

## DURABILITY OF CONCRETE - QUALITY CRITERIA

Olav Gautefall  
M. ScTech



Magne Maage  
Dr.ing.

Cement and Concrete Research Institute  
SINTEF div FCB  
N-7034 Trondheim

### ABSTRACT

The rate of carbonation and penetration of chlorides into concrete have been studied by field exposures and accelerated laboratory test methods. The results are compared. Five concretes of different material composition but of same compressive strength have been tested. Other experimental variables were use of silica fume, curing condition and cement content. Accelerated test methods are found to classify the different concretes as found in field exposure. Curing condition and silica fume had a significant effect. Cement content had no effect.



### Key words:

Material science, concrete, reinforcement corrosion, durability, chlorides, carbonation, test methods

## 1 INTRODUCTION

In Norway it has traditionally been accepted that there exists a relationship between compressive strength and the durability of reinforced concrete structures. However, recent advances in cement development and concrete technology have changed this relationship. Today it is possible to produce concretes of a given compressive strength grade within a wide range of material composition, e.g. water-to-cement ratio which is the most important durability controlling factor.

The current study attempted to improve the level of understanding of how to specify the composition of a concrete according to environmental exposure. This has been done by:

- 1) Comparing chloride diffusion parameters and the rates of carbonation for concretes of the same strength grade, different binder composition and with different amounts of binder.
- 2) Use of accelerated test methods to evaluate concrete durability properties like carbonation and chloride diffusion.

## 2 TEST SPECIMENS

### 2.1 Concrete composition

The composition of the mixes tested are shown in Table 1. All mixes were designed to meet the requirement of a C25 concrete according to Norwegian Standard. This strength grade is widely used in Norway (e.g. in building facades and walls) and the water-to-cement ratio might be as high as 0.9 - 1. The cement content of the mixes varied from 260 kg/m<sup>3</sup> (medium) to 162 kg/m<sup>3</sup> (lean). In mix no. 3 and 4, 10 % silica fume by weight of cement was added. The water-to-cement ratio varied from 0.77 to 1.03. Plasticizer and superplasticizer were used to obtain necessary workability.

Table 1. Mix proportions (kg/m<sup>3</sup>)

Component	Mix No.				
	1	2	3	4	5
Cement (MP30-10 % fa) *)	260	230	180	162	210
Silica fume	0	0	18	16.2	0
Filler	80	80	80	80	80
Sand	930	956	970	988	973
Crushed stone	930	956	970	988	973
Water (total)	200	179	182	166	166
Plasticizer	0	1.8 l	1.9 l	2.0 l	2.0 l
Superplasticizer	0	0	0	3.5 l	3.3 l
w/c-ratio	0.77	0.78	1.01	1.03	0.79
w/c+s-ratio	0.77	0.78	0.92	0.94	0.79

\*) Blended cement, 10 % fly ash

The physical properties of the concretes are listed in Table 2. The compressive cube strength after 28 days of water curing varies from 28.7 MPa (mix no. 3) to 32.9 (mix no. 4). This implies that the concretes fulfill the designed quality based on compressive strength criteria. It should also be noted that the strength development of the concretes containing silica fume are somewhat slower than that of the other concretes.

Table 2. Physical properties

Physical parameters	Mix No.				
	1	2	3	4	5
Slump (cm)	14	17	14	12	17
Unit weight (kg/m <sup>3</sup> )	2408	2418	2392	2416	2446
Compressive strength (MPa)					
7 days, water curing	23.7	23.7	17.6	19.6	28.5
7 days, air curing	24.7	24.8	18.4	19.1	29.9
28 days, water curing	28.8	28.8	28.7	32.9	31.6
28 days, air curing	32.9	32.0	29.4	30.4	31.5

## 2.2 Production program

Out of each mix the following type of concrete specimens were cast:

1. 10 x 10 x 60 cm prisms
2. 10 x 10 x 10 cm cubes
3. 25 x  $\phi$ 10 cm cylinders
4. 25 x  $\phi$ 10 cm cylinders with a centered  $\phi$ 10 mm steel rod

## 2.3 Curing condition

The specimens were demoulded 20 hours after casting. Thereafter one half of the prisms and cubes were aged in water and the other half in laboratory temperature and atmosphere (20°C, 50 % RH) in 6 months before further testing or exposure. The cylinders were all water cured until testing.

## 3 TESTING PROGRAM

### 3.1 Field

#### Chloride penetration

The concrete prisms were exposed in the following environments:

1. In flowing fresh sea-water (taken from 20 depth) in a laboratory tank
2. In the tidewater zone
3. In the sea at 20 m depth

The chloride content of the sea-water was 1.9 % by weight.

### Carbonation

The concrete prisms were exposed in the following unsheltered environments:

1. Coastal climate (Trondheim fjord)
2. Urban climate (Trondheim)
3. Rural climate (Trøndelag)
4. Indoor, laboratory (20 °C, 50 % RH)

### 3.2 Laboratory

#### Chloride penetration

##### Method I

This method is similar to a method used by Federal Highway Administration, USA /1/. The method is an accelerated impressed current test which measures time to failure of reinforced concrete specimens.

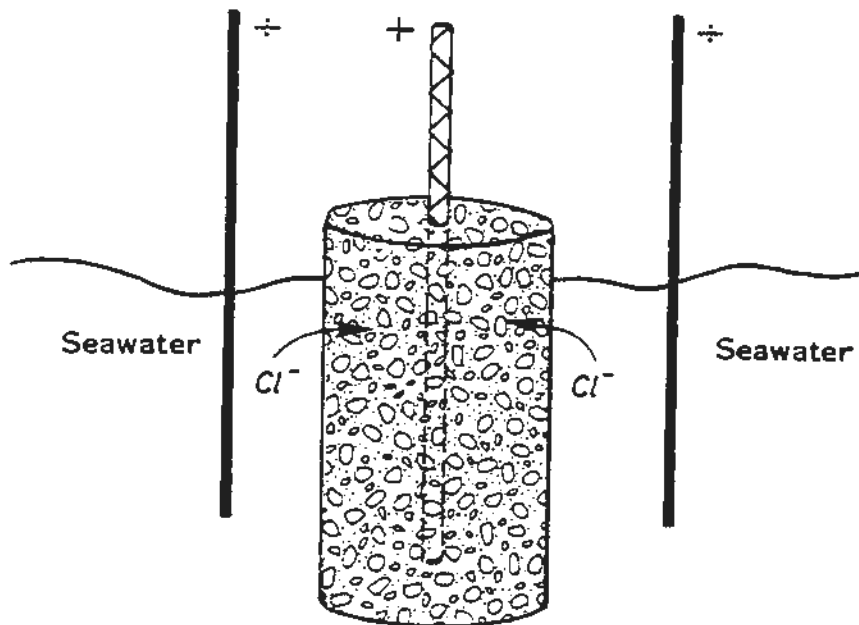


Fig 1. Polarization of embedded steel. Experimental arrangement

A constant direct current field of 5 volt was kept between the embedded steel (positive) and a steel sheet (negative) outside the concrete cylinder. The electric current in the circuit was recorded and when chlorides reaches the reinforcing steel, the current will increase (Fig 2). Time to

failure is defined as time corresponding to the observation of crack. In addition to cracks, brown colored liquid can be seen on the concrete cylinder.

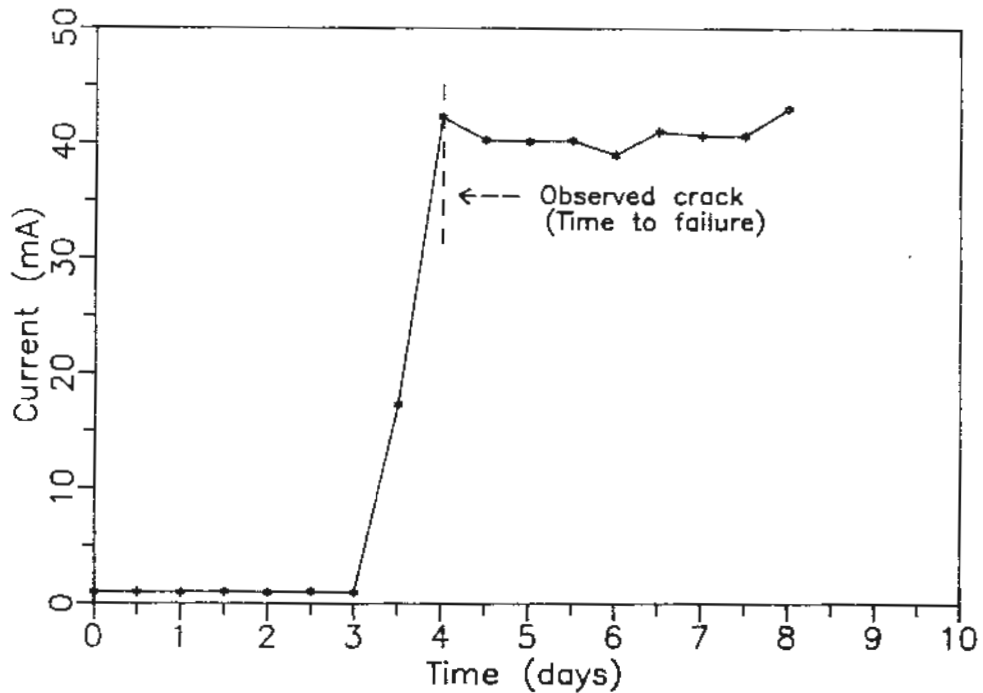


Fig 2. Polarization of embedded steel. Current vs time relationship

### Method II

Another laboratory method used to study the relative chloride permeability of concrete is the AASHTO test T277-831 "Rapid Determination of the Chloride Permeability of Concrete" /2/. It consists of monitoring the electrical current passed through 95 mm diameter by 51 mm long concrete cores when one end of the core is immersed in a sodium chloride solution and a potential gradient of 60 volt DC is maintained across the specimen for 6 hours. The total charge passed is said to be related to chloride permeability. Actually, the charge passed during 6 hours is mainly dependant upon the electrical resistivity of the concrete. So far reported, however, this method gives good correlation to practical experience. It should also be noted that the heat evolution in the test cell might reduce the experimental reproducibility. The test results are evaluated from Table 3 according to /3/.

Table 3. Chloride permeability based on charge passed /3/

Charged passed (Coulombs)	Chloride permeability
> 4000	High
2000-4000	Moderate
1000-2000	Low
100-1000	Very low
< 100	Negligible

### Carbonation

The specimens were exposed in an atmosphere of 3 % carbon-dioxide and 60 % RH.

## 4 RESULTS AND DISCUSSION

### 4.1 Chloride penetration

#### Field

The chloride profile of the specimens after 17 months of exposure are plotted on Figs 3-5 (water cured) and Figs 6-8 (air cured). The total chloride content, which is the sum of free chloride in the porewater and bound chloride, was determined by a potentiometric titration method.

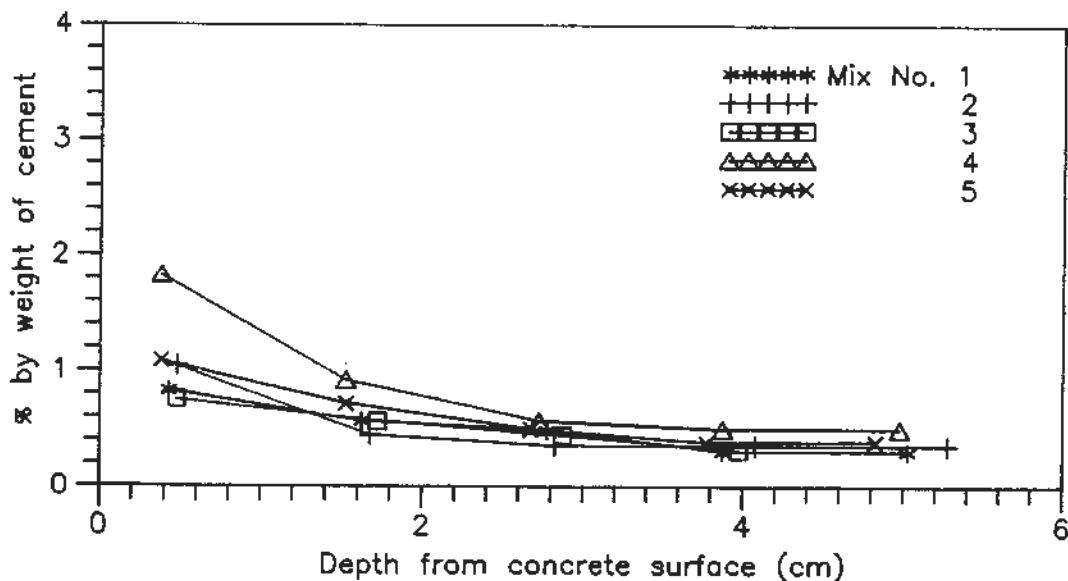


Fig 3. Chloride profiles. Water cured 6 months before 17 months exposure in flowing sea water (temperature 5-10°C)

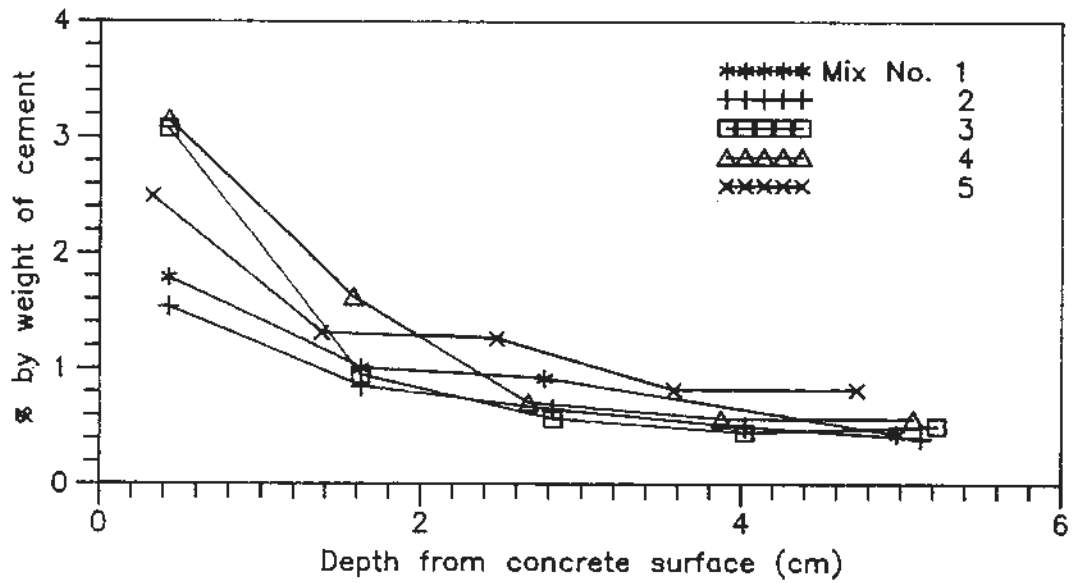


Fig 4. Chloride profiles. Water cured 6 months before 17 months exposure in the tidewater zone

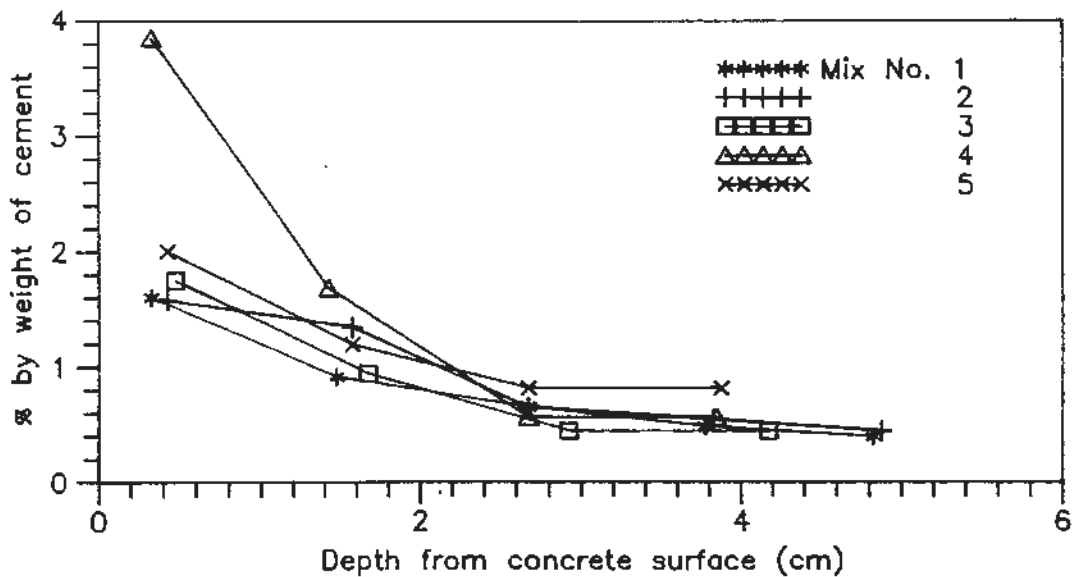


Fig 5. Chloride profiles. Water cured 6 months before 17 months exposure at 20 m depth in the sea (temperature 4-10°C)

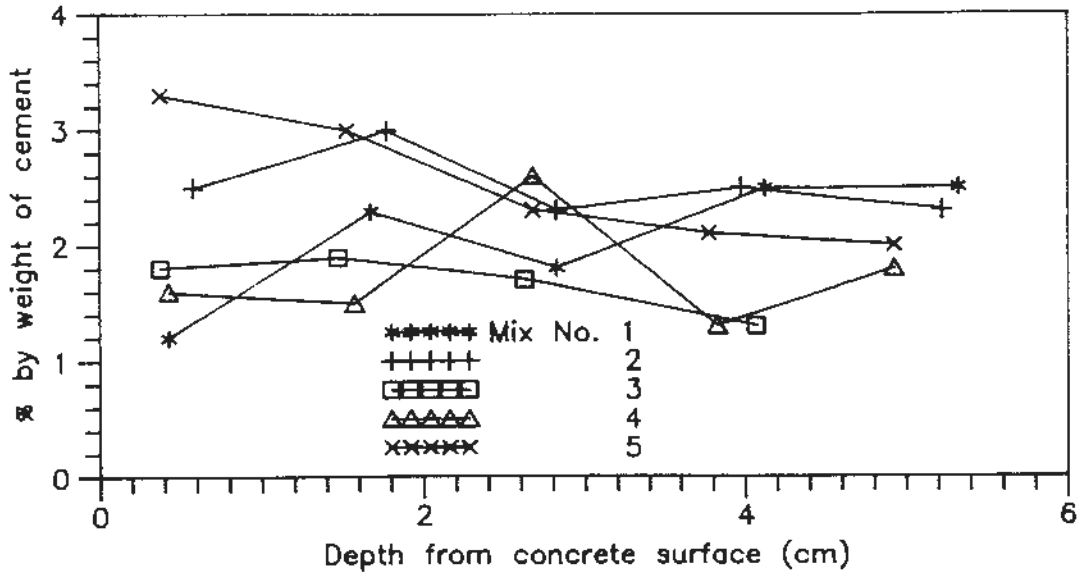


Fig 6. Chloride profiles. Air cured 6 months before 17 months exposure in flowing sea water (temperature 5-10°C)

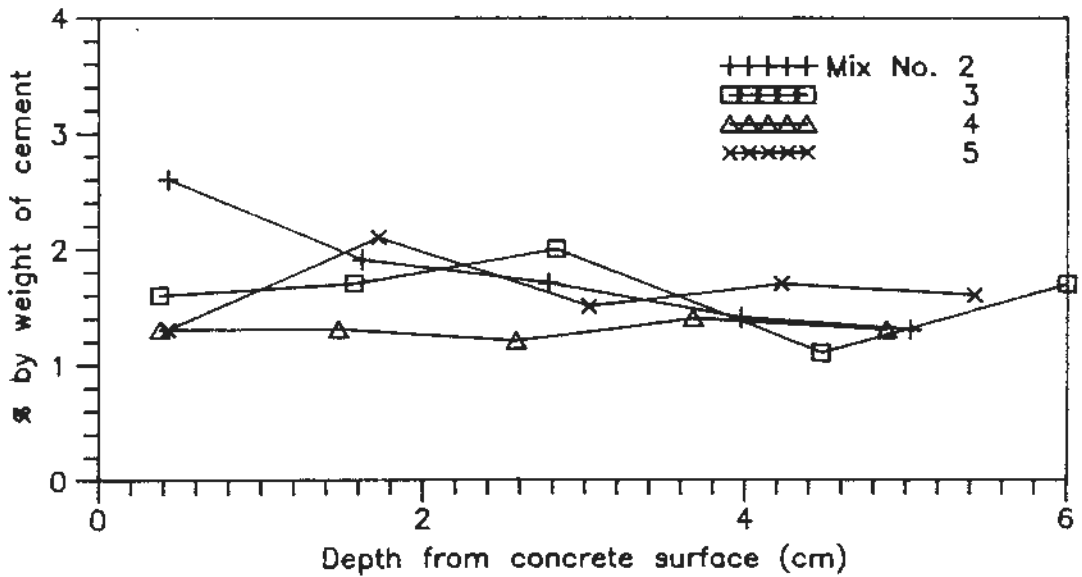


Fig 7. Chloride profiles. Air cured 6 months before 17 months exposure in the tidewater zone



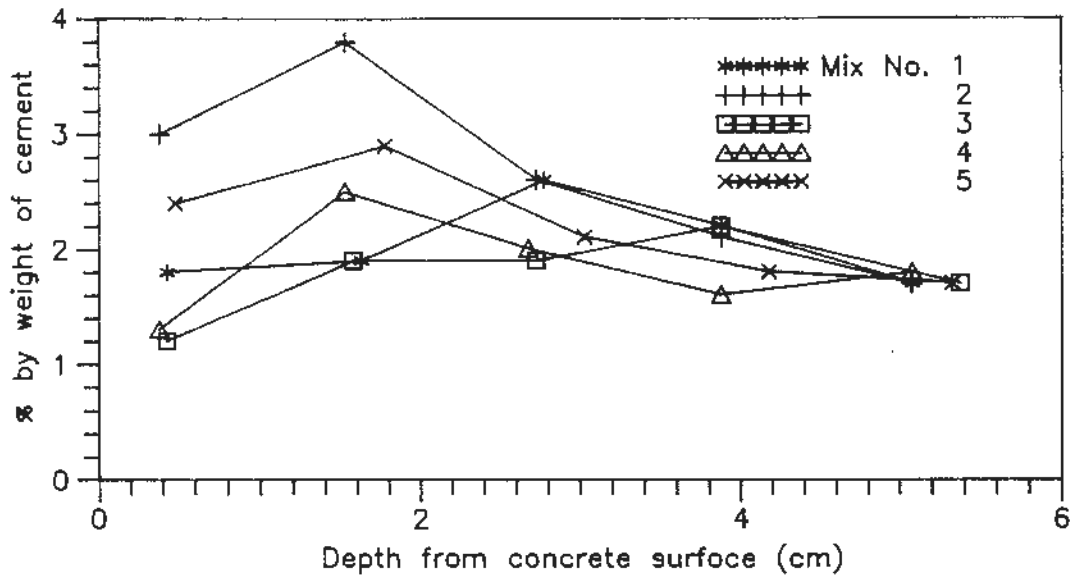


Fig 8. Chloride profiles. Air cured 6 months before 17 months exposure at 20 m depth in the sea (temperature 4-10°C)

It is evident from Figs 3-8 that the curing condition has a dominating effect on the ingress of chloride. The air cured specimens (Figs 6-8) have to some extent been dried out and when exposed to sea water, chlorides have been transported into the concrete by water conveyance.

The ingress of chloride into concrete cured in water before exposure is an ordinary diffusion process which obeys Fick's second law /4/, which states:

$$\delta C / \delta t = D_e * (\delta^2 C / \delta x^2) \quad (1)$$

The solution of equation (1) for one dimension diffusion is according to /5/:

$$C(x,t) - C' = (C_0 - C') * \{1 - \text{erf}[x / (2 * \sqrt{D_e * t})]\} \quad (2)$$

where:

- C(x,t): Chloride concentration at x and t (weight %)
- C<sub>0</sub> : Chloride supply concentration (weight %)
- C' : Background chloride concentration (weight %)
- x : Depth from concrete surface (cm)
- t : Exposure time (s)
- D<sub>e</sub> : Effective diffusion coefficient (cm<sup>2</sup>s<sup>-1</sup>), i.e. the diffusion coefficient is assumed to be constant.

In equation (2)  $C(x,t)$ ,  $C'$ ,  $x$  and  $t$  are measured values.  $C^0$  and  $D_e$  are determined by successive approximation to give the best fit according to the method of least square.

The chloride supply concentration,  $C_0$ , is the total chloride content close to the concrete surface. The value of  $C_0$  is therefore higher than the content of free chloride close to the concrete surface,  $C_{sw}$  (weight %) which correspond to porewater in contact and in equilibrium with sea-water.

The background chloride concentration,  $C'$ , is defined as the total chloride content in the interior of the concrete. In the result presented here this correspond to the chloride content at 5 cm depth from the concrete surface (see Figs 3-5).

The effective diffusion coefficient,  $D_e$ , is determined by the slope of the chloride profile. A less permeable concrete has a low  $D_e$  (more steep curve) compared to a more permeable concrete with a high  $D_e$  (less steep curve).

The free chloride content at the concrete surface,  $C_{sw}$ , has been calculated and the result are presented in Table 4. It has been assumed that all pores (i.e. gel, capillary, contraction and air pores) are waterfilled and the water has a chloride content of 1.9 % which correspond to the 3 % sodium chloride content of sea-water. The degree of hydration was assumed to be 75 % at water-to-cement ratio 0.8 and 80 % at water-to-cement ratio 1.0. The values of  $C'$  in Table 4 are from Figs 3-5.

Table 4. Free chloride content at the concrete surface,  $C_{sw}$ , and background chloride content,  $C'$ , of water cured concretes. Exposure conditions 1-3: See Chapter 3.1.

Mix No.	% by weight of cement			
	$C_{sw}$	$C'$		
		1	2	3
1	1.3	0.30	0.40	0.40
2	1.4	0.35	0.40	0.45
3	1.8	0.30	0.45	0.45
4	1.9	0.45	0.55	0.55
5	1.4	0.35	0.80	0.80

From the experimental data in Figs 3-5  $C_0$  and  $D_e$  have been calculated and are presented in Table 5. The  $C_0$  values in brackets are lower than the values of  $C_{sw}$  in Table 4. These values and the corresponding values of  $D_e$  are therefor not subject to further calculation and discussion.

Table 5. Calculated chloride supply concentration,  $C_o$ , and effective diffusion coefficient,  $D_e$ , of water cured concretes. Exposure conditions 1-3: See Chapter 3.1.

Mix No.	$C_o$ (% by weight of cement)					$D_e * 10^8 \text{ cm}^2/\text{s}$				
	1	2	3	Av.	S.D.	1	2	3	Av.	S.D.
1	[0.9]	2.0	1.8	1.9	0.1	[5.4]	5.8	3.6	4.7	1.6
2	1.4	1.7	1.9	1.7	0.3	1.4	4.2	5.4	3.8	1.8
3	[0.8]	4.2	2.1	3.2	1.5	[6.2]	1.3	2.6	2.0	0.9
4	2.2	3.9	4.7	3.6	1.3	2.2	2.5	1.8	2.2	0.4
5	[1.2]	2.8	2.4	2.6	0.3	[3.5]	2.4	1.9	2.2	0.4

According to Table 5 the concretes exposed in flowing seawater in the laboratory (exposure condition 1) have lower  $C_o$  values and higher corresponding  $D_e$  values than the other concretes. The reason for this is not known.

The concretes made with 10 % silica fume (mix no. 3 and 4) have higher  $C_o$  values and lower  $D_e$  values than concretes without silica fume. This might be due to higher chloride binding capacity of concretes made with silica fume as previously reported /6/, /7/.

In Table 6 the quotients between  $C_o$  (av.) from Table 5 and  $C_{sw}$  from Table 4 are presented. A high value of the quotient indicate a higher chloride binding capacity than a lower value. According to Table 6 the concretes made with 10 % silica fume (mix no. 3 and 4) have higher binding capacity than mix no. 1 and 2 which are without silica fume. Mix no. 5 (without silica fume), however, shows the same binding capacity as mix no. 4.

Table 6. Calculated quotient of chloride supply concentration,  $C_o$  (av.) and free chloride concentration at concrete surface,  $C_{sw}$ .

Mix No.	% by weight of cement		$C_o \text{ (av.)}/C_{sw}$
	$C_o \text{ (av.)}$	$C_{sw}$	
1	1.9	1.3	1.5
2	1.7	1.4	1.2
3	3.2	1.8	1.8
4	3.6	1.9	1.9
5	2.6	1.4	1.9

Laboratory testing

The test results are listed in Table 7 (Method I - polarization of embedded steel) and Table 8 (Method II - 60V potential gradient). Both methods use impressed current to accelerate the transport of chloride ions (and other negative ions) through the concrete. The free diffusion of chloride is superseded by ionic migration in an electric field as transport mechanism. In addition the mass transport through the concrete is influenced by the effect of the so-called double layer /4/ (electro-osmosis). Since the accelerated methods introduce other transport mechanisms it is difficult to compare results with natural exposure.

Table 7. Chloride penetration - polarization of embedded steel method

Mix No.	Silica fume (%)	W/c-ratio	W/c+s-ratio	Time to failure (days)		
				# 1	# 2	Average
1	0	0.77	0.77	4	7	5.5
2	0	0.78	0.78	6	8	7
3	10	1.01	0.92	12	16	14
4	10	1.03	0.94	11	16	13.5
5	0	0.79	0.79	5	7	6

The specimen made with 10 % silica fume had a greater lifetime than the other concrete qualities despite of the higher water-to-cement ratio and the lower cement content. The cement content of the mixes does not influence the time to failure.

Table 8. Chloride penetration - potential gradient method

Mix No.	Charge passed (Coulombs)				Classification (table 3)
	# 1	# 2	# 3	Average	
1	2200	2160	2170	2180	Moderate
2	1520	1800	1800	1710	Low
3	780	990	960	910	Very low
4	690	800	760	750	Very low
5	1550	2000	1720	1760	Low

The specimens made with 10 % silica fume had a much lower charge passed than specimens without silica fume. The

charge passed is not influenced by the cement content. The effect of silica fume as a cement replacement on the ingress of chloride is much more significant in the accelerated laboratory tests than what is observed during field exposure. This can be attributed to the influence of silica fume on the pore system of the concrete. Silica fume might reduce the ionic migration and/or electro-osmosis properties of the concrete.

Both the natural exposure tests and the accelerated tests indicate that a concrete with 10 % silica fume of cement weight give better protection against reinforcement corrosion than a concrete made without silica fume. In addition concrete with silica fume has a higher electric resistance /8/. This will reduce the corrosion rate if the corrosion process is of macro-cell type (localized corrosion). In a micro-cell corrosion process (uniform corrosion) the effect of electric resistance is small.

#### 4.2 Carbonation

##### Field

The measured carbonation depths after 3 years of exposure are given in Table 9 and 10. The listed values are maximum values, i.e. carbonation depth of the side which has the highest typical value. The depth of carbonation was determined by using phenolphthalein indicator. (See Fig 9.)

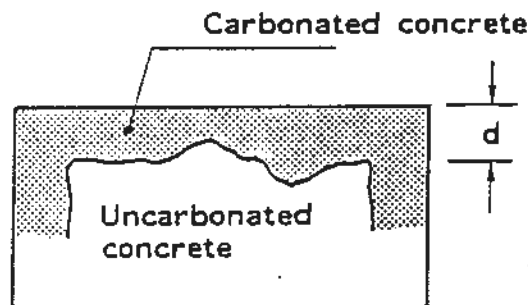


Fig 9. Measured carbonation depth.  $d$  is the typical value for side 1 of the specimen

Table 9. Carbonation depths (mm). Specimens water cured 6 months before exposure

Mix No.	Exposure conditions (3 years unsheltered)			
	Costal area	Urban area	Rural area	Buried in peat soil 1)
1	< 1	1.5	0	1
2	< 1	1.5	< 1	0.5
3	4	5	3	1.5
4	3	5	3	1
5	1	3	0	3

1) 2.5 years of field exposure

Table 10. Carbonation depth (mm). Specimens air cured 6 months before exposure

Mix No.	Exposure conditions (3 years unsheltered)				
	Costal area	Urban area	Rural area	Buried in peat soil 1)	Indoor 2)
1	8	7.5	4.5	2.5	12.5
2	8	8	6.5	3	12
3	10	10	6	2.5	16
4	9	9	7	5	18
5	5	5	6	3.5	16

1) 2.5 years field exposure

2) 3.5 years exposure

The rate of carbonation in specimens with 10 % silica fume is found to be somewhat higher than in specimens without silica fume despite of curing condition. This is in accordance with a previous investigation /9/. There is no significant difference between depth of carbonation and cement content of concrete specimens without silica fume. Extra water curing highly reduce the rate of carbonation as earlier reported /9/, /10/, /11/.

#### Laboratory testing

It should be noted that during the first 3-4 weeks of exposure the carbondioxide consumption was so great that the actual concentration was lower than the specified 3 % in the conditioning chamber. The depth of carbonation was determined by using phenolphthalein indicator. (See Fig 9.)

The results from the accelerated tests are given in Table 11.

Table 11. Carbonation depth (mm) after different exposure time (weeks). Exposure condition: 22°C, 3 % CO<sub>2</sub>, 60 % RH

Mix No.	Pre-treatment					
	Water cured - 6 months			Air cured - 6 months		
	Initial depth: 1 mm			Initial depth: 4 mm		
	8	21	90	8	21	90
1	3	7	>50	10	18	>50
2	2	10	25	10	20	>50
3	4.5	12	35	13	24	>50
4	4	6	28	15	29	>50
5	2	8	22	14	20	>50

Concrete specimens made with 10 % silica fume have a higher rate of carbonation than specimens made without silica fume. Extra water curing highly reduces the rate of carbonation and the cement content has no effect. These results are in accordance with the results from the field test.

## 5 CONCLUSIONS

From the results reported here, the following conclusions can be drawn:

1. Water curing of concrete after demolding reduces both the rate of chloride penetration and the rate of carbonation.
2. There was no effect of cement content.
3. The accelerated methods used for testing of concrete resistance against chloride ingress gave a good classification between concrete qualities. Based on calculated efficient diffusion coefficients and chloride supply concentrations from the field tests the differences were smaller, however, the classification was the same as in accelerated tests.
4. Exposure tests in 3 % carbondioxide and 60 % RH gave a good agreement with field exposures tests. 20 weeks seems to be enough time to separate between concrete qualities.

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