liquid junction and membrane potentials are all so-called *diffusion potentials* and arise due to concentration gradients in combination with differences in mobility between the diffusing ions. The significance of diffusion potentials for electrochemical measurements in concrete has been recently discussed by the author in ref. [6].

#### Consequences for experimental setups

In order to measure reliable free chloride contents in concrete with potentiometric sensors one has to be very careful with regard to the setup. Liquid junction potentials at the interface between an external reference electrode and the sample surface can be minimised (but never completely avoided) if the reference electrode is contacted to the concrete with an appropriate solution [6]. With regard to membrane potentials the position of the reference electrode appears to be decisive: To minimise contributions from membrane potentials arising from internal concentration gradients the reference electrode should ideally be placed at the same depth as the chloride sensors (e.g. by use of embedded reference electrodes).



*Figure 1* Disturbing phenomena when measuring potentials in concrete

# 5.3 Conclusions on the PhD project

The following sums up the current project plans and the results obtained so far:

- 1 The current project aims at using a practice-related testing setup for determining the critical chloride content in dense concrete, based on both free chloride and total chloride contents.
- 2 Ag/AgCl electrodes can be used as ion selective electrodes embedded in concrete to measure the free chloride content non-destructively. However, the technique is highly sensitive to errors in potential measurements arising from phenomena such as diffusion potentials. This is not a problem of the sensor itself, but of potential measurements through concrete as such.

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