# Silica fume concrete after 155 months

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## ABSTRACT

Long-term experimental studies on the interaction between silica fume and Portland cement in concrete subjected to air, water or sealed curing, are outlined. For this purpose 6000 kg of eight qualities of concrete were studied at 5 ages over a period of 155 months. Half of the concrete contained silica fume. Parallel studies of strength, hydration and relative humidity were carried out. After 155-month tests were also performed on the salt freezing and thawing frost resistance of the concrete. The results show a great effect of silica fume on compressive and splitting tensile strength, hydration, relative humidity in low-water-cement ratio concrete and on salt-thaw frost resistance of silica fume compared to the effect of Portland cement. The work was performed 1989 – 2003.

**Key-words:** Compressive strength, Hydration, Internal relative humidity, Splitting tensile strength, Salt-frost scaling, Silica fume.

## 1. INTRODUCTION, OBJECTIVE AND LIST OF SYMBOLS

### 1.1 Introduction

The novelty was the long-term aspect, i.e. studies on the same concrete over 155 months. For numerous years silica fume has been used to obtain high strength, high fluidity and other high qualities in concrete. However, the efficiency factor of silica fume compared to Portland cement as related to strength, hydration and self-desiccation has not yet been subject to long-term analysis, particularly not with regard to the effect of age and water-cement ratio. Reports have been presented over the years dealing with the decrease of strength of concrete over time due to content of silica fume [1]. Some of the observations have been explained by different moisture conditions in the concrete when the compressive tests were carried out [1]. The decrease of splitting tensile strength in a concrete with silica fume compared to Portland cement concrete has been related to the pronounced autogenous shrinkage that occurred in a concrete with silica fume which may cause micro-cracking [2]. The development of hydration differs substantially between concrete with and without silica fume [3]. Some calcium hydroxide is transformed into silicate hydrates which decreases the degree of hydration but increases the strength. No additional chemical shrinkage occurrs since no water was used during the pozzolanic reaction [3]:

$$3\ Ca(OH)_2 + 2\ SiO_2 = 3\ CaO.2\ SiO_2.3\ H_2O. \qquad (1)$$

These findings are related to extensive experimental studies performed on mortar with the same mix proportions as in this experiment (except for aggregate > 4 mm) and very clearly shows that the reduction of the water volume during hydration was related to the amount of Portland cement only [3]. Once the silica fume is consumed the properties of Portland cement concrete and silica fume concrete are quite different.

### 1.2 Objective

No other investigation in this field exists spanning of a 10-year period of time. It was the objective of this work to compare, over at least 10 years, compressive and splitting tensile strength, RH and hydration of sealed, air- or water-cured concrete with 10% silica fume with the corresponding properties of concrete without silica fume. Another purpose was to study salt freezing and thawing resistance of silica fume concrete after at least 10 years.

### 1.3 List of symbols

| | |
|---|---|
| $f_c$ | compressive strength with interlayers (MPa) |
| $f_{cc}$ | compressive strength without interlayer (MPa) |
| $f_{cd}$ | compressive strength at 5 months' age with 1 month of intensive drying at 55°C (MPa) |
| $f_{ct}$ | splitting tensile strength (MPa) |
| $f_{cw}$ | 5 months' age with 1 month of drying at 55°C followed by 1 month of water curing (MPa) |
| t | time (days, month) |
| RH | relative humidity (-, %) |
| S | 10% silica fume calculated on the amount of cement |

## 2. EXPERIMENTAL

### 2.1 Specimen

Compressive strength, $f_c$, splitting tensile strength, $f_{ct}$, and hydration were studied on cores, 80 mm long and 40 mm in diameter, drilled out of large specimens (250 kg each). Half of the specimens contained silica fume. All other material parameters were held constant. The concrete was poured in the shape of a disc, 1 m in diameter and 0.1 m thick. The flat sides of the disc were sealed by at least 2-mm thick layers of epoxy resin. Also the circular rim of one third of the specimens was sealed by a minimum of 2 mm epoxy resin. The diffusion of moisture through the epoxy resin was negligible compared to the diffusion through the porous concrete. The rims of one third of the specimens were subjected to a climate with a temperature varying between 18°C and 24°C and an ambient RH varying between 24% and 48% [4]. The rims of the remaining one third of the specimens were water-cured (submerged) for 155 months, Table 1. For studies of diffusivity and capillarity 100-mm circular specimens were used, 15 mm long for young concrete, 15 and 45-mm for mature concrete. The rim of the discs was treated with 2-mm epoxy.

### 2.2 Testing methods

All tests were carried out with three equal specimens in parallel. The average value of the three test results was used. At all ages, except for 155 months, cylindrical cores were taken in equal numbers at a distance of 50, 150 or 350 mm from the exposed surface in order to study strength and hydration. At 155 months' age cores were taken at about 100 mm distance from the exposed surface of the large concrete specimens. During the testing of strength 4.5-mm interlayers of hardboard were used. The width of the interlayers was 4.5 mm at the split tensile testing. The testing rate was 1 MPa/s (compressive strength) or minimum 30 s (split tensile strength). Cast-in plastic tubes were placed at different distances, 50, 150 and 350 mm, from the exposed surface in order to measure RH of the concrete. Parallel to the cast-in items, thermocouples were used [5]. The measurement period of RH was 22 h. The probes were carefully calibrated [6]. Three cylinders 40 mm in diameter and 80 mm long were submerged in 3% sodium chloride and frozen/thawed once a day between ± 20 °C. The weight was taken before and after the freezing, after 56, 112 and 300 cycles. Ignition tests at 1050 °C for 16 h were carried out to obtain the hydration of concrete that was crushed into maximum 5 mm pieces and dried for 1 week at 105

°C in advance [3,4]. Within the method of calculating the hydration compensation was made for the ignition loss of cement and aggregate in the calculation of hydration losses, Table 2 [7,4]. For studies of diffusivity and capillarity six specimens of each concrete were prepared. The test of drying took place at 1 or 250 days' age. The weight decrease was studied at RH = 33% in an environment free of carbon dioxide until the weight stabilized, which was not the case for 45-mm specimens. The diffusivity was estimated by comparison with a Fourier analysis at drying potentials of 20% and 50%. In this way only on average diffusivity is derived but not its dependence on the moisture content. After the tests of drying the 15-mm specimens were used for capillary suction with 2-mm water height at the rim of the discs with one of its flat sides toward the water. The capillary tests of the 15-mm discs took place at 560 days' age. Another capillary test of 45-mm discs was carried out at 750 days' age, also on specimens from diffusivity tests [4,5].

*Table 1 - Number of measurements (m = months)*

| Parameter | 1 m | 3 m | 5 m | 15 m | 90 m | 155 m |
|---|---|---|---|---|---|---|
| $f_c$ | 144 | 144 | 72 | 144 | 144 | 72 |
| $f_{ct}$ | 72 | 72 | - | 72 | 72 | 72 |
| Hydration | 144 | 144 | 72 | 144 | 144 | 72 |
| RH | 72 | 72 | - | 72 | 18 | 72 |
| Salt freezing and thawing resistance | | | | | | 72 |
| Total | 432 | 432 | 180 | 432 | 378 | 360 |

*Table 2 - Cement composition CEM I 42.5 BV/SR/LA (%,Blaine in m$^2$/kg)*

| CaO | $SiO_2$ | $Al_2O_3$ | $Fe_2O_3$ | MgO | $K_2O$ | $Na_2O$ | $SO_3$ | $CO_2$ | $C_2S$ | $C_3S$ | $C_3A$ | $C_4AF$ | Blaine |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| 64.6 | 21.8 | 3.34 | 4.39 | 0.84 | 0.62 | 0.07 | 2.23 | 0.14 | 22.5 | 53.0 | 1.4 | 13.4 | 325 |

### 2.3 Materials

Table 2 shows the chemical composition of the low-alkali cement CEM I 42.5 BV/SR/LA that was used [4]. Eight types of concrete were studied with aggregate consisting of crushed quartzite sandstone 8-12 mm (compressive strength: 333 MPa, splitting tensile strength: 15 MPa, Young's modulus: 60 GPa [8] and ignition losses: 0.25% [9]) and natural gravel 0-8 mm (granite, ignition losses: 0.85% [9]). The silica fume was granulated (ignition losses: 2.25% [9], specific surface: 17.5 m$^2$/g). The superplasticiser (naphthalene sulphonate) was added 30 s after other materials during the mixing (mixing time: 240 s). Table 3 shows the mix composition (kg/m$^3$ dry material), the properties in fresh state and the strength (MPa) [4]. The aggregate content by weight varied between 0.70 and 0.75 and this variation had very little influence on the properties. Silica fume was treated as replacement since one purpose of the study was to set the efficiency of it. Absorption of material, i.e. 0.05% for quartzite and 0.5% for gravel, was not taken into account.

## 3. RESULTS

### 3.1 Strength

Figures 1 to 3 show strength with sealed, air and water curing. Figure 4 shows strength of all the cores studied, i.e. on average strength of three sealed specimens, three air-cured specimens and three water-cured specimens. Figure 5 shows strength with interlayer, $f_c$, during testing versus strength without interlayer, $f_{cc}$. The following influence of interlayers on strength compared to strength without interlayer was obtained (MPa):

$$f_c = 0.94 \cdot f_{cc} \quad (2)$$

*Table 3 - Composition (kg/m$^3$ dry material), ignitions losses and properties of the concrete*

| w/c (Portland cement) | 0.22 | 0.25 | 0.24 | 0.33 | 0.36 | 0.47 | 0.48 | 0.58 | Ignition loss (%) |
|---|---|---|---|---|---|---|---|---|---|
| Quartzite 8-12 mm | 1360 | 1310 | 1310 | 1210 | 1160 | 1150 | 1150 | 1150 | 0.27 |
| Gravel 0-8 mm | 525 | 630 | 549 | 723 | 730 | 846 | 825 | 812 | 1.14 |
| Cement, low-alkaline | 484 | 456 | 476 | 400 | 389 | 303 | 298 | 299 | 0.56 |
| Silica fume | 48 | - | 48 | - | 39 | - | 30 | - | 2.60 |
| Superplasticiser | 13.3 | 8.84 | 7.78 | 3.35 | 3.07 | 3.01 | 2.13 | - | |
| Water | 113 | 114 | 117 | 137 | 142 | 141 | 147 | 159 | |
| Density | 2530 | 2510 | 2500 | 2470 | 2460 | 2440 | 2450 | 2420 | |
| Aggregate content | 0.71 | 0.74 | 0.75 | 0.75 | 0.73 | 0.71 | 0.73 | 0.70 | |
| Air content (%) | 0.95 | 1.5 | 0.8 | 1.4 | 1.1 | 1.1 | 0.95 | 0.75 | |
| Workability (vebe) | 29 | 34 | 13 | 25 | 12 | 9 | 12 | 15 | |
| Strength – 1 month (MPa) | 114 | 93 | 112 | 77 | 93 | 58 | 65 | 38 | |
| - 3 months | 128 | 104 | 128 | 91 | 100 | 70 | 76 | 45 | |
| - 15 months | 142 | 121 | 139 | 105 | 104 | 78 | 81 | 51 | |
| - 90 months | 139 | 121 | 131 | 106 | 106 | 74 | 79 | 49 | |
| - 155 months | 141 | 129 | 142 | 114 | 117 | 80 | 94 | 56 | |

The moisture conditions in the core also had an influence on strength. Figure 6 shows strength at 5 months' age with 1 month of intensive drying at 55°C, $f_{cd}$, and strength with the above-mentioned drying period followed by 1 month of water curing, $f_{cw}$, for 1 month versus sealed strength, $f_{cs}$. The following eqs were obtained:

$f_{cd} = 1.19\cdot f_{cs}$ (3)

$f_{cw} = 0.87\cdot f_{cs}$ (4)

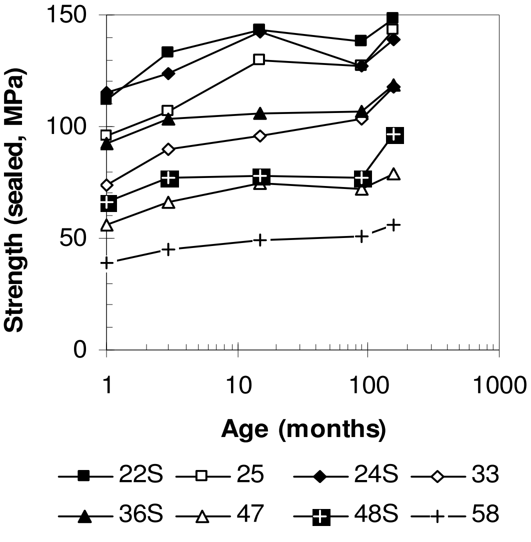


*Figure 1 – Compressive strength with sealed curing vs age. S = 10% silica fume; 22 = w/c (%).*

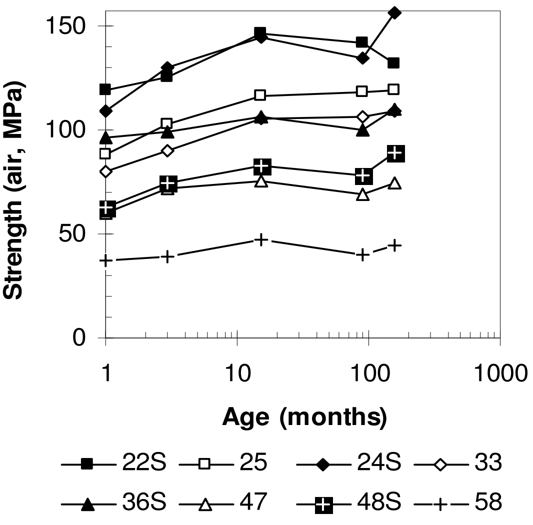


*Figure 2 – Compressive strength with air curing vs age. S = 10% silica fume; 22 = w/c (%).*

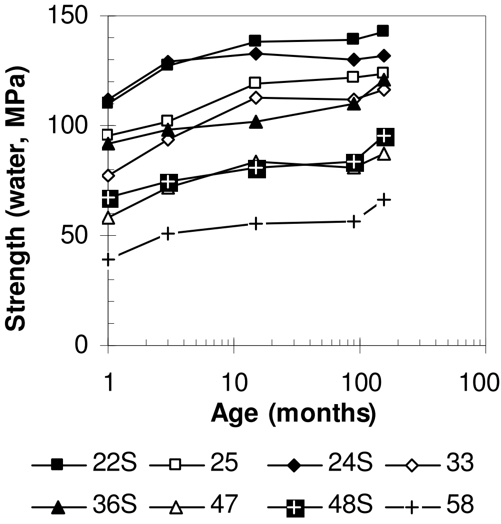


*Figure 3 – Compressive strength with water curing vs age. S = 10% silica fume; 22 = w/c (%).*

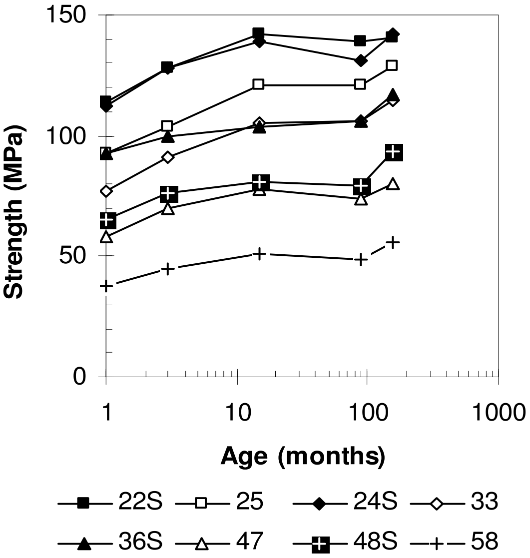


*Figure 4 – Compressive strength independent curing vs age. S = 10% silica fume; 22 = w/c (%).*

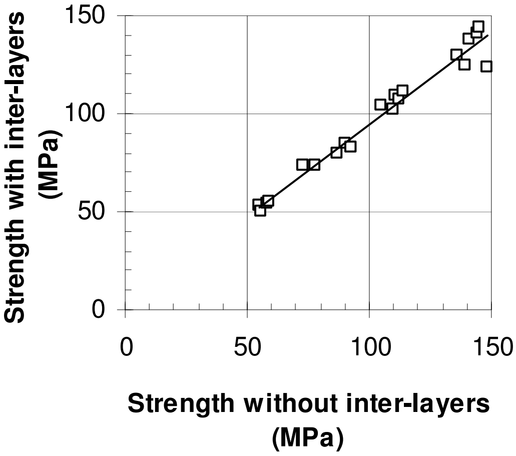


*Figure 5 – Strength with interlayers vs strength without interlayers.*

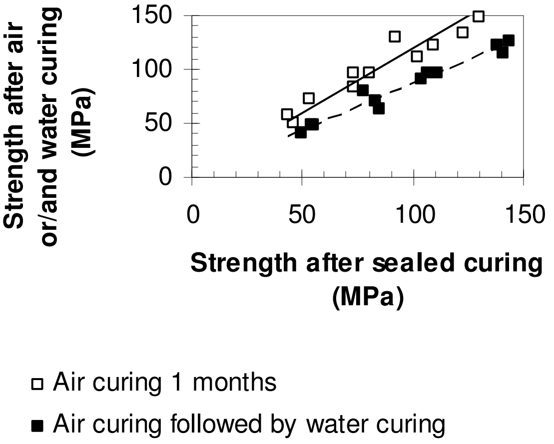


*Figure 6 – Effect of curing type on strength.*

Figures 7 to 10 show strength versus w/c for sealed, air, water and all kinds of curing (MPa):

$f_c(w/c)=A\cdot(w/c)+B$ (5)

$f_c(w/c)$ denotes the compressive strength (MPa) and A, B constants given in Table 4. Curing condition had a minor effect on long-term strength. However, the influence of silica fume was significant. At 1 months age concrete with 10% silica fume obtained about 15 MPa higher strength than concrete without silica fume (w/c held constant). On average the development of strength increase rate was twice as large in concrete without silica fume as in concrete with 10% silica fume. Figures 11 to 13 show splitting tensile strength with sealed, air and water curing. Figure 14 shows splitting tensile independent of curing.

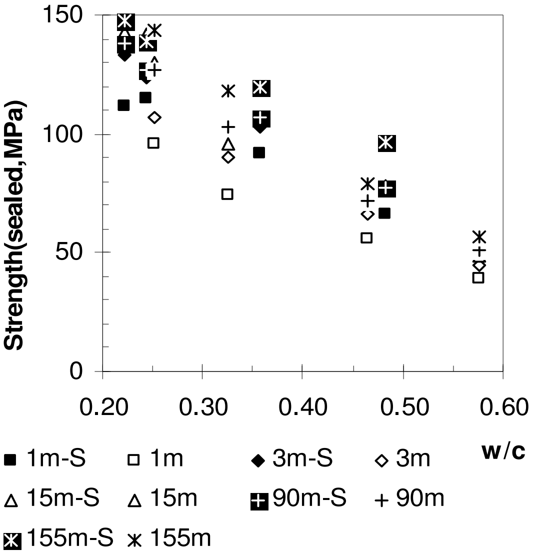


*Figure 7 – Strength with sealed curing vs w/c. S = 10% silica fume; 22 = w/c (%).*

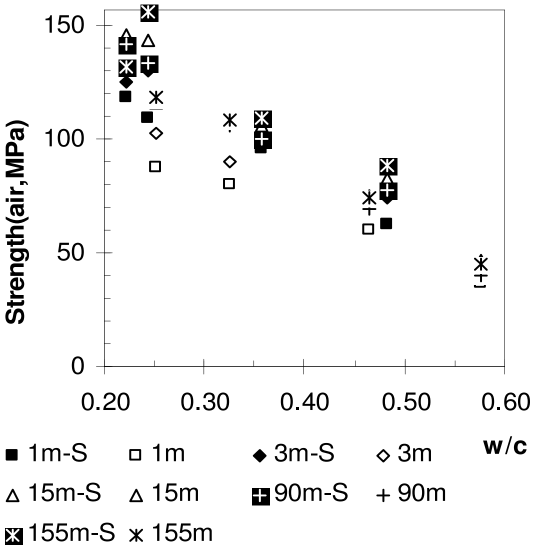


*Figure 8 – Strength with air curing vs w/c. S = 10% silica fume; 22 = w/c (%).*

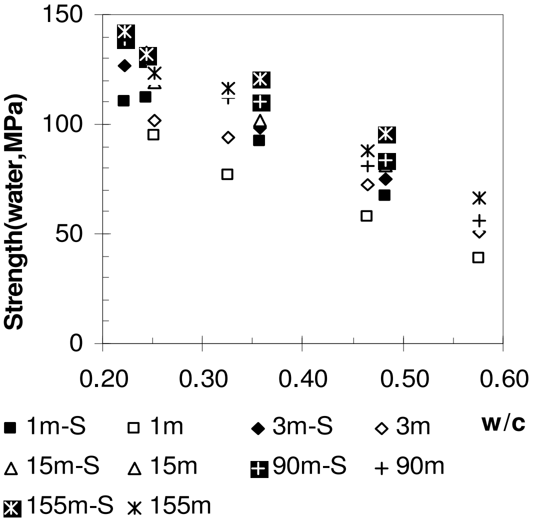


*Figure 9 – Strength, water curing, vs w/c.*

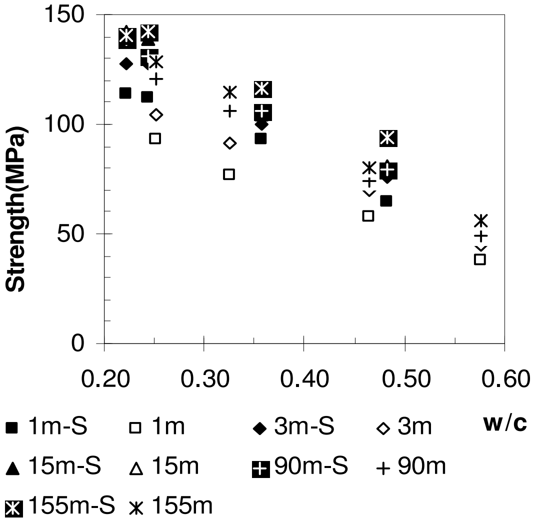


*Figure 10 – Strength independent curing type.*

[redacted]

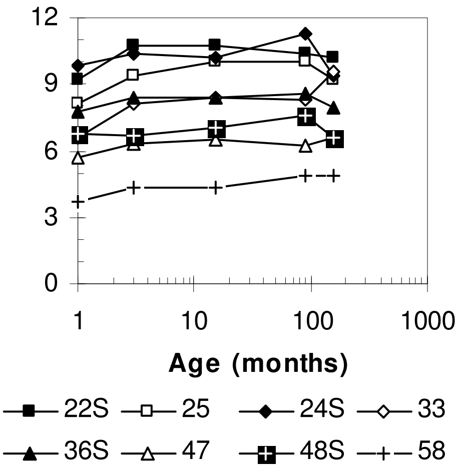


*Figure 11 – Splitting strength (sealed curing) vs age. S = 10% silica f.; 22 = w/c (%).*

[redacted]

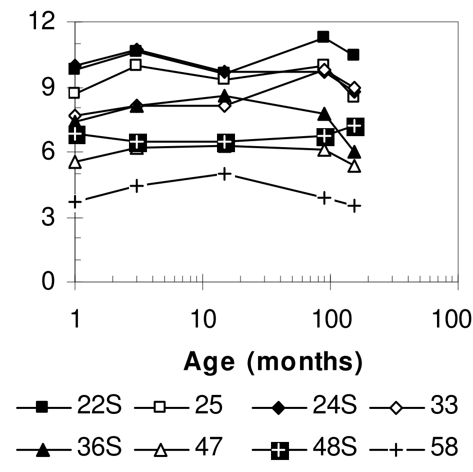


*Figure 12 – Splitting strength with air curing vs age. S = 10% silica fume; 22 = w/c (%).*

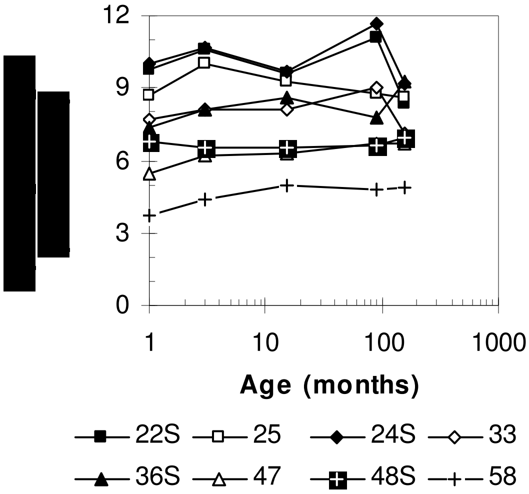


*Figure 13 – Splitting strength with water curing vs age. S = 10% silica fume; 22 = w/c (%).*

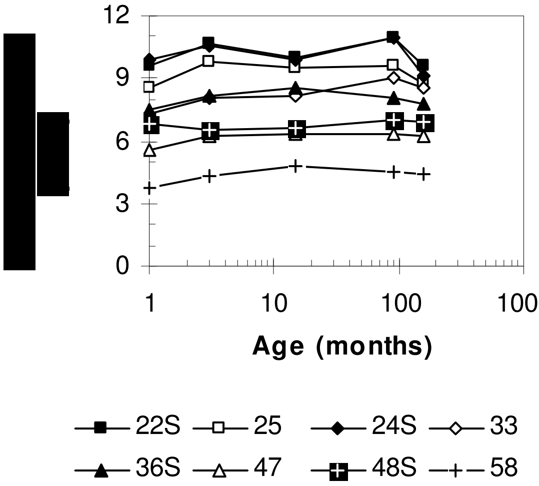


*Figure 14 – Splitting strength independent of curing vs age. S = 10% silica fume; 22 = w/c (%).*

*Table 4 - Constants of equations (4) and (5)(9 specimens at each age and combination) (MPa)*

| Age (months) | Silica fume | A | B | R² | C | D | R² |
|---|---|---|---|---|---|---|---|
| 1 | 10% | -188.2 | 157.4 | 0.99 | 0.195 | 0.0658 | 0.98 |
| 3 | 10% | -207.9 | 175. 9 | 0.99 | 0.2056 | 0.0635 | 0.95 |
| 15 | 10% | -241.9 | 195.5 | 0.99 | 0.232 | 0.0523 | 0.98 |
| 90 | 10% | -225.0 | 187.2 | 1.00 | - | - | |
| 155 | 10% | -189.2 | 185.0 | 0.99 | 0.275 | 0.0487 | 0.97 |
| 1 | - | -163.5 | 132.7 | 0.99 | 0.175 | 0.0878 | 0.92 |
| 3 | - | -177.2 | 149.2 | 0.99 | 0.197 | 0.0926 | 0.92 |
| 15 | - | -212.0 | 174.6 | 1.00 | 0.218 | 0.0981 | 0.89 |
| 90 | - | -222.6 | 177.6 | 1.00 | | | |
| 155 | - | -226.6 | 186.5 | 1.00 | 0.231 | 0.0928 | 0.97 |

### 3.2 Hydration

Figures 15 to 17 show hydration (non-evaporable water to cement, $w_n/c$) with sealed, air and water curing. Figure 18 shows hydration of all the cores. The following equation was found for the hydration, $w_n/c$:

$$w_n/c = C\cdot(w/c)+D \qquad (6)$$

C, D denotes constants given in Table 4. The maximum degree of hydration, $\alpha_{max}$, of concrete with w/c < 0.39 is linearly dependent on w/c [4,10]:

$$\alpha_{max} = w/(0.30\cdot c) \qquad (7)$$

The degree of hydration, α, can be expressed with the following equation:

$$\alpha = w_n/(0.25\cdot c) \qquad (8)$$

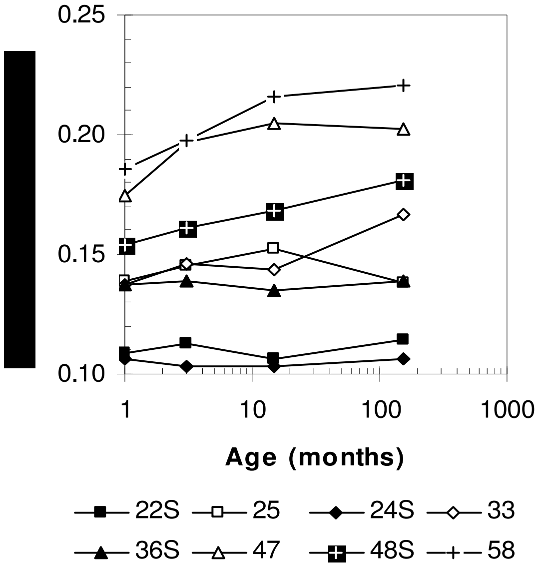


*Figure 15 – Hydration, $w_n/c$, vs age (sealed).*

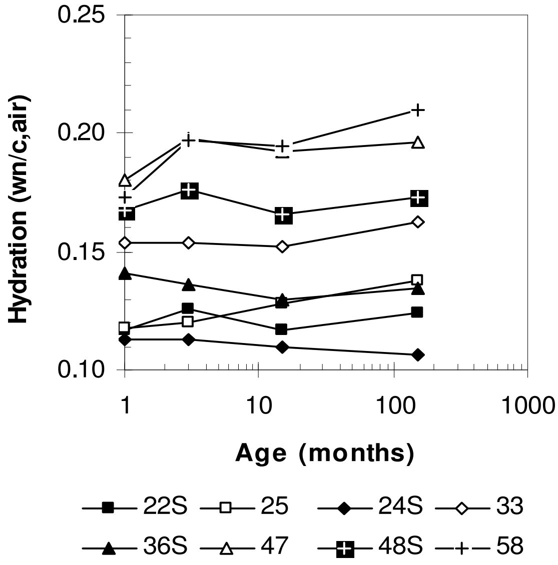


*Figure 16 – Hydration, $w_n/c$, vs age (air curing).*

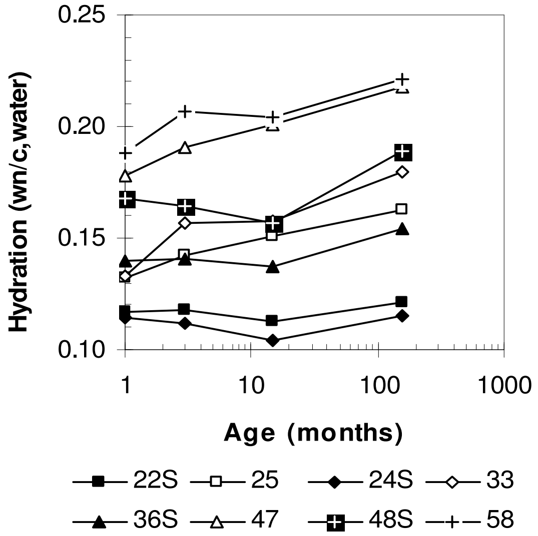


*Figure 17 – Hydration, $w_n/c$, vs age (water).*

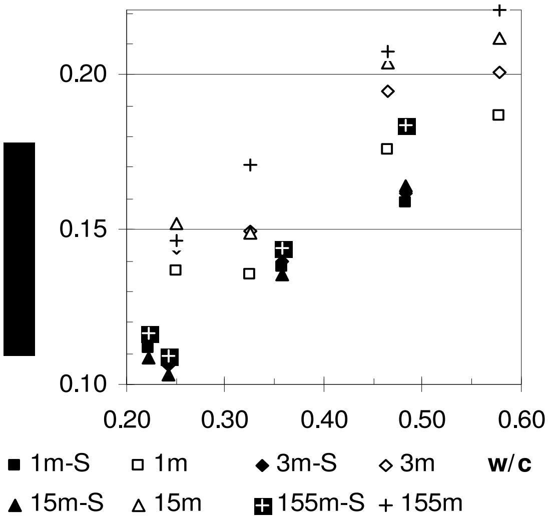


*Figure 18 – Hydration vs w/c (indep. curing).*

Dividing eqs (10) and (9) gives the maximum value of the relative hydration:

$(w_n/w)_{max}$=0.64 $\{0 < w/c < 0.39\}$ (9)

$(w_n/w)_{max}$=0.25·c/w $\{w/c > 0.39\}$ (10)

Figure 19 shows relative hydration (non-evaporable water to mixing water, $w_n/w_0$) independent of curing. Figure 19 gives an equation of the development of the relative hydration with sealed curing versus w/c:

$w_n/w_{0S}=(0.012\cdot\ln(t)+0.219)\cdot(w/c)^{(0.0312\cdot\ln(t)-0.522)}$ {10% silica fume} (11)

$w_n/w_0=(0.0116\cdot\ln(t)+0.243)(w/c)^{(0.0149\cdot\ln(t)-0.568)}$ {no silica fume} (12)

### 3.3 Relative humidity, RH, and salt-frost resistance

RH of the concrete explained the hydration [10]. Figures 20 to 22 show RH with sealed, air and water curing. From Figure 23, which shows RH of sealed concrete versus w/c, the following eqs were obtained:

$$RH_S(t,w/c)= (0.62\cdot((1-0.1\cdot\ln(t))\cdot(w/c)+1)\ \{1 < t < 155 \text{ months}; 0.25 < w/c < 0.55\} \quad (13)$$

$$RH(t,w/c)= 0.44\cdot(1+0.038\cdot\ln(t))\cdot(w/c)-0.032\cdot\ln(t)+0.75\ \{1<t<155 \text{ m}; 0.25 < w/c < 0.55\} \quad (14)$$

ln(t) denotes the natural logarithm of age, t, in months (m). After 15 months' age RH remained constant with sealed curing, Figure 20, except at high w/c and no silica fume in the concrete (decreasing RH). No air-entrainment was used in the concrete. The salt-frost scaling was larger in concrete without silica fume than with silica fume, due to self-desiccation and the ability of silica fume to prevent chloride ingress.

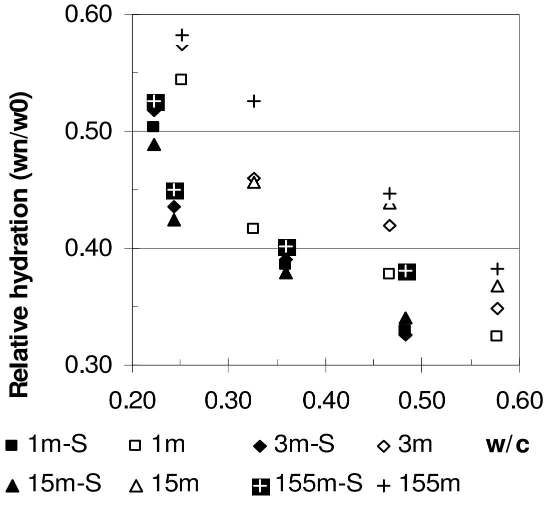


*Figure 19 – Rel. hydration vs w/c (indep. curing).*

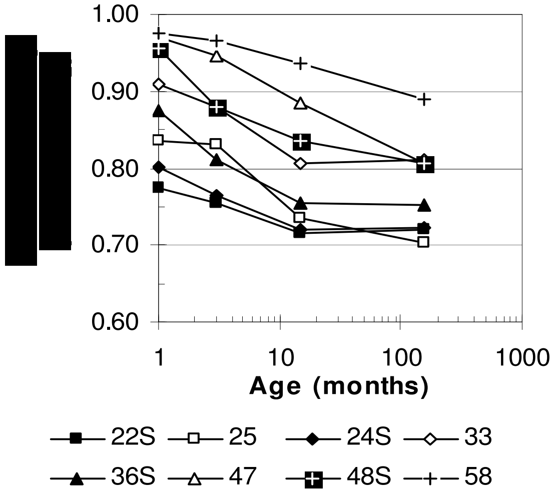


*Figure 20 - RH with sealed curing vs age.*

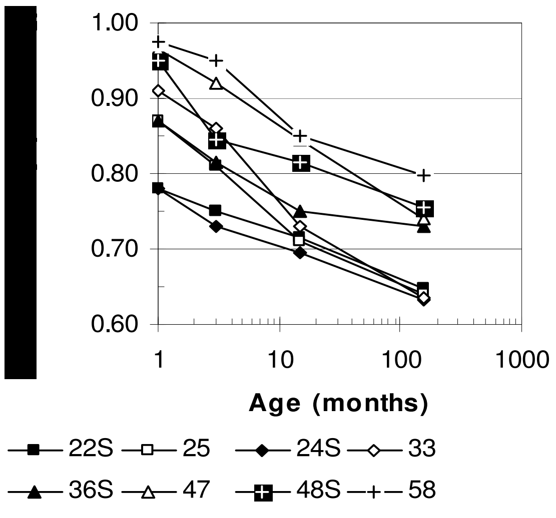


*Figure 21 - RH with air curing vs age (months). S = 10% silica fume; 22 = w/c (%).*

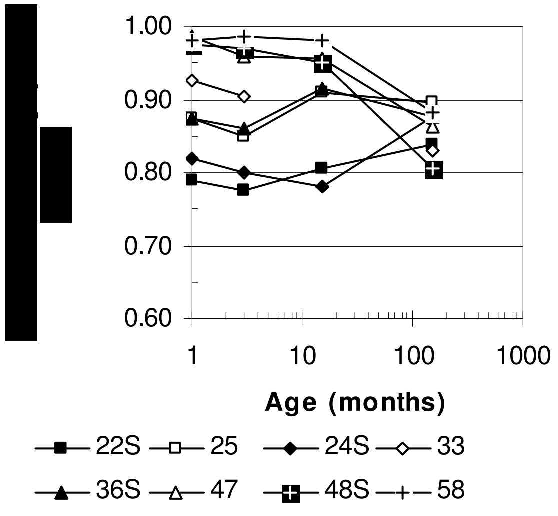


*Figure 22 - RH with water curing vs age (months). S = 10% silica fume; 22 = w/c (%).*

# 4. ANALYSIS AND DISCUSSION

## 4.1 General

The coefficient of variation was 6.3% for compressive strength and 7.2% for splitting tensile strength which is very low for strength test on drilled-out concrete, Tables 5-8. For tests of hydration a 4.4%-variation coefficient was obtained and for relative humidity 2.2%, which is an even higher accuracy. It seems that there is not one single efficiency factor for silica fume but several. However, in this study the number of efficiency factors has been limited to four, of which the one for the purpose has to be chosen. Only one kind of efficiency factor exists calculated in the same way but with different values dependent on the application, compressive or splitting tensile strength, hydration or relative humidity. The pozzolanic effect of silica fume, k, was defined according to the following equation:

$$(w/c)_{eff} = w/(c+k\cdot s) \qquad (15)$$

k denotes the efficiency factor of silica fume, s denotes 10% silica fume and $(w/c)_{eff}$ denotes w/c in concrete without silica fume in order to obtain identical properties (compressive and splitting tensile strength, hydration or RH) to concrete with 10% silica fume, with w/c held constant. About 60% of the amount of silica fume (10% calculated on the basis of the cement content) was available for the reaction with Portland cement to come to an end given a degree of hydration, $\alpha=1$. After 90 months $w_n/c$ in concrete without silica fume varied between 0.16 and 0.22, i.e. less than 0.25. $w_n/c \approx 0.25$ was required for the reaction between water and cement to come to an end [10]. At 155 months' age $w_n/c < 0.15$ for concrete with $w/c < 0.39$ was observed, which theoretically implied that a sufficient amount of silica fume still remained in concrete with $w/c < 0.39$ for the long-term interaction between Portland cement and silica fume to continue. For concrete with $w/c \geq 0.39$ the pozzolanic reaction between Portland cement and silica fume took place mainly before 1 month's age. For concrete with $w/c < 0.39$ the pozzolanic interaction was observed until 15 months' age.

*Table 5 - Coefficient of variation for compressive strength (sealed curing).*

| Mix | 1 m | 3 m | 15 m | 90 m | 155 m |
|---|---|---|---|---|---|
| 1 | 0.092 | 0.032 | 0.029 | 0.035 | 0.025 |
| 2 | 0.040 | 0.046 | 0.041 | 0.042 | 0.061 |
| 3 | 0.021 | 0.046 | 0.042 | 0.050 | 0.063 |
| 4 | 0.036 | 0.037 | 0.041 | 0.096 | 0.177 |
| 5 | 0.119 | 0.042 | 0.030 | 0.029 | 0.043 |
| 6 | 0.047 | 0.073 | 0.052 | 0.029 | 0.000 |
| 7 | 0.090 | 0.057 | 0.100 | 0.115 | 0.098 |
| 8 | 0.098 | 0.106 | 0.098 | 0.117 | 0.124 |
| av. | 0.069 | 0.055 | 0.054 | 0.065 | 0.074 |

*Table 6 - Coefficient of variation for splitting tensile strength (sealed curing).*

| Mix | 1 m | 3 m | 15 m | 90 m | 155 m |
|---|---|---|---|---|---|
| 1 | 0.060 | 0.068 | 0.053 | 0.177 | 0.106 |
| 2 | 0.057 | 0.033 | 0.047 | 0.067 | 0.160 |
| 3 | 0.012 | 0.034 | 0.049 | 0.041 | 0.168 |
| 4 | 0.084 | 0.037 | 0.036 | 0.018 | 0.131 |
| 5 | 0.030 | 0.018 | 0.036 | 0.040 | 0.146 |
| 6 | 0.035 | 0.033 | 0.081 | 0.145 | 0.088 |
| 7 | 0.043 | 0.046 | 0.041 | 0.112 | 0.079 |
| 8 | 0.016 | 0.024 | 0.098 | 0.109 | 0.210 |
| av. | 0.042 | 0.037 | 0.055 | 0.089 | 0.136 |

*Table 7 - Coefficient of variation for hydration (sealed curing).*

| Mix | 1 m | 3 m | 15 m | 90 m | 155 m |
|---|---|---|---|---|---|
| 1 | 0.032 | 0.037 | 0.036 | 0.041 | 0.029 |
| 2 | 0.020 | 0.035 | 0.072 | 0.027 | 0.045 |
| 3 | 0.073 | 0.052 | 0.118 | 0.024 | 0.049 |
| 4 | 0.044 | 0.045 | 0.024 | 0.038 | 0.054 |
| 5 | 0.068 | 0.051 | 0.030 | 0.032 | 0.049 |
| 6 | 0.028 | 0.038 | 0.030 | 0.051 | 0.021 |
| 7 | 0.035 | 0.041 | 0.065 | 0.022 | 0.012 |
| 8 | 0.052 | 0.054 | 0.066 | 0.046 | 0.097 |
| av. | 0.044 | 0.042 | 0.055 | 0.035 | 0.045 |

*Table 8 - Coefficient of variation for internal relative humidity (sealed curing).*

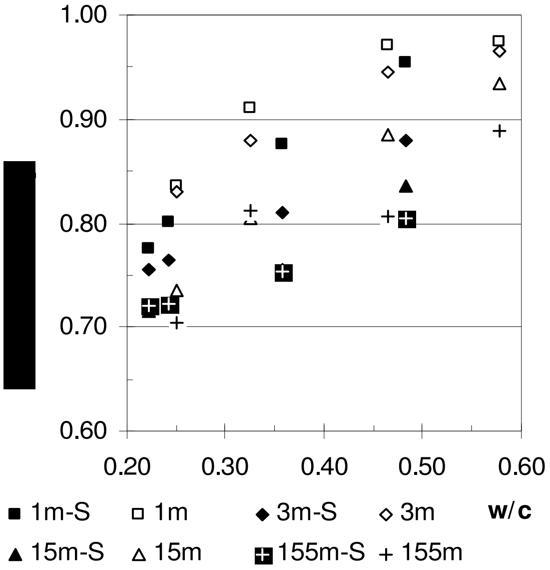
| Mix | 1 m | 3 m | 15 m | 90 m | 155 m |
|---|---|---|---|---|---|
| 1 | 0.021 | 0.017 | 0.020 | 0.068 | 0.013 |
| 2 | 0.004 | 0.012 | 0.037 | - | 0.002 |
| 3 | 0.018 | 0.02 | 0.038 | 0.046 | 0.014 |
| 4 | 0.023 | 0.016 | 0.046 | - | 0.013 |
| 5 | 0.021 | 0.019 | 0.057 | - | 0.000 |
| 6 | 0.008 | 0.013 | 0.026 | - | 0.011 |
| 7 | 0.008 | 0.022 | 0.043 | 0.006 | 0.003 |
| 8 | 0.003 | 0.007 | 0.020 | - | 0.008 |
| av. | 0.013 | 0.016 | 0.036 | 0.040 | 0.008 |

## 4.2 Relative humidity, RH

RH limited the hydration of Portland cement in concrete (RH > 72%) [11]. Calcium hydroxides from the hydration were required for the pozzolanic reaction to take place [11,12]. When RH decreased, the pozzolanic reaction also decreased and finally ceased [13-26]. Self-desiccation was also of great importance and more pronounced in concrete with silica fume than in concrete without silica fume [3]. Self-desiccation was caused by depression in the pore water, expressed more in concrete with silica fume. Self-desiccation caused autogenous shrinkage which in turn caused tensile stresses in the cement paste and compression in the aggregate of the concrete. Self-desiccation partly explained the development of mechanical properties of the concrete such as compressive and splitting tensile strength. Due to the compression of the aggregate in low-w/c concrete (increasing due to the ongoing autogenous shrinkage) the splitting tensile strength capacity may decrease in concrete with silica fume with low w/c. When the autogenous shrinkage exceeded the tensile strain of the cement paste, which may occur in concrete with low w/c with silica fume, cracks occurred. To evaluate the efficiency factor related to self-desiccation, $k_{se}$, eqs (13) and (14) above were used, i.e. w/c in equation (16) was replaced by $(w/c)_{eff}$ according to equation (17). After this replacement eqs (13) and (14) were equalized and $k_{se}$ easily calculated. Figure 24 shows $k_{se}$ which was correlated to w/c and age, t (months), by the following equation:

$$k_{se}(t,w/c)=(0.71\cdot(1+0.038\cdot\ln(t))\cdot(w/c)/((1-0.1\cdot\ln(t))\cdot(w/c)+0.0516\cdot\ln(t)-0.21)-1)\cdot 10$$
$$\{1<t<155\text{ months}; 0.25<w/c<0.50\} \qquad (16)$$

At w/c = 0.43 $k_{se}$ =3.8 was observed independent of age, which shows that w/c > 0.39 was required for full hydration to take place [10]. Between 1 and 15 months' age $k_{se}$ > 3.8 was observed in concrete with w/c < 0.43. At 155 months' age and in concrete with w/c < 0.43, $k_{se}$ < 3.8 was observed since the hydration and the pozzolanic reaction ceased. The contrary was observed at w/c $\geq$ 0.43 where the hydration continued and also the pozzolanic reaction. The pozzolanic reaction in concrete with silica fume caused smaller average pore diameter in the gel and thus lower RH than in concrete without silica fume [12].



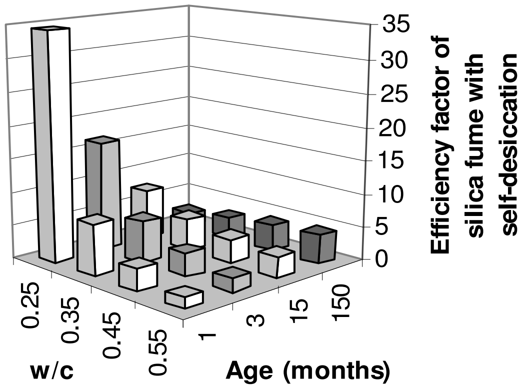


*Figure 23 - RH with sealed curing vs w/c. m = month; S= 10% silica fume.*

*Figure 24 - Efficiency factor of silica fume related to relative humidity with sealed curing.*

### 4.3 Strength

Initially the effect of silica fume on compressive strength was pronounced, especially in concrete with low w/c. However, due to self-desiccation in concrete with silica fume, especially in concrete with low w/c, the hydration ceased, thus the pozzolanic reaction and also the increase of strength, cp. eqs (5) and (6) above. The rate of long-term strength was about 50% larger in concrete without silica fume than with 10% silica fume in the concrete. All kinds of curing were studied regarding the efficiency factor related to strength, $k_{sc}$. To evaluate $k_{sc}$ eqs (17) and (18) were used, i.e. w/c in the equation valid for concrete without silica fume was replaced by $(w/c)_{eff}$ according to equation (17):

$$f_{cS}=(0.00661\cdot t^2 - 0.902\cdot t - 206)\cdot w/c+4.98\ln(t) + 167 \quad \{1 < t < 150 \text{ months}; 0.25 < w/c < 0.55\} \quad (17)$$

$$f_c=(0.00433\cdot t^2 - 0.979\cdot t - 17)\cdot w/c+9.99\ln(t) + 137 \quad \{1 < t < 150 \text{ months}; 0.25 < w/c < 0.55\} \quad (18)$$

After this replacement the eqs valid for concrete with and without silica fume were equalized and $k_{sc}$ easily estimated. Figure 25 shows $k_{sc}$ which may be described by the following equation:

$$k_{sc}(t,w/c)=10\cdot((0.00433\cdot t^2-0.979\cdot t-177)\cdot(w/c)/((0.00661\cdot t^2-0.902\cdot t-206)\cdot w/c)-5\cdot\ln(t)+30)-1)$$
$$\{1<t<155 \text{ months}; 0.25 < w/c < 0.55\} \quad (19)$$

All kinds of curing were also studied regarding the efficiency factor related to splitting tensile strength, $k_{st}$. To evaluate $k_{st}$ eqs (20) and (21) were used, i.e. w/c in the equation valid for concrete without silica fume was replaced by $(w/c)_{eff}$ according to equation (15). After this replacement the eqs valid for concrete with and without silica fume were equalized and $k_{st}$ was easily estimated.

$$f_{ctS}(t,w/c)= (0.719\cdot\ln(t)-14.5)\cdot(w/c)-0.285\cdot\ln(t)+13.5 \quad \{1<t<155 \text{ m}; 0.25 < w/c < 0.55\} \quad (20)$$

$$f_{ct}(t,w/c)= (0.197\cdot\ln(t)-15.2)\cdot(w/c)+0.0278\cdot\ln(t)+12.8 \quad \{1<t<155 \text{ m}; 0.25 < w/c < 0.55\} \quad (21)$$

Figure 26 shows $k_{st}$ which may be described by the equation:

$k_{st}=10\cdot((0.197\cdot\ln(t)-15.2)\cdot(w/c)/((0.719\cdot\ln(t)-14.5)\cdot(w/c)-0.313\cdot\ln(t)+0.7)-1)$
$\{1<t<155 \text{ months}; 0.25 < w/c < 0.55\}$ (22)

At $w/c < 0.43$ $k_{st}$ decreased with time ($k_{st} = 0$ at $w/c = 0.25$), which probably was due to the splitting tensile strength of the aggregate ($f_{sp,max} \approx 0.75\cdot 15= 12$ MPa). The relationship between compressive and tensile strength was also influenced by the pozzolanic interaction between Portland cement and silica fume. As mentioned above, the pozzolanic effect of silica fume caused micro-cracking in the cement paste and thus lower tensile strength in comparison to the compressive strength. Figure 27 shows tensile strength versus compressive strength for about 1000 specimens. The relationship between splitting and compressive strength decreased more in concrete with 10% silica fume (both with higher strength and with age) compared to the corresponding property of concrete without silica fume. The following eqs were obtained :

$f_{ctS}=f_{cS}\cdot 0.0866\cdot(1-0.0468\cdot\ln(t))$ $\{60< f_c < 150 \text{ MPa}; 1 < t < 90 \text{ months}\}$ (23)

$f_{ct}=f_c\cdot 0.0938\cdot(1-0.0447\cdot\ln(t))$ $\{30< f_c < 120 \text{ MPa}; 1< t < 155 \text{ months}\}$ (24)

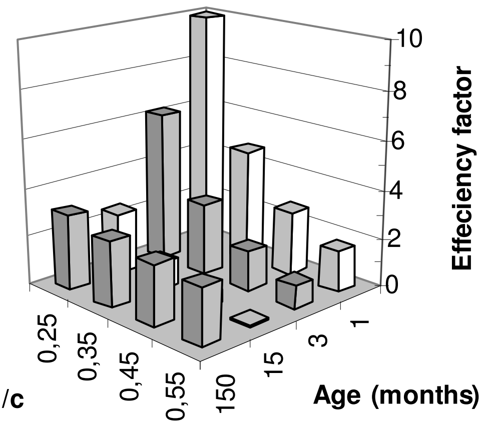


*Figure 25 - Efficiency factor of silica fume related to strength with all kinds of curing.*

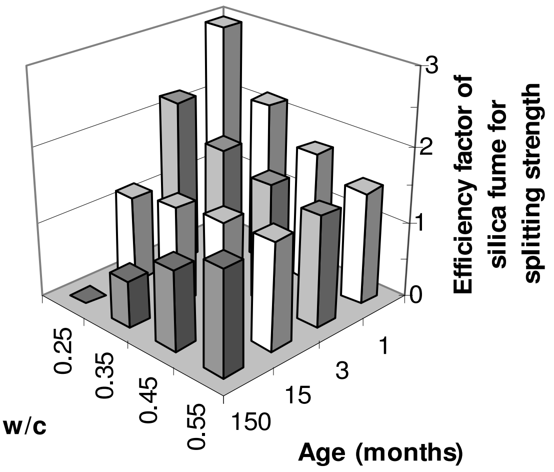


*Figure 26 - Efficiency factor of silica fume related to splitting tensile strength indep. curing.*

### 4.4 Hydration

Hydration studies were limited to concrete without and with 10% silica fume only [27-29]. Sealed curing was studied for the hydration efficiency factor, $k_{wn}$. Eqs (25) and (26) were used to evaluate $k_{wn}$, i.e. w/c in equation (26) without silica fume was replaced by $(w/c)_{eff}$ according to equation (15), i.e. the same method was used to calculate the efficiency factor of silica fume for hydration as for strength.

$w_n/c_S=(0.0162\cdot\ln(t)+0.191)(w/c)-0.00365\cdot\ln(t)+0.0657$ $\{1<t<155 \text{ months}; 0.25<w/c< 0.55\}$ (25)

$w_n/c=(0.0108\cdot\ln(t)+0.182)(w/c)+0.000966\cdot\ln(t)+0.0907$ $\{1<t<155 \text{ months}; 0.25<w/c<0.55\}$ (26)

After this replacement the eqs for concrete with and without silica fume were equalized and $k_{swn}$ was easily estimated. Figure 28 shows $k_{wn}$, which may be described by the following equation:

$$k_{wn}(t,w/c) = -10\cdot((0.0108\cdot\ln(t)+0.182)\cdot(w/c)/((0.0162\cdot\ln(t)+0.191)\cdot(w/c)-0.00462\cdot\ln(t)-0.025)-1)$$
$$\{1 < t < 150 \text{ months}; 0.25<w/c<0.55\} \qquad (27)$$

The interaction of Portland cement and silica fume clearly affected the relationship between hydration and strength, Figure 29. In concrete with 10% silica fume the maximum strength was obtained at $w_n/w \approx 0.52$ and at $w_n/w \approx 0.61$ in concrete without silica fume. No sign of decreasing hydration was observed.

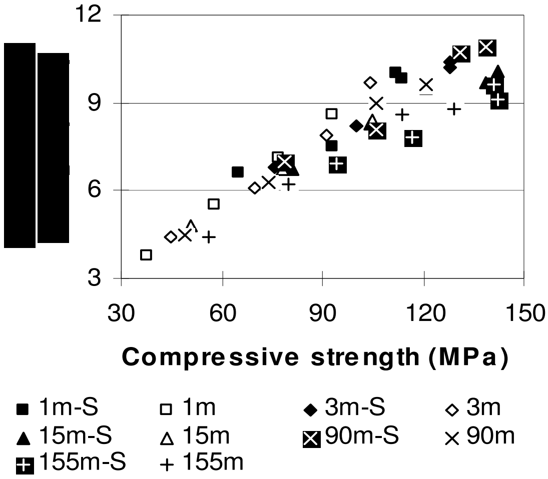


*Figure 27 – Split tensile strength vs compressive.*

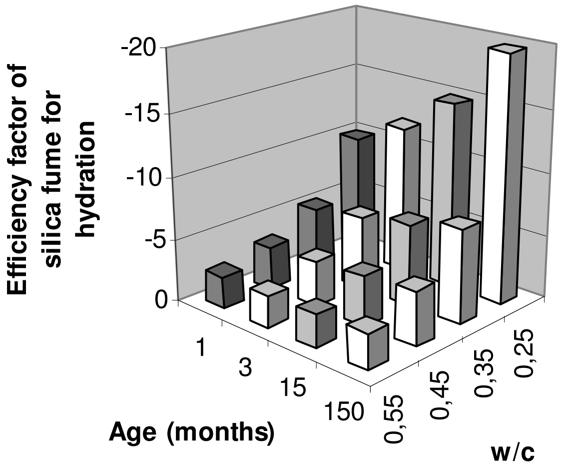


*Figure 28 –Efficiency factor of silica fume on hydration.*

### 4.6 Salt and freezing resistance - effect of silica fume on RH, strength and hydration

Figure 30 shows the effect of silica fume on the salt freezing and thawing resistance of concrete without air entrainment. Ten percent silica fume lowered the salt freezing and thawing frost scaling substantially. At 112 cycles the effect of silica fume on salt freezing and thawing scaling remains identical to that of 56 cycles. At 56 frost cycles salt-frost scaling < 0.5 kg/m² is normally acceptable. Concrete with 10% silica fume but without air-entrainment thus fulfills the requirement at w/c < 0.55. Concrete without silica fume required w/c < 0.35 to fulfill the requirement. The ability of silica fume to prevent chloride ingress probably explains the great salt freezing and thawing resistance of silica fume concrete [30]. If chlorides may not enter the concrete no decrease of the freezing point of the water will take place and water will then not enter the concrete. Behind the low chloride ingress is probably the early self-desiccation in silica fume concrete. Chlorides may not be transported in air-voids or vacuum created due to self-desiccation [31]. Figures 24, 31 and 32 show early age effect of silica fume on self-desiccation. However, after a long time of water curing the effect of silica fume on RH was low, Figure 33. Figure 34 shows that no significant relationship existed between the salt frost scaling and RH in concrete. Figures 35 and 36 show calculations with eqs (17) - (18) on compressive strength of concrete. The result of Figure 25 was confirmed, i.e. the effect of silica fume decreased with age in order to stabilize at 150 months' age, i.e. at about 2. Figures 37 and 38 show a substantial effect of silica fume on the splitting tensile strength estimated with eqs (20) and (21). In Figures 39 and 40 the splitting tensile strength is shown versus compressive strength given by eqs (23) and (24). From Figures 41 and 42 based on estimations with eqs (25) and (26) it was concluded that hydration was decreased in silica fume concrete due to pozzolanic reaction [32].

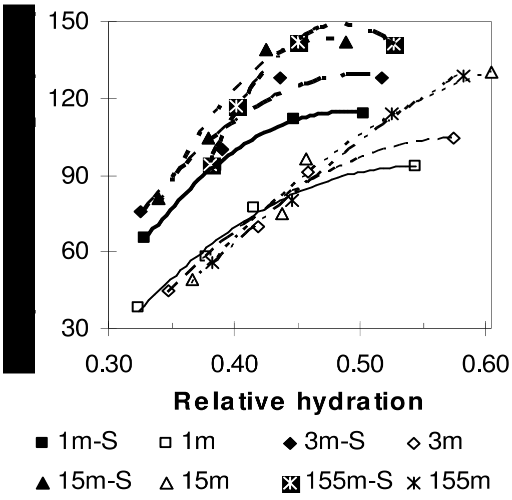

[redacted]

*Figure 29 – Compr. strength vs relative hydration.*

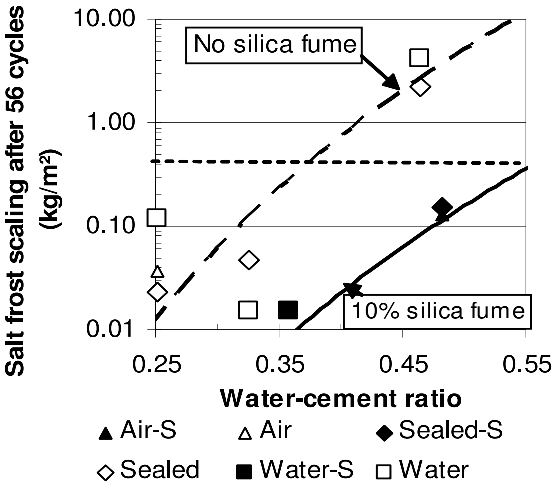


*Figure 30 – Salt frost scaling after 56 cycles.*

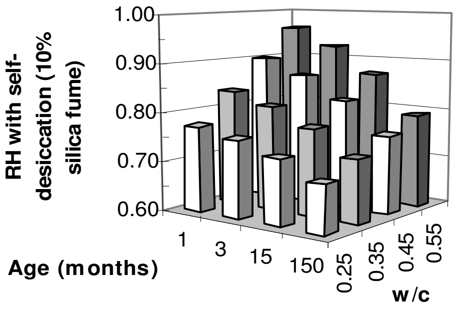


*Figure 31 - RH in concrete with self-desiccation (10% silica fume) and equation (14).*

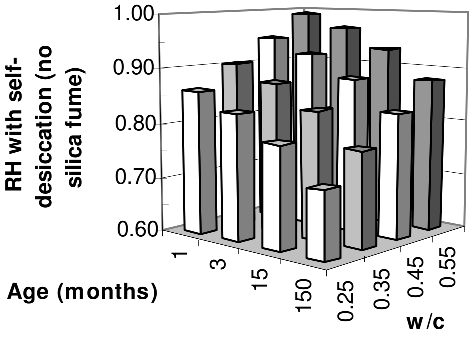


*Figure 32 - RH in concrete with self-desiccation (no silica fume) and equation (15).*

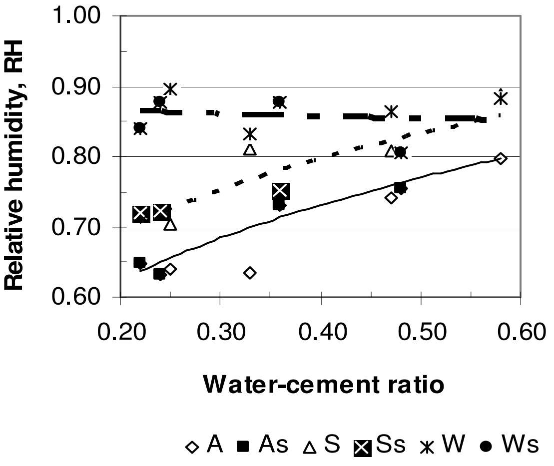


*Figure 33 – RH after 155 months. s= 10% silica fume; A= air; S= sealed; W= water.*

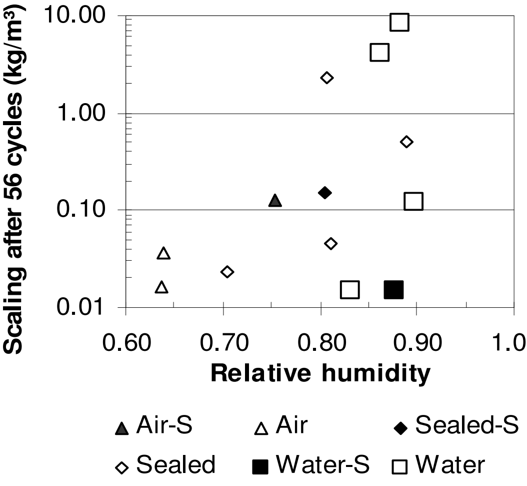


*Figure 34 – Salt scaling after 56 cycles versus RH. S= 10% silica fume.*

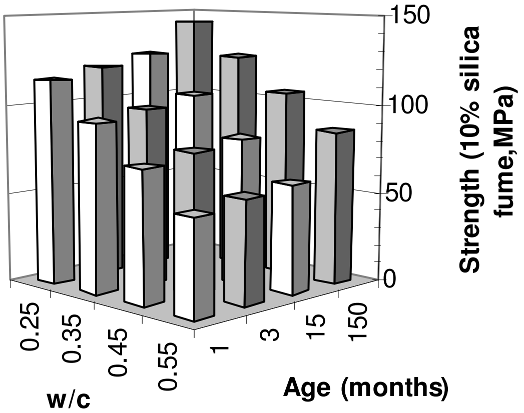


*Figure 35 – Compressive strength with eq. (19) (10% silica fume,MPa).*

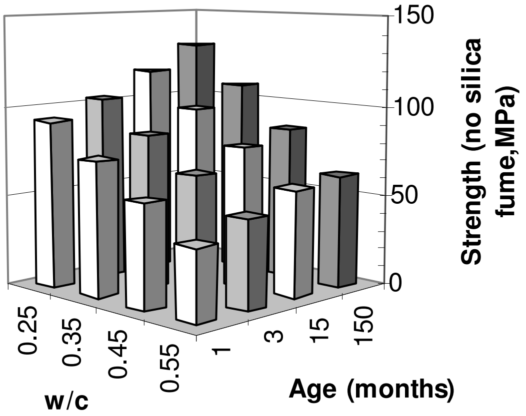


*Figure 36 – Compressive strength with eq. (20) (no silica fume,MPa).*

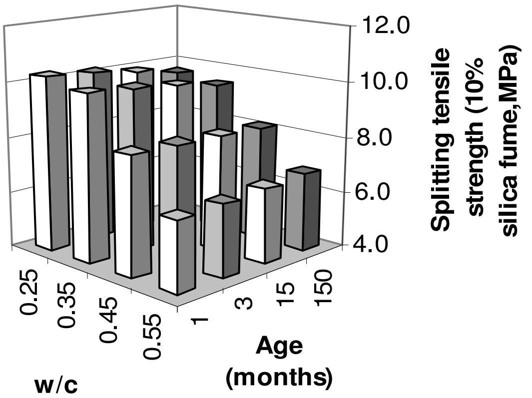


*Figure 37 – Split tensile strength with eq. (22) (10% silica fume,MPa).*

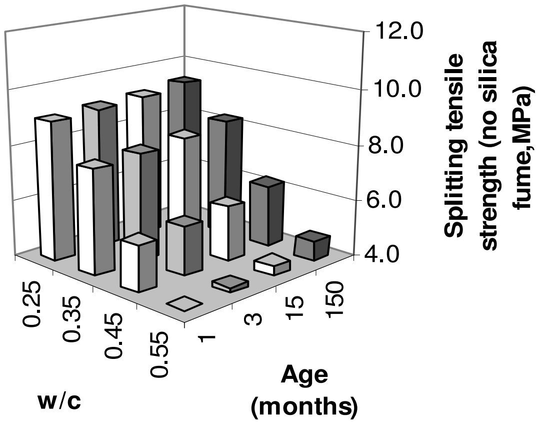


*Figure 38 – Split tensile strength with eq. (23) (no silica fume,MPa).*

### 4.7 Capillarity and diffusivity

Low RH in water-cured concrete after 155 months and low chloride migration was explained by internal stresses in the concrete due to autogenous shrinkage [4,5]. At a ≈ 15-mm surface layer expansion took place due to water contact. Internally contraction arise due to self-desiccation [2]. In between moisture transport cannot occurs due to the stress gap. Still some capillarity and diffusivity is measurable on 15-mm specimens even at low w/c, Tables 9-11 [4,5]. For 45-mm specimens with low w/c the weight during the diffusivity tests did not stabilise not even after 500 days, i.e. a stress-related interlayer was build up in the concrete with low w/c. For 15-mm specimens the following estimation was done for the resistance to water penetration, m (s/m²):

$$m = 28\cdot(0.29-(P_k)_p)\cdot 10^7 \tag{28}$$

where m denotes resistance to water penetration (s/m²) and $(P_k)_p$ the capillary porosity of paste.

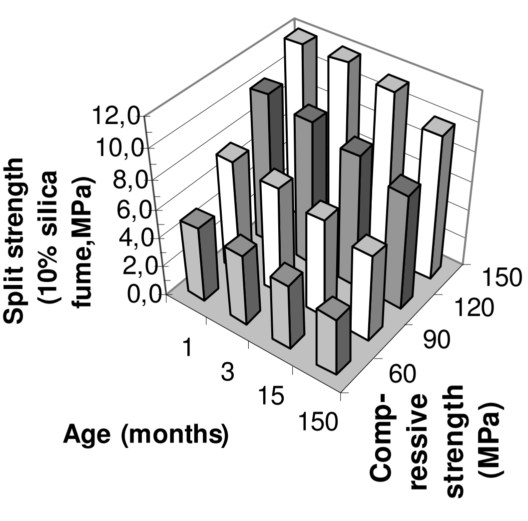


*Figure 39 – Split tensile strength with eq. (25) vs compr. strength (10% silica fume,MPa).*

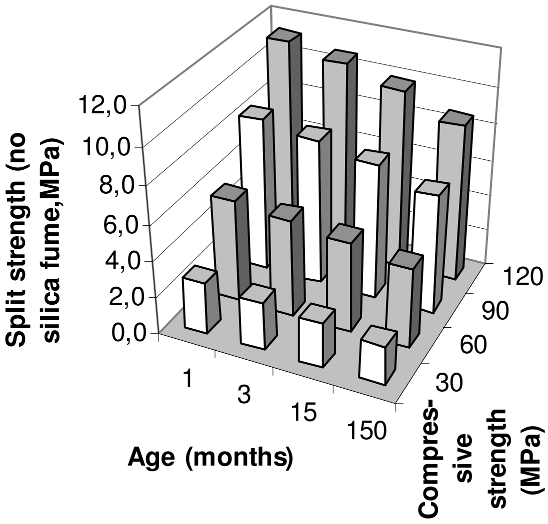


*Figure 40 – Split tensile strength with eq. (26) compr. strength (no silica fume,MPa).*

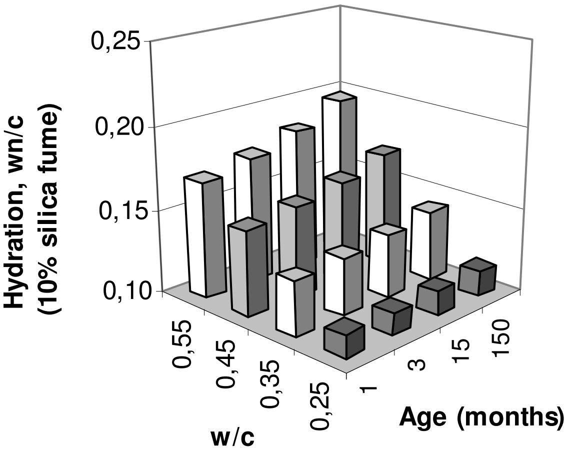


*Figure 41 - Hydration, $w_n/c$, with equation (27) (10% silica fume).*

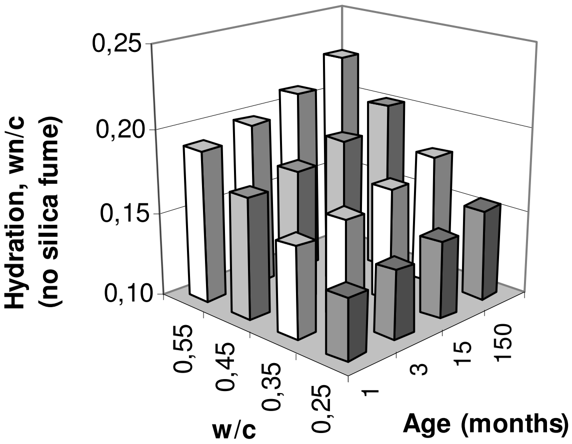


*Figure 42 - Hydration, $w_n/c$, with equation (28) ( silica fume).*

*Table 9 – Estimated capillarity data of concrete at different age [4].*

| Age | Young concrete (1 day' age) | | | | | Mature concrete (750 days' age) | | | | |
|---|---|---|---|---|---|---|---|---|---|---|
| w/c | m (s/ m²·$10^{-7}$) | k (kg/ m²√s) | α (4·$w_n$/c) | $P_k$ | $(P_k)_p$ | m (s/ m²·$10^{-7}$) | k (kg/ m²√s) | α (4·$w_n$/c) | $P_k$ | $(P_k)_p$ |
| 0.22 | 4.9 | 0.0065 | 0.45 | 0.046 | 0.086 | 0.64 | 0.0011 | 0.45 | 0.003 | 0.006 |
| 0.25 | 6.4 | 0.0065 | 0.51 | 0.052 | 0.091 | 1.9 | 0.0014 | 0.51 | 0.006 | 0.011 |
| 0.33 | 4.3 | 0.011 | 0.62 | 0.072 | 0.13 | 3.3 | 0.0030 | 0.62 | 0.017 | 0.03 |
| 0.47 | 2.4 | 0.019 | 0.77 | 0.093 | 0.21 | 8.5 | 0.0030 | 0.77 | 0.028 | 0.06 |

Notations: k = capillarity (kg/ m²√s), m = resistance to water penetration (s/ m²·), α = hydration.

The influence of age and w/c on moisture diffusivity thus was remarkable, Figures 43 and 44.

*Table 10 –Moisture diffusivity of concrete, $\delta_w$, at different age and thickness ($\cdot 10^{-12}$ m²/s)[4].*

| Thickness | 15-mm | | 45-mm |
|---|---|---|---|
| Age (days) | 1 - 250 | 250 - 500 | 250 - 750 |

| Drying potential/ w/c/ | $\delta_{w0.2}$ | $\delta_{w0.5}$ | $\delta_{w0.2}$ | $\delta_{w0.5}$ | $\delta_{w0.2}$ | $\delta_{w0.5}$ |
|---|---|---|---|---|---|---|
| 0.22 | 29 | 50 | 3.5 | 7 | 3.7 | 4 |
| 0.25 | 28 | 50 | 7.2 | 12 | 5.3 | 7 |
| 0.33 | 40 | 50 | 7.3 | 11 | 9 | 10 |
| 0.47 | 80 | 120 | 13 | 20 | 15 | 26 |

Notations: $\delta_{w0.2}$ = drying of 80% of the excess water, $\delta_{w0.5}$ = drying of 50% of the excess water.

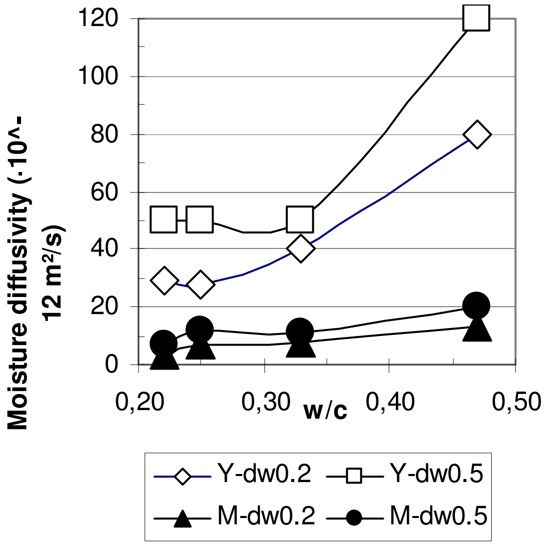


*Figure 43 - Moisture diffusivity versus w/c, 15-mm thickness. M = mature concrete, Y = young, $d_{w0.2}$ = drying of 80% of the excess water, $d_{w0.5}$ = drying of 50% of water.*

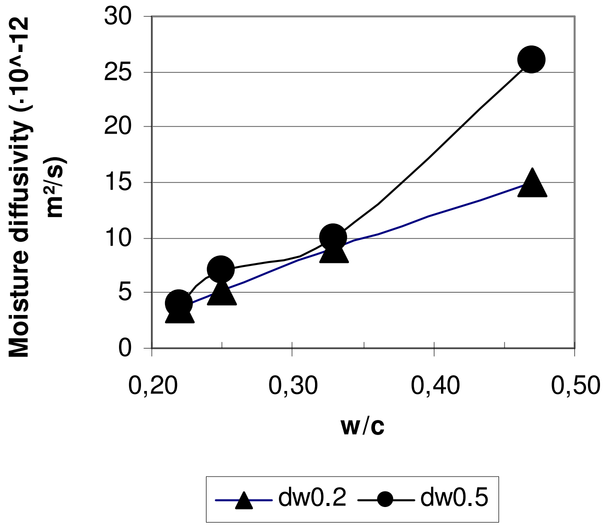


*Figure 44 - Moisture diffusivity versus w/c, 45-mm thickness, mature concrete. $d_{w0.2}$ = drying of 80% of the excess water, $d_{w0.5}$ = drying of 50% of the excess water.*

## 5. CONCLUSIONS

This article reports on a study of silica fume concrete compared to Portland cement concrete. Mechanical characteristics are measured and their evolution interpreted as a function of the role of silica fume. Completing information on effect of porosity calculated as moisture diffusivity and resistance to water penetration is given. The following conclusions were drawn [32]:

1) Silica fume had a positive effect on strength and relative humidity of concrete but not for the degree of hydration.
2) The effect was pronounced at low water-cement ratio.
3) Silica fume had a larger specific effect on compressive and splitting tensile strength and relative humidity than Portland cement did.
4) Due to low degree of hydration of cement in concrete with low water-cement ratio < 0.43, silica fume remained available for the pozzolanic interaction with the Portland cement, at least until 15 months' age.
5) In concrete at higher water-cement ratio $\geq$ 0.43 the pozzolanic effect ceased before 1 month's age.
6) After a long period of time, 155 months, the efficiency factor of silica fume on the strength became about 2.
7) At water-cement ratio < 0.43 the efficiency factor of silica fume on splitting tensile strength decreased with time (= 0 at water-cement ratio = 0.25). This phenomenon was ex-

plained by the pronounced self-desiccation which consequently stopped the hydration in low-water-cement ratio concrete.

8) The relationship between degree of hydration and strength developed differently in concrete with and without silica fume due to the pozzolanic interaction between cement and silica fume.
9) Hydration was an inconsistent parameter for mechanical properties of silica fume concrete.
10) Acceptable salt-frost scaling in concrete without air-entrainment was obtained at water-cement ratio < 0.55 combined with 10% silica fume in the mix proportions.
11) For concrete without silica fume water-cement ratio < 0.35 was required for salt-frost resistance in concrete without air-entrainment. The explanation for this observation was the ability of silica fume to prevent chloride ingress in the concrete.
12) The influence of age and water-cement ratio on resistance to water penetration, moisture diffusivity was remarkable and an explanation for low relative humidity in the concrete even after 155 months in water and also to the low penetration of chlorides.

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## REFERENCES

1. Perraton, D.; de Larrard, F., Aitcin, P. C., ”Additional data on the strength retrogression of air-cured silica fume concrete”, 2nd CANMET/ACI conf. on durability, 1994, pp. 2-15.
2. Persson, B., ”Self-desiccation and Its Importance in Concrete Technology”, *Materials and Structures,* Vol. 30, 1997, pp. 293-305.
3. Persson, B., ”Hydration and Strength of High Performance Concrete”, *Advanced Cement Based Material,* Vol. 3, 1996, pp. 107-123.
4. Persson, B., ”Hydration, Structure and Strength of High-Performance Concrete”, TVBM-1009, Lund Institute of Technology, Lund, 1992, 400 pp.
5. Persson, B., ”Drying of Concrete after Different Kinds of Curing”, *Materials and Structures*, Vol. 30, 1997, pp. 533-544.
6. ASTM E 104-85., ”Standard Practice for Maintaining Constant Relative Humidity by Means of Aqueous Solutions”, ASTM, 1985, pp. 33-34, 637.
7. Byfors, J., ”Plain concrete at early ages”, FO 3:80, The Swedish Cement and Concrete Institute, Stockholm, 1980, pp. 40-43.
8. Hassanzadeh, M., ”Fracture mechanical properties”, Report M4:05, Div. Building Materials, Lund Institute of Technology, Lund, 1992, pp. 8-13.
9. Persson, B., ”Hydration, structure and strength of High Performance Concrete”, TVBM-7011, Lund Institute of Technology, Lund, 1992, pp 38.
10. Powers, T. C. & Brownyard, T. L., ”Studies of Physical Properties of Hardened Portland Cement Paste”, Research Laboratories of PCA, *ACI Journal,* Vol. 43, 1947, pp. 984-987.
11. Norling Mjörnell, K., ”Self-desiccation in concrete”, P-94:2, Div. of Building Materials, Chalmers University of Technology, Gothenburg, 1993, pp. 21-28.
12. Zhang, M. H., Gjörv, O. E., ”Effect of Silica Fume on Cement Hydration in Low Porosity Cement Pastes”, *Cement and Concrete Research,* Vol. 21, 1991, pp. 800-808.
13. Persson, B., ”The Effect of Silica Fume on the Principal Properties of Concrete”, Symposium on Advanced Design of Concrete Structures, Gothenburg, 1997, pp. 161-168.
14. Persson, B., ”Long-term Effect of Silica Fume on the Properties of Low-Temperature-Cured Ceramic”, *Cement and Concrete Research*, Vol. 27, 1997, pp. 1667-1680.

15. Persson, B., ”Seven-year Study of the Effect of Silica Fume in Concrete”, *Advanced Cement Based Material*, Vol. 7, 1998, pp. 139-155.
16. Persson, B., ”Effect of Cement Type, Silica Fume, Water-Cement Ratio, Age and Moderate Shift in Temperature on Self-desiccation in Concrete”, *Nordic Concrete Research,* Vol. 21, 1999, pp. 97-116.
17. Persson, B., ”Pozzolanic Interaction between Portland Cement and Silica Fume in Concrete, 6th CANMET/ACI conf. on Fly Ash, Silica Fume, Slag and Natural Pozzolans, 1998, pp. 631-660.
18. Persson, B., ”Self-desiccation and Its Importance in Concrete Technology”, *Nordic Concrete Research,* Vol. 20*,* 1998, pp. 120-129.
19. Persson, B., ”Consequence of Cement Constituents, Mix Composition and Curing Conditions for Concrete Self-Desiccation”, *Materials and Structures,* Vol. 33, 2000, pp. 352-362.
20. Persson, B., ”A NORDTEST Method for Verification of Self-desiccation in Concrete”, *Cement and Concrete Research,* Vol. 31, 2001, pp. 199-203.
21. Persson, B., ”Hydration and Strength of High-Performance Concrete”, Proc. of Nordic Concrete Research Meeting, Trondheim, Norwegian Concrete Association, Oslo, Ed: J Jensen, 1990, pp. 100-102.
22. Persson, B., ”Self-Desiccation of High-Strength Concrete”, Proc. 3rd Int. Symp. of High-Strength Concrete, Norwegian Concrete Association, Oslo, Ed: Holand, I, Sellevold, E., 1993, pp. 882-889.
23. Persson, B., ”Self-Desiccation of High-Strength Concrete Slabs, Proc. Nordic Research Meeting, Gothenburg, The Norwegian Concrete Association, Oslo, Ed. by Nilsson, L.O., 1993, pp. 62-64.
24. Persson, B., ”Effect of Silica Fume on Self-Desiccation and Strength”, 16th Symp. on Nordic Concrete Research, Helsinki, Norwegian Concrete Association, Oslo, Ed.: Söderlund, K. 1996, pp. 128-130.
25. Persson, B., ”Ninety-month Pozzolanic Interaction between Portland Cement and Silica Fume in Concrete”, Contribution at a Nordic Mini Seminar on Hydration of Cement, Skagen, Ed. by Bager, D., 1999, 25 pp.
26. Persson, B., ”Self-desiccation”, fib Technical Committee 8.2, Ed.: Müller, 2001, 16 pp.
27. Persson, B., Fagerlund, G., ”Hydration of High-Performance Concrete”, Co-author, 93rd Annual Meeting Abstracts, The American Ceramic Society, Westerville, 1991, pp. 257.
28. Persson, B., ”Specific Volume of Chemically Bound Water and Self-desiccation in Silica Fume and Portland Cement Mortars”, Nordic Seminar, Skagen, 1999, 19 pp.
29. Persson, B., On the Under-Pressure in the Pore Water of Sealed High Performance Concrete, *Concrete Science and Engineering* 2, 2000, pp. 213-221.
30. Persson, B., ”Assessment of Chloride Migration Coefficient, Salt Frost Resistance, Internal Frost Resistance and Sulphate Resistance”, TVBM-3100, Lund Institute of Technology, Lund, 2001, 86 pp.
31. Persson, B., ”Chloride Migration Coefficient of Self-Compacting Concrete”, *Materials and Structures,* Vol. 37, 2004, pp. 82-91.
32. Persson, B., “Thirteen-year Pozzolanic Interaction between Portland Cement and Silica Fume in Concrete”, Suppl. Proc. Eight CANMET/ACI International Conference on Fly Ash, Silica Fume, Slag and Natural Pozzolans in Concrete, Ed. by Gupta, P., 2004, pp. 455-480.