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Porosity And Diffusivity Of Concrete With Long-Term Compressive Strength Increase Due To Addition Of The Set Accelerator Calcium Nitrate

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Summary: Concrete with additions of the set accelerator calcium nitrate (CN) exhibit a long-term strength increase. Investigations by capillary suction of water and chloride migration showed that the strength gain could not be explained by any decrease in porosity. Water vapor diffusivity obtained by mass loss during drying did not reveal any differences either. Visual inspections by scanning electron microscope indicated that the interface between coarse aggregate and paste may be somewhat denser when CN is added and that calcium hydroxide may be in the form of smaller crystals. Pore water analysis from mature paste showed a substantial increase in alkali concentration when alkali-free CN was added. This could indicate a substitution of bound alkalis in the CSH structure by calcium ions. Thus, improved aggregate-paste interface, CSH and CH morphology and CSH composition could contribute to the observed strength increase.

Keywords. Concrete, calcium nitrate, porosity, diffusivity, strength enhancement

1 INTRODUCTION

Calcium nitrate (CN) is used world wide as an admixture for concrete, in particular for applications utilising its properties as set accelerator (Justnes and Nygaard, 1997a). However, CN may be denoted a multifunctional concrete admixture (Justnes and Nygaard, 1999) since it also works as an inhibitor against chloride induced corrosion of rebars (Justnes et al., 1999). Furthermore, CN also enhances the long-term compressive strength of concrete. CN will obviously increase the very early strength due to the earlier start of C_3S hydration and increased maturity at for instance 8 h (Fig.1). However, for concrete (w/c = 0.45 and 4% SF) an increased strength has been observed also at 28 (Fig. 2) and 220 days (Fig. 3) moist curing, in particular for high strength type cement (HSC) compared to ordinary Portland cement (OPC). The strength gain increased until a dosage of 2 % CN of cement weight and flattened out at this level for higher dosages. This paper present the efforts made to explain this unexpected long-term strength increase, since this property should not be utilised in practice before its origin is understood.

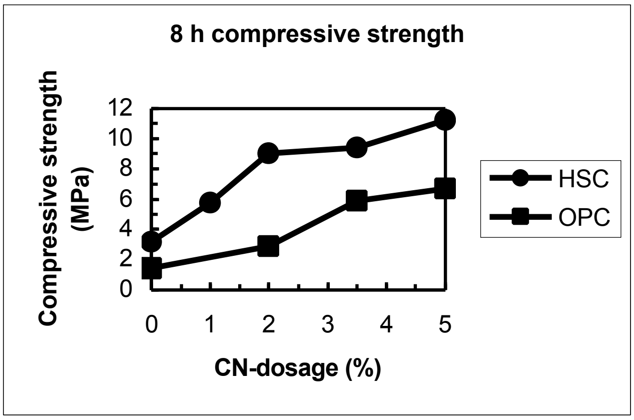

Fig. 1 Early compressive strength (8 h) for high strength Portland cement (HSC) and ordinary Portland cement (OPC) concrete with different dosages of calcium nitrate (CN).

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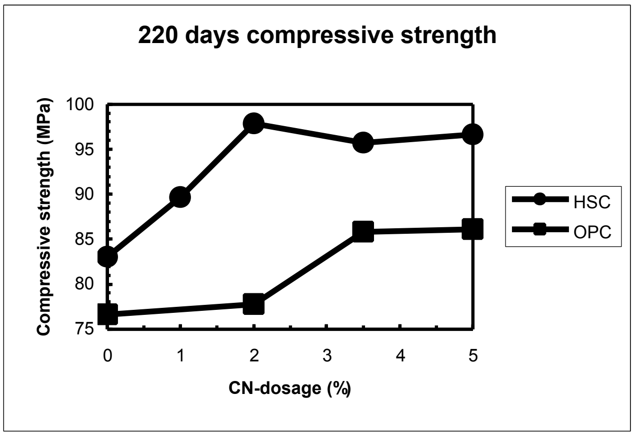

Fig. 2 **Compressive strength at 28 days for HSC and OPC concrete with different dosages of calcium nitrate (CN).**

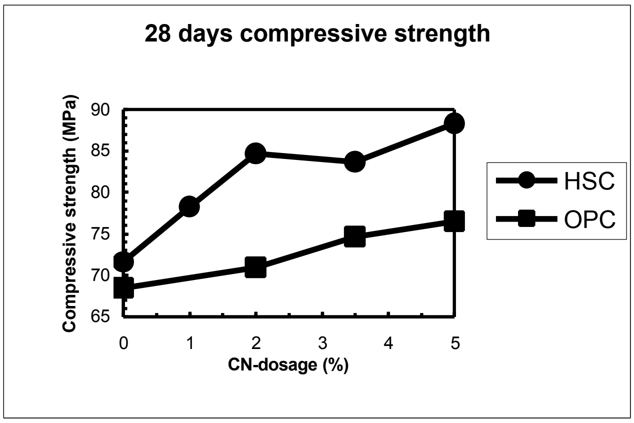

Fig. 3 **Compressive strength at 220 days for HSC and OPC concrete with different dosages of calcium nitrate (CN).**

The first thought was to investigate any change in porosity as a consequence of the CN addition. This was done by capillary suction/saturation by water. Permeability is related to porosity, so chloride migration experiments were carried out as well. Both these parts are also of interest for the discussion of the durability of concrete with CN additions, since it has been common in later years to replace calcium chloride with CN. Additional information was achieved by visual inspection of the microstructure with scanning electron microscopy of plane polished sections, and by studying changes in pore water composition of cement paste with CN additions.

The cement and concrete composition are reported elsewhere (Justnes and Nygaard, 1997a and 1999) but the base recipe (w/(c+s) = 0.45, 4% SF) was of course identical with and without the addition of CN. A granulated, technical quality calcium nitrate (CN) was used that may be expressed as $xNH_4NO_3 \cdot yCa(NO_3)_2 \cdot zH_2O$, with x = 0.092, y = 0.500 and z = 0.826. In other words it consisted of 19.00 % Ca^{2+}, 1.57 % NH_4^+, 64.68 % NO_3^- and 14.10 % H_2O.

2 EXPERIMENTAL

Epoxy coated ϕ100 mm·200 mm cylinders of the 220 days old concrete were available for the present study.

2.1 Capillary suction and relative water vapor diffusion

Deriving concrete parameters from capillary suction is perhaps not so well known and may deserve a closer presentation: Six 20 mm slices were cut from the middle of each of the epoxy coated concrete cylinders for this experiment. The technique usually requires 105°C drying before capillary suction measurements. Since 105°C may coarsen the porosity, prolonged drying at 50°C was tried out first followed by the normal procedure of 105°C drying. After drying, the discs are placed on a grating 1 mm below the water surface in a covered box. The increase in weight as the specimen suck water is monitored for 5 days and plotted versus square root of time as sketched in Fig. 4.

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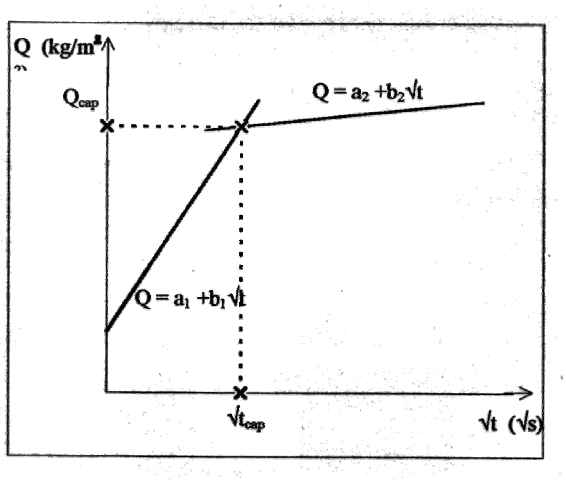

Fig. 4 Principle of water absorption by a sample in the capillary suction test

The results from the capillary suction test can be expressed by means of linear regression analysis. Two lines are obtained; the first line represents the value until the waterfront reaches the top of the specimen and the second line the value thereafter. The crossing between the two straight lines is calculated and expressed by the water absorption value (Q_{cap}) and time for exposure (t_{cap}). These values can be used to calculate the capillary coefficient, k, and the resistance coefficient, m;

$$k = \frac{Q_{cap}}{\sqrt{t_{cap}}} \qquad [\mathrm{kg/m^2 \cdot \sqrt{s}}] \qquad (1)$$

$$m = \frac{t_{cap}}{h^2} \qquad [\mathrm{s/m^2}] \qquad (2)$$

where h is the thickness of the concrete sample (NT Build 368, 1992). Thus, the capillary coefficient, k, gives an estimate of the velocity by which the concrete absorbs water, while the resistance coefficient, m, indicates the time needed for the water front to reach the top.

The procedure consist of 6 important steps for the specimen:

1. Drying to constant weight (W_1)
2. Capillary suction for 5 days with weight monitoring
3. Water saturation by submersion 3 days in water at 1 atm (W_2)
4. Pressure saturation by submersion 3 days in water at 80 atm (W_3)
5. The outer volume (V) is recorded by differential weighing the specimen under water and saturated surface dry in air according to the principle of Archimedes.
6. Drying the specimen to constant weight at 105°C (W_4)

From these 6 steps, one can calculate the initial moisture content, total porosity (ε_{tot}), the capillary porosity (ε_{cap}), the air volume (ε_{air}), the average density of the concrete solids (ρ_{sol}) and the dry density of the concrete (ρ_{dry}) according to the following formulas;

$$Initial \quad moisture = \frac{(W_1 - W_4) \cdot 100\%}{V \cdot \rho_w} \qquad [\mathrm{vol\%}] \qquad (3)$$

$$\varepsilon_{tot} = \frac{(W_3 - W_1) \cdot 100\%}{V \cdot \rho_w} \qquad [\mathrm{vol\%}] \qquad (4)$$

$$\varepsilon_{cap} = \frac{(W_2 - W_1) \cdot 100\%}{V \cdot \rho_w} \qquad [\mathrm{vol\%}] \qquad (5)$$

$$e_{air} = \frac{(W_3 - W_2) \cdot 100\%}{V \cdot r_w} \qquad \text{[vol\%]} \qquad (6)$$

$$r_{sol} = \frac{W_1}{V - \frac{(W_3 - W_1)}{r_w}} = \frac{W_1}{V \cdot \left(1 - e_{tot} / 100\%\right)} \qquad \text{[kg/m}^3\text{]} \qquad (7)$$

$$r_{dry} = \frac{W_1}{V} \qquad \text{[kg/m}^3\text{]} \qquad (8)$$

During the 50°C drying process, the mass versus time to constant weight was monitored as well. And the time, t, to loose 25, 50 and 75 % of the free water was calculated. The diffusion coefficient, D, according to Fick's second law relative to a reference concrete (in this case HSC without CN) can simply be taken as the ratio between the time to obtain the same level of relative free water for the concrete (i) in question and the reference concrete (ref);

$$\frac{D_i}{D_{ref}} = \left(\frac{t_{ref}}{t_i}\right)_{25\%} = \left(\frac{t_{ref}}{t_i}\right)_{50\%} = \left(\frac{t_{ref}}{t_i}\right)_{75\%} \qquad (9)$$

2.2 Chloride migration

Another epoxy coated cylinder was cut in 25, 50, 50, 50 and 25 mm, where the 3 middle 50 mm slices were used as parallels for the chloride migration test (NT Build 355, 1989). In this test a ϕ10mm·50 mm cylindrical specimen is placed between two compartments filled with 3% NaCl solution (compartment 1 with chloride concentration C_1) and 0.3 N NaOH solution (compartment 2), respectively. In both solution a metal screen is placed close to the concrete surface and a constant direct current potential of $\Delta U = 12$ V is applied between them (Fig. 5). The electric resistivity of the specimen is monitored before and after the experiment. Due to the electric field, the chloride ions will migrate through the concrete from the NaCl compartment to the NaOH compartment. The concentration of chloride ion (C_2) in the NaOH compartment as a function of time (t) is determined by a spectrophotometric method, and the results plotted like the sketch in Fig. 6.

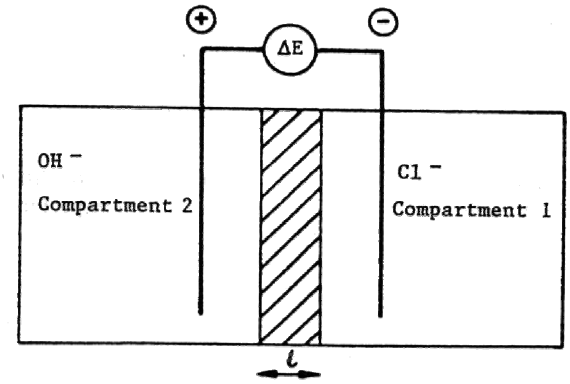

Fig. 5 Principle set-up of a chloride migration experiment

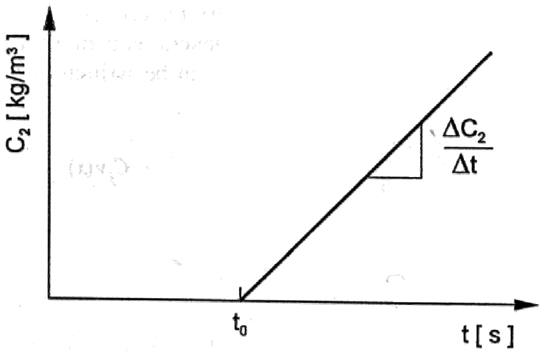

Fig. 6 Sketch of the chloride concentration in compartment 2 as a function of time

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When a straight line is obtained between the 5 last data points, $\Delta C_2/\Delta t$ is determined as the slope of this line as shown in Fig. 6. Assuming that migration is the dominating transport mechanism and that diffusion and convection can be neglected, the migration coefficient for chlorides can be determined by the formula

$$D_m = \frac{R \cdot T \cdot \lambda \cdot V_2}{z_{Cl} \cdot F \cdot \Delta E \cdot g_{Cl} \cdot C_1 \cdot A} \cdot \frac{\Delta C_2}{\Delta t} \qquad (10)$$

where:

R = the universal gas constant = 8.314 J/mole·K

T = absolute temperature [K]

∝ = thickness of the sample [m]

V_2 = volume of compartment 2 [m^3]

Z_{Cl} = electrical charge of chloride = -1

F = Faradays number = 96,500 C/equivalent

ΔE = voltage drop over the specimen [V]

γ_{Cl} = activity coefficient of chloride (taken as 1 here for simplicity)

C_1 = chloride concentration in compartment 1 [kg/m^3] or [mole/l]

A = cross-sectional area of sample [m^2]

$\Delta C_2/\Delta t$ = steady state change in chloride concentration versus time in compartment 2 [$kg/m^3 \cdot s$] or [mole/l·s]

It should be noted that the voltage drop across the specimen, ΔE, is less than the applied voltage, ΔU:

$$\Delta U = \Delta E + E^{rev}_{H_2O} + h \qquad (111)$$

where E^{rev}_{H2O} = reversible dissociation potential of water = 0.463 V at 25°C and pH = 13 and η = polarisation effects [V]. In this study $\Delta U = \Delta E$ for simplicity.

2.3 Other methods

10 mm cubes cut from concrete were impregnated with epoxy under vacuum and plane polished. The microstructure of these specimens was inspected by scanning electron microscopy in the backscattered electron mode.

Finally, pore water was pressed from mature cement paste specimen and analyzed for a number of ions to gain some supplementary information about the microstructure.

3 RESULTS AND DISCUSSION

3.1 Capillary suction/adsorption and relative diffusion

The results from the capillary suction test for concrete based on high strength cement dried at 50 and 105°C are listed in Tables 1 and 2, respectively, while similar results for concrete based on the ordinary Portland cement dried at 50 and 105°C are given in Tables 3 and 4, respectively.

There is no significant change in capillary porosity within each series with varying CN dosage. The capillary coefficient, k, and the resistance number, m, are relatively constant within each series too. However, the values are larger when the samples are dried at 105°C instead of 50°C due to the drier interior and possible changes in pore connectivity (i.e. coarsening of the pore structure) caused by the more severe drying. There are no significant changes in porosity or dry density that can explain the long-term strength (220 days) gain observed when calcium nitrate is added to HSC concrete.

The diffusion coefficients relative to HSC without CN (ref) according to Eq. 9 were for HSC with 1, 2, 3.5 and 5 % CN 0.96±0.02, 0.87±0,01, 0.88±0.02 and 0.92±0.05, respectively, and for OPC with 0, 2, 3.5 and 5 % CN 0.87±0.09, 0.79±0.09, 0.82±0.06 and 0.84±0.84, respectively. Differences are hardly significant, but marginally lower for samples with CN (lower pore water pressure caused by salts in solution) and for OPC vs HSC (more alkalis; 1.2% vs 0.6%).

Table 1. Results with relative standard deviation of the values from the capillary suction/saturation method for HSC concrete as a function of calcium nitrate (CN) dosage when it is pre-dried at 50°C.

CN dosage [%]	0.0	1.0	2.0	3.5	5.0
Initial moisture [vol%]	12.3	13.0	13.6	14.4	15.6
	±9%	±2%	±1%	±1%	±2%
ε_{tot} [vol%]	14.2	14.8	15.1	15.9	15.6
	±6%	±5%	±7%	±5%	±6%
ε_{cap} [vol%]	13.5	13.8	14.1	14.5	14.4
	±6%	±4%	±6%	±5%	±6%
ε_{air} [vol%]	0.7	1.0	1.0	1.4	1.2
	±9%	±16%	±15%	±9%	±13%
m [10^7s/m^2]	10.4	9.6	12.0	15.0	14.3
	±20%	±13%	±39%	±42%	±29%
k [10^{-2}kg/m$^2\sqrt{s}$]	1.22	1.31	1.26	1.13	1.13
	±13%	±8%	±18%	±16%	±17%
ρ_{sol} [kg/m^3]	2683	2687	2685	2697	2703
	±0.2%	±0.1%	±0.3%	±0.2%	±0.1%
ρ_{dry} [kg/m^3]	2302	2291	2281	2270	2282
	±0.9%	±0.7%	±1.2%	±1.1%	±1.0%

Table 2. Results with relative standard deviation of the values from the capillary suction/saturation method for HSC concrete as a function of calcium nitrate (CN) dosage when it is pre-dried at 105°C.

CN dosage [%]	0.0	1.0	2.0	3.5	5.0
Initial moisture [vol%]	0.0	0.0	0.0	0.0	0.0
	±0%	±0%	±0%	±0%	±0%
ε_{tot} [vol%]	14.2	14.7	14.9	15.7	15.4
	±6%	±4%	±6%	±5%	±6%
ε_{cap} [vol%]	13.2	13.3	13.5	14.0	13.8
	±6%	±3%	±6%	±5%	±6%
ε_{air} [vol%]	1.0	1.4	1.4	1.7	1.6
	±8%	±8%	±8%	±6%	±7%
m [10^7s/m^2]	6.03	5.89	5.80	5.36	6.36
	±6%	±7%	±6%	±3%	±18%
k [10^{-2}kg/m$^2\sqrt{s}$]	1.59	1.66	1.74	1.78	1.62
	±7%	±4%	±7%	±4%	±8%
ρ_{sol} [kg/m^3]	2682	2687	2685	2693	2696
	±0.2%	±0.2%	±0.3%	±0.3%	±0.1%
ρ_{dry} [kg/m^3]	2302	2291	2281	2270	2282
	±0.9%	±0.7%	±1.2%	±1.1%	±1.0%

Table 3. Results with relative standard deviation of the values from the capillary suction/saturation method for OPC concrete as a function of calcium nitrate (CN) dosage when it is pre-dried at 50°C.

CN dosage [%]	0.0	2.0	3.5	5.0
Initial moisture [vol%]	13.1	13.4	14.3	14.9
	±1%	±2%	±3%	±6%
ε_{tot} [vol%]	14.1	14.9	15.6	15.9
	±12%	±11%	±5%	±6%
ε_{cap} [vol%]	13.0	14.0	14.7	14.9
	±11%	±10%	±4%	±6%
ε_{air} [vol%]	1.1	0.9	0.9	1.0
	±21%	±18%	±18%	±9%
m [10^7s/m^2]	14.4	26.0	23.9	17.9
	±16%	±19%	±15%	±14%
k [10^{-2}kg/m$^2\sqrt{}$s]	1.02	0.84	0.95	1.03
	±10%	±14%	±4%	±9%
ρ_{sol} [kg/m^3]	2688	2696	2699	2700
	±0.3%	±0.2%	±0.3%	±0.3%
ρ_{dry} [kg/m^3]	2310	2295	2275	2272
	±2.1%	±2.0%	±0.8%	±1.0%

Table 4. Results with relative standard deviation of the values from the capillary suction/saturation method for OPC concrete as a function of calcium nitrate (CN) dosage when it is pre-dried at 105°C

CN dosage [%]	0.0	2.0	3.5	5.0
Initial moisture [vol%]	0.0	0.0	0.0	0.0
	±0%	±0%	±0%	±0%
ε_{tot} [vol%]	14.1	14.7	15.6	15.8
	±12%	±11%	±5%	±6%
ε_{cap} [vol%]	12.9	13.7	14.3	14.5
	±11%	±10%	±4%	±6%
ε_{air} [vol%]	1.2	1.0	1.3	1.3
	±17%	±19%	±15%	±6%
m [10^7s/m^2]	6.56	7.62	6.90	6.80
	±7%	±13%	±10%	±18%
k [10^{-2}kg/m$^2\sqrt{}$s]	1.51	1.53	1.73	1.64
	±12%	±14%	±7%	±11%
ρ_{sol} [kg/m^3]	2691	2691	2695	2702
	±0.4%	±0.2%	±0.4%	±0.4%
ρ_{dry} [kg/m^3]	2312	2295	2275	2275
	±2.0%	±2.0%	±0.8%	±1.0%

3.2 Chloride migration

The results from the chloride migration test; the time to chloride penetration (t_0), the steady state migration coefficient (D_m) and the specific resistance before (ρ_b) and after (ρ_a) the experiment, are given in Tables 5 and 6 for concrete based on HSC and OPC, respectively, with 0, 2 and 5% calcium nitrate added by weight of cement.

Table 5. Results with absolute standard deviation from the chloride migration test for concrete based on HSC

CN dosage [%]	0.0	2.0	5.0
t_0 [h]	825±30	744±39	603±18
D_m [10^{-12} m^2/s]	49±20	50±1	100±25
ρ_b [Ω·m]	122±12	110±12	118±5
ρ_a [Ω·m]	115±4	103±19	99±3

Table 6. Results with absolute standard deviation from the chloride migration test for concrete based on OPC

CN dosage [%]	0.0	2.0	5.0
t_0 [h]	843±27	762±84	566±24
D_m [10^{-12} m^2/s]	71±19	58±16	121±13
ρ_b [Ω·m]	118±22	118±20	117±8
ρ_a [Ω·m]	118±26	56±27	-

There is a tendency for the time to penetration, t_0, to decrease with increasing CN dosage, although the change is hardly significant at the 2% CN level. The time to penetration will increase with increasing porosity or pore connectivity and with decreasing chloride binding capacity of the matrix. The steady state migration coefficient, D_m, seems to be unchanged by the addition of 2% CN, but increases when 5 % CN is added. D_m should be independent of chloride binding, but increases with increasing porosity and pore connectivity.

The specific resistance, ρ_b, before the experiment is apparently independent of the calcium nitrate dosage and cement type. However, there is a tendency for the specific resistance to decrease after the experiment, which can be explained by the increased ionic strength of the pore water due to the sodium chloride content.

There are no observations from the migration experiment that should indicate a decrease in porosity that could explain the long-term strength increase when calcium nitrate is added. On the contrary, the highest dosage seems to increase porosity even though the long-term strength is higher than the reference.

3.3 Scanning electron microscopy

The scanning electron microscopy of the HSC and OPC concrete with 0, 2 and 5 % CN additions indicated two trends; 1) the interface between the coarse aggregate and the paste was denser when CN was added and 2) the crystalline mass of calcium hydroxide had a “dotted” structure indicating it to be composed of smaller crystallites. The difference in interface can be observed from the backscattered electron image (BEI) of HSC concrete without CN in Fig. 7 and the HSC concrete with 2 % CN in Fig. 8.

When calcium nitrate is added, the calcium concentration in the water of the fresh mix will be rather high and the hydroxide concentration depressed due to the limited solubility of calcium hydroxide. The Ca^{2+} concentration for HSC paste with w/c = 0.55 without and with 1.55% CN was reported (Justnes and Nygaard, 1997b) to 20 mM and 90 mM, respectively, after 10 min and declining to 16 mM and 52 mM, respectively, after 4 hours (past initial set). At the same time, the hydroxyl concentration was 63 mM and 25 mM after 10 min, and stabilising at 32 mM for both mixes after 4 h. The initial suppression of the calcium hydroxide solubility, could lead to less initial precipitation of calcium hydroxide (CH) on the surface of the coarser aggregate which consequently could mean a tighter interface in the hardened concrete. The suppressed CH solubility could also lead to the formation of smaller crystallites rather than larger CH platelets that could mean weaker zones in the CSH-gel + CH binder. The same applies for any other sparingly soluble calcium salts.

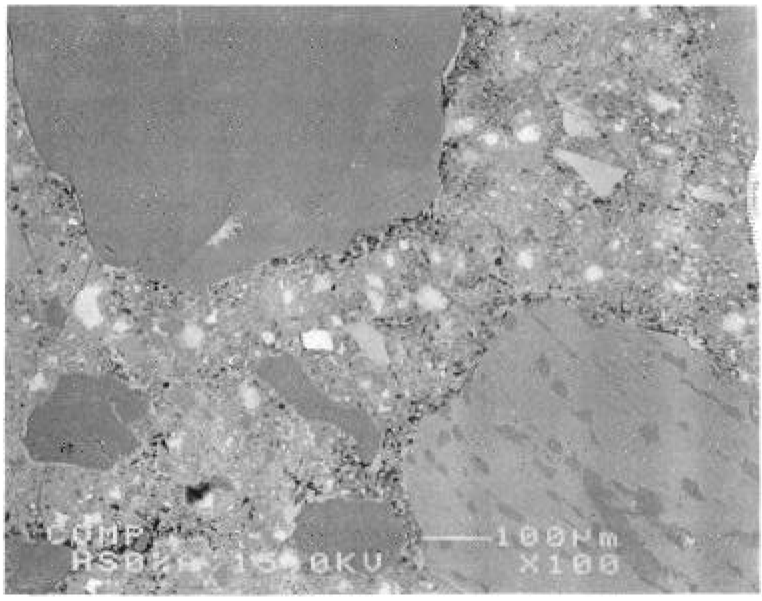

Fig. 7 100x BEI of HSC concrete without CN

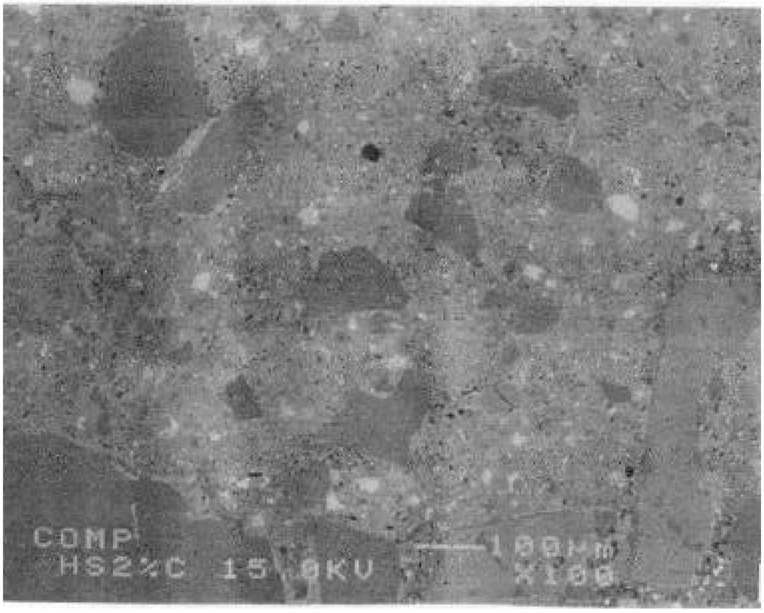

Fig. 8 100x BEI image of HSC concrete with 2 % CN

3.4 Pore water analyses

The ionic composition of water pressed from mature HSC paste with w/c = 0.55 and without and with 5% CN is listed in Table 7. Even though the CN used was free of alkalis, the alkali content increased substantially when CN was added. This can only be explained by Ca^{2+} replacing K^+ and Na^+ bound in the CSH gel. Such a substitution can heal dislocations in the CSH structure on an atomic layer and possibly increase its strength. An earlier study of changes in microstructure of paste (Justnes and Nygaard, 1997c) showed by ^{29}Si NMR that the average length of the polysilicate anions in the amorphous CSH-gel was prolonged by 17% for OPC and 5 % for SRC (sulphate resistant cement), but unfortunately the HSC was not included in that investigation.

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Table 7. Pore water composition in ppm [mM] and sums of cation (S_+) and anion (S_-) charge of mature HSC paste with w/c = 0.55 and without/with 5% CN.

CN-dosage	0.0%	5.0%
K^+ (39.10 g/mole)	690 [17.6]	9,810 [250.9]
Na^+ (22.99 g/mole)	230 [10.0]	3,390 [147.5]
Ca^{2+} (40.08 g/mole)	70 [1.7]	210 [5.2]
NO_3^- (62.0 g/mole)	0.5 [0.0]	16,700 [269.4]
NO_2^- (46.00 g/mole)	2.6 [0.1]	13 [0.3]
OH^- (17.01 g/mole)	- [100]	- [63.1]
SO_4^{2-} (96.06 g/mole)	135 [1.4]	24 [0.2]
Σ_+	- [31]	- [409]
Σ_-	- [103]	- [333]

Note the excess of negative charge (Σ_-) in the paste without CN and the excess in positive charge (Σ_+) in the paste with 5% CN. However, the OH^- concentration is based on pH meter readings to 13.0 and 12.8, respectively, and represents the highest uncertainty. A change in 0.1 pH units in this range corresponds to about 20 mM alone.

4 CONCLUSIONS

Concrete with additions of the set accelerator calcium nitrate (CN) exhibit a long-term strength increase. The addition of 2% CN to concrete based on high strength Portland cement, 4 % silica fume and w/(c+s) = 0.45 lead to an increase in 220 days compressive cube strength from 83 to 98 MPa.

Investigations by capillary suction of water, relative water vapour diffusion and chloride migration showed that the strength gain could not be explained by any decrease in porosity. The porosity was rather constant as CN was added, with a marginal porosity increase for the highest CN dosage (5 %).

Visual inspections by scanning electron microscope indicated that the interface between coarse aggregate and paste may be somewhat denser when CN is added and that calcium hydroxide may be in the form of smaller crystals. Morphology changes due to suppressed calcium hydroxide solubility by CN additions due to common ion effects can not be ruled out. Both these effects could contribute to strength increase.

Pore water analysis from mature paste showed a substantial increase in alkali concentration when alkali-free CN was added. This could indicate a substitution of bound alkalis in the CSH structure by calcium ions. Such a healing of CSH dislocations could possibly also strengthen the binder.

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