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High Strength Concretes made with Densified and Undensified CSF – strength development and resistance to carbonation and soft water attack

Authors: Y Ballim and N Ananmalay

ABSTRACT:
This paper discusses the results of a research project aimed at assessing the performance of condensed silica fume (CSF) concretes in soft water and carbonation environments. Since CSF consumes portlandite during hydration, an important motivation for this investigation was to assess the effects of a reduced portlandite content on the durability of concrete in environments which involve the alteration or consumption of portlandite as the 'deterioration' mechanism.

Concretes were prepared with a nominal strength of 70 MPa, with 0%, 5%, 10% and 15% of the portland cement (OPC) replaced by undensified CSF. A second group of concretes was also prepared in which 10% and 15% of the OPC was replaced by densified CSF. These concretes were exposed to a range of curing environments and the near surface zones were tested for fluid transport characteristics using the water sorptivity tests. Samples were exposed in a purpose-made accelerated soft water environment and the rate of deterioration was monitored by determining the mass loss of the samples at regular intervals. Sample groups for carbonation testing were allowed to dry for up to 1095 days indoors in the laboratory and exposed to the elements outdoors. A separate set of samples were also exposed to an accelerated carbonation facility in the laboratory.

The results indicate that, for initial moist curing periods of 3 days or less, the CSF concretes show similar resistance to soft water attack. However, for well cured concretes, CSF significantly increases the resistance of concrete to soft water attack, compared with OPC concrete of the same nominal strength. In the case of carbonation resistance, except for the concrete with 5% undensified CSF, the CSF concretes all show higher rates of carbonation than the OPC after approximately 3 years of exposure.

Journal Contact Details:
PO Box 75364
Lynnwood Ridge
Pretoria, 0040
South Africa
+27 12 348 5305

CONCRETE SOCIETY
OF SOUTHERN AFRICA

admin@concretesociety.co.za
www.concretesociety.co.za

High strength concretes made with densified and undensified CSF

- Strength development and resistance to carbonation and soft water attack

TECHNICAL PAPER

Y. Ballim and N Ananmalay

SYNOPSIS

This paper discusses the results of a research project aimed at assessing the performance of condensed silica fume (CSF) concretes in soft water and carbonation environments. Since CSF consumes portlandite during hydration, an important motivation for this investigation was to assess the effects of a reduced portlandite content on the durability of concrete in environments which involve the alteration or consumption of portlandite as the "deterioration" mechanism.

Concretes were prepared with a nominal strength of 70 MPa, with 0%, 5%, 10 % and 15% of the portland cement (OPC) replaced by undensified CSF. A second group of concretes was also prepared in which 10% and 15 % of the OPC was replaced by densified CSF. These concretes were exposed to a range of curing environments and the near surface zones were tested for fluid transport characteristics using the water sorptivity tests. Samples were exposed in a purpose-made accelerated soft water environment and the rate of deterioration was monitored by determining the mass loss of the samples at regular intervals. Sample groups for carbonation testing were allowed to dry for up to 1095 days indoors in the laboratory and exposed to the elements outdoors. A separate set of samples were also exposed to an accelerated carbonation facility in the laboratory.

The results indicate that, for initial moist curing periods of 3 days or less, the CSF concretes show similar resistance to soft water attack. However, for well cured concretes, CSF significantly increases the resistance of concrete to soft water attack when compared with OPC concrete of the same nominal strength. In the case of carbonation resistance, except for the concrete with 5% undensified CSF, the CSF concretes all show higher rates of carbonation than the OPC concrete after approximately 3 years of exposure.

1.0 INTRODUCTION

Condensed silica fume (CSF) first became commercially available in South Africa as a cement extender in 1987. In the early days of its introduction, it was used in a relatively small number of projects, the primary reason for this being a lack of knowledge of the performance of the local material. However, as the knowledge and experience base has grown, CSF has found increasing acceptability in the concrete industry. More recently, it was used in the construction of a new aluminium smelting plant in which concrete durability was a prime consideration in the design and construction of the project.

CSF is available for use in concrete in South Africa in two forms:

- undensified condensed silica fume (UCSF) - this is CSF in its natural captured state, with a characteristic particle size of approximately 0,1 μm and a bulk density approximately 200 kg/m^3.
- densified condensed silica fume (DCSF) - in order to ease handling and transport, the CSF is subjected to a patented electrostatic process, in which the particles are agglomerated into small pellets, increasing the average particle size and bulk density to approximately 0,2 mm and 600 kg/m^3 respectively.

It is now generally accepted that CSF has the effect of reducing permeability(1,2,3) and refining the pore structure(1,4,5) of hardened cement paste and concrete. This has often been translated into an indication of the improved durability of CSF concrete since most of the mechanisms of concrete deterioration are associated with the flow of fluids through the pore structure of the concrete. Indeed, the durability benefits of CSF in concrete have been demonstrated in deterioration mechanisms such as salt scaling(6) and reinforcing steel corrosion(7). However, in the case of carbonation, which involves a reaction between carbon dioxide (CO_2) and the calcium hydroxide ($Ca(OH)_2$ or portlandite) released in the hydration of cement, there are two competing factors to be considered with the use of CSF concrete. The reduced permeability caused by the addition of CSF reduces the accessibility of the pore structure to CO_2. On the other hand, the hydration of the CSF is a pozzolanic reaction which consumes $Ca(OH)_2$. Hence, for a unit amount of CO_2 entering the pore structure, the carbonation depth will be greater in the case of CSF concrete when compared with a plain OPC concrete of a similar permeability and binder content. In this regard, Skjolsvold(8) shows that, after 4 years of exposure, the carbonation depth of a 50 MPa CSF concrete is similar to that of a 30 MPa OPC concrete.

Given this durability effect of the reduced $Ca(OH)_2$ content of CSF concretes, the project reported in this paper was undertaken to assess the strength and durability performance of concretes subjected to carbonation and soft water environments. Soft water was included in the study because, like carbonation, the mechanism of deterioration involves an interaction between the environment and the $Ca(OH)_2$ present in the pore structure of the concrete. Based on a search of the literature, it appears that no work has been undertaken to assess the performance of CSF concretes in soft water environments.

The project involved testing a wide range of concretes made with both the types of CSF mentioned above in order to characterise the material and relate these characteristics to the measured performance of the concretes when exposed to aggressive environments. However, this paper reports only on the compressive strength (briefly) and durability performance of the high-strength concretes used in the investigation.

For each of the concretes assessed, compressive strength was determined at various ages up to 1095 days of wet curing. Other samples were subjected to a range of early-age water curing before being tested for surface water sorptivity or exposed to the soft water or carbonation environments. For the soft-water environment, samples were exposed to continuously circulating, de-ionised water and the mass loss of the samples was determined under both surface brushed and unbrushed conditions. Carbonation results are presented for samples allowed to dry both in the laboratory and in an outdoor, unsheltered environment.

2.0 MATERIALS AND MIXES

2.1 ORDINARY PORTLAND CEMENT (OPC)

Sufficient bags of OPC were randomly selected from one day's production at a local cement plant. Samples were then assessed to ensure compliance with the requirements of SABS 471(9). Also, a particle size distribution analysis conducted by the supplier showed that 50% of the sampled material fell in the 3 to 32 µm size range, while 26% was greater than 50 µm. The result of a chemical analysis of the cement is shown in Table 1.

2.2 CONDENSED SILICA FUME

The CSF used in this investigation was obtained from Alpha Limited's Witbank plant. Sufficiently large samples of both the densified and undensified materials were drawn from the plant and stored in sealed containers in the laboratory. A chemical analysis of the CSF is also shown in Table 1.

	Portland Cement (%)	Silica Fume (%)
SiO_2	22.78	90.89
$A1_2O_3$	4.16	1.58
Fe_2O_3	2.54	1.55
CaO	64.33	0.41
MgO	1.45	0.60
K_2O	0.27	0.99
TiO_2	0.31	-
SO_3	1.84	-
Mn_2O_3	0.13	0.02
V_2O_5	0.01	0.01
P_2O_5	0.17	0.09
Free Lime	1.10	-
LOI	1.58	3.05

Table 1: Chemical composition of the ordinary portland cement and condensed silica fume

2.3 AGGREGATES

A nominal 19 mm crushed dolerite stone and dolerite crusher sand were used as the coarse and fine aggregates for all the concretes. The crusher sand was found to be slightly too coarse with a fineness modulus of 3.8, which is greater than the upper limit of 3.6 recommended(10) for South African concretes. Nevertheless, trial mixes indicated that concrete of suitable workability could be made without the use of an additional fine sand.

2.4 SUPERPLASTICISER

Where necessary, a sulphonated naphthalene superplasticiser was used in the concrete. The material used complies with ASTM C494(11) as a Class F high-range water-reducing admixture. It is supplied in liquid form with a relative density of 1.18 and is stated to contain no chloride.

2.5 CONCRETE MIX PROPORTIONS

Basis of mix design: Using trial mixes with a water content of 205 ℓ/m^3, an OPC mixture was prepared to achieve a slump of 60 ± 10 mm (by adjusting the superplasticiser content) and a 28-day target strength of 70 MPa. Discussions with local concrete practitioners indicated that concretes containing CSF have higher workability when subjected to mechanical vibration than equivalent ordinary portland cement concretes with the same slump values. This led to the decision to reduce the target slump requirement of the CSF mixtures to 50 ± 10 mm.

Using the same water and stone contents of the OPC mixture as determined above, five further concrete mixtures were prepared as follows:

- three mixtures in which 5%, 10% and 15% of the binder was made up of UCSF;
- two mixtures in which 10% and 15% of the binder was made up of DCSF.

For each of the CSF mixtures, the required superplasticiser content and water/binder (w/b) ratio were determined during trial mixing. The final proportions of each of the mixtures are given in Table 2.

Binder Type:	OPC	OPC + 5% UCSF	OPC + 10% UCSF	OPC + 15% UCSF	OPC + 10% DCSF	OPC + 15% DCSF
Mix ID:	**PC**	**5U**	**10U**	**15U**	**10D**	**15D**
PC (kg)	574	497	424	401	424	401
UCSF (kg)	-	26	47	71	-	-
DCSF (kg)	-	-	-	-	47	71
Water (kg)	205	205	205	205	205	205
Stone (kg)	1100	1100	1100	1100	1100	1100
Sand (kg)	714	750	788	777	788	777
Admix. (ℓ)	3.13	2.19	1.98	3.57	1.84	3.16
w/b ratio	0.36	0.39	0.43	0.43	0.43	0.43
Slump (mm)	70	60	55	60	55	55

Table 2: Mixture proportions (per m^3) for the concretes tested

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DCSF (kg)	**-**	**-**	**-**	**-**	**47**	**71**
Water (kg)	**205**	**205**	**205**	**205**	**205**	**205**
Stone (kg)	**1100**	**1100**	**1100**	**1100**	**1100**	**1100**
Sand (kg)	**714**	**750**	**788**	**777**	**788**	**777**
Admix. (ℓ)	**3.13**	**2.19**	**1.98**	**3.57**	**1.84**	**3.16**
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Slump (mm)	**70**	**60**	**55**	**60**	**55**	**55**

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3. LABORATORY PROCEDURE

3.1 TEST SAMPLES

100 mm cube samples were used for all the tests in this investigation. The fresh concrete was compacted in the moulds using a mechanical vibrating table. Samples were de-moulded at 24 hours after casting, after which they were placed in the respective curing environments.

3.2 CURING REGIMES

The extents of initial curing varied for the different test methods and these are shown in Table 3. Wet curing was achieved by placing the de-moulded cubes into a curing bath controlled at 22 ± 1 °C. In the case of the samples wet cured for 1 day, these samples were placed in the drying environment upon de-moulding.

After the wet curing period, the samples were placed in a drying environment in the laboratory where the temperature and relative humidity were controlled at 22 ± 2 °C and 60 ± 5% respectively. In the case of the samples stored outdoors, these were wet cured for 3 days, after which they were placed in an outdoor environment in an unsheltered area.

Test Method	Wet Curing Period (days)	Age at Test (days)
Compressive Strength	1, 3, 7, 28, 90, 1095	At the end of the wet curing period
Water Sorptivity	1, 3, 7, 28	28
Carbonation	1 3 7 28	1095 for indoor and outdoor exposed samples; 87 for accelerated test samples
Soft Water Attack	1 3 28	Exposed at 28 days; tested at various ages up to 190 days

Table 3: Water curing and testing periods for the different test methods

3.3 TEST METHODS

Compressive Strength: Three cube samples were crushed at each of the test ages indicated in Table 3. The test was conducted in accordance with SABS 863[3].

Water Sorptivity: At 28-days and after the respective curing/drying regimes shown in Table 3, a central 68 mm ø core was drilled from one of the vertical moulded faces of each cube. Two disks were then cut from this core to represent the 0-20 mm and 20-40 mm sections of the surface. These disks were tested for water sorptivity using the procedure described by Ballim(12).

Carbonation: Samples to be tested for carbonation were stored in the indoor and outdoor drying environments as described above. The initial wet curing periods and times of testing are shown in Table 3.

An additional group of samples was prepared for testing in an accelerated carbonation facility. This facility consists of a non-pressurised chamber in which the CO_2 concentration is maintained at 10 % via a commercially available CO_2 control module, fed from a pressurised CO_2 cylinder. The samples were stored on open-grid shelving in the chamber and CO_2 was continuously circulated around the samples by a small electric fan placed in the chamber.

The temperature in the room housing the carbonation chamber was controlled at 24 ± 1°C. Relative humidity inside the chamber was controlled at 50 ± 3% by suspending trays of saturated magnesium nitrate-hexa-hydrate ($Mg(NO_3)_2.6H_2O$)(13) below each of the shelves.

After water curing, the samples were air dried in the laboratory up to age 87 days before being placed in the carbonation

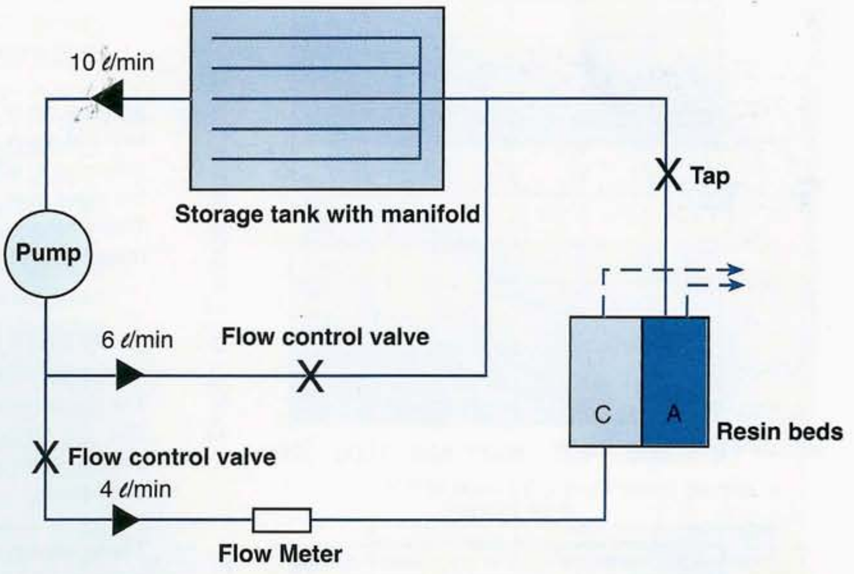

Figure 1: Schematic arrangement of the accelerated soft water attack facility

chamber. At an age of approximately 70 days, four contiguous faces of each cube were coated with a water-based epoxy, leaving two opposite, moulded faces open for CO_2 to penetrate the concrete.

In all cases, the depth of carbonation was measured using phenolphthalein sprayed onto a freshly broken surface of the concrete sample. This process was carried out in accordance with the procedure recommended by RILEM CPC-18(14).

Soft Water Attack: A facility was built in the laboratory to allow concrete samples to be exposed to an accelerated soft water environment. As shown schematically in Figure 1, the facility consists of a storage tank which contains the samples submerged in water. Water is drawn off at the top of the tank using a pump with a capacity of 10 ℓ/min. The flow is then split so that 4 ℓ/min is allowed to pass through de-ionising resin beds in which the anions and cations in the water are removed. This de-ionised water then re-enters the tank through a manifold system placed at the bottom of the tank. The remaining 6 ℓ/min of flow is allowed to bypass the resin beds and flows directly into the manifold. The reason for this arrangement was the mismatch in the capacities of the pump and resin beds. This flow rate allowed for the a volume of water equal to that contained in the storage tank, to be passed through the resin beds in a 24-hour period.

The cation and anion resin beds (marked C and A on Figure 1 respectively) are designed to allow in-line regeneration, which is necessary when their capacity to absorb ions is depleted. The tap on the downstream end of the resin beds was used to obtain water samples for conductivity measurements to determine the time for regeneration.

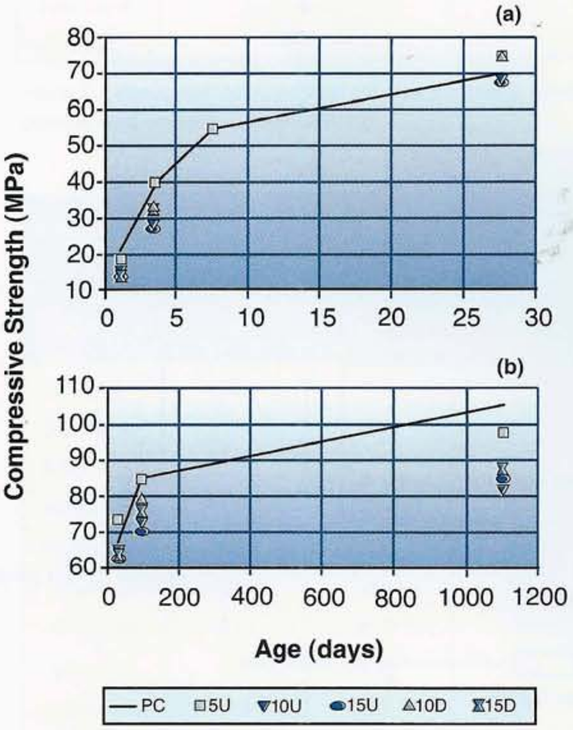

Figure 2: Compressive strength results for the various concretes for the periods (a) 1 to 28 days, and (b) 28 to 1095 days.

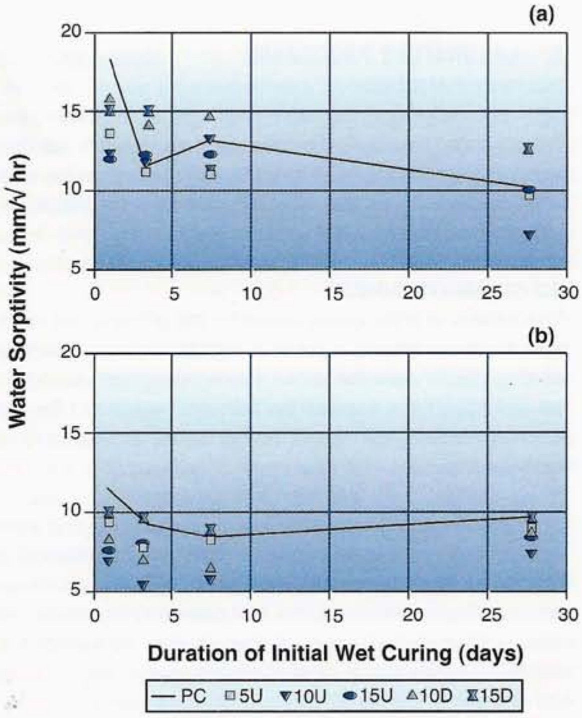

Figure 3: Variation in water sorptivity with duration of initial wet curing for the various concretes for the: (a) 0 to 20 mm segments, and (b) 20 to 40 mm segments

The outlet water from the resin beds during regeneration was routed to waste. Municipal tap water was used to top-up the storage tank when the water level dropped as a result of evaporation or water loss during regeneration.

Samples were placed in the soft-water storage tank at 28 days after casting, having been exposed to the curing/drying regimes indicated in Table 3. At regular intervals up to 190 days of exposure in the tank, companion sets of samples were weighed under the following conditions:

- unbrushed: weighed after gently drying the surface of the sample;
- brushed: the sample was manually brushed on all surfaces using a stiff nylon brush to remove all loosely adhering material. The sample was then rinsed in water, surface dried and weighed.

A further set of samples was placed in a lime-saturated curing tank and these were weighed at the same times as the samples subjected to soft water attack. This allowed a determination of the mass gain of the samples upon re-absorption of water. These measurements were then used to determine the effective mass loss of the deteriorated samples.

4. RESULTS AND DISCUSSION

4.1 COMPRESSIVE STRENGTH

The compressive strength results are shown in Figures 2(a) and 2(b). For clarity, the curves have been separated to show the strength gains over the 0 to 28 day period and the 28 to 1095 day periods.

These results show that the measured 28-day strengths lie between 67 MPa and 75 MPa, representing a range of -4% to +7% with respect to the target strength. Regarding the strength performance of the CSF concretes, the 5U concrete shows similar strength and rate of strength gain as that of the OPC concrete, up to 90 days after casting. However, at 1095 days after casting, the compressive strength of the 5U concrete is approximately 7% lower than that of the OPC concrete. The compressive strengths of the remaining four CSF mixes are generally lower than that of the OPC concrete and it appears that an increase in the rate of strength gain occurs between 7 and 28 days. This rate of strength gain is not sustained beyond 28 days and, by 1095 days after casting, the compressive strength of this group of concretes is approximately 19% lower than that of the OPC concrete.

The effect of a superplasticiser in concrete is to prevent flocculation of the cement or CSF, improve dispersion and subsequent hydration(15). It is therefore likely that a part of the reason for the variation in strengths at the different ages can be ascribed to the variation in superplasticiser content of the concretes. However, for the results shown in Figure 2, there is no clear relationship between the compressive strength at any age and the superplasticiser content of the mixture.

4.2 WATER SORPTIVITY

The variation in the 28-day water sorptivity value, in response to the initial water curing period for each of the concretes, is shown in Figures 3(a) and 3(b) for the 0 to 20 mm and 20 to 40 mm segments respectively. For the 0-20 mm segments, except for the relatively high value of water sorptivity of the OPC concrete cured for 1 day, the results all fall within a narrow band of sorptivity values. This band becomes even narrower for the 20-40 mm segments and there are no clear trends regarding the effect of CSF on the water sorptivity of the internal sections of the concretes. It is interesting to note that the concretes with 10% densified and undensified CSF generally showed the lowest water sorptivity values for both the surface and internal segments.

4.3 CARBONATION

The results of carbonation depth measurements for the indoor, outdoor and acceleration chamber exposed samples are shown in Figures 4(a), 4(b) and 4(c) respectively.

The general observation from these results is that, after 3 years, the concretes have all shown fairly low rates of carbonation for initial wet-curing periods of 3 days or more. Also, for all the durations of initial wet curing used, the OPC and 5U concretes show the lowest rates of carbonation. Aside from the 5U concrete, the CSF concretes all show an unexpected trend of increasing rates of carbonation when the initial wet-curing period is increased from 7 to 28 days. While the differences in the carbonation depths are fairly small and may result from experimental error, the fact that it occurred with this group of concretes only may indicate that this requires further investigation. This phenomenon is, however, not noted with the accelerated carbonation results. The lower rates of carbonation of the OPC and 5U concretes is also reflected in the results of the outdoor exposure tests. The magnitude of the results obtained for the outdoor exposure samples is lower than that for the indoor samples because of the effects of cyclic wetting and drying of the outdoor samples. Wetting of the samples during a rain period has the effect of arresting the carbonation reaction until the concrete has dried sufficiently to the depth of carbonation before the rain period(16).

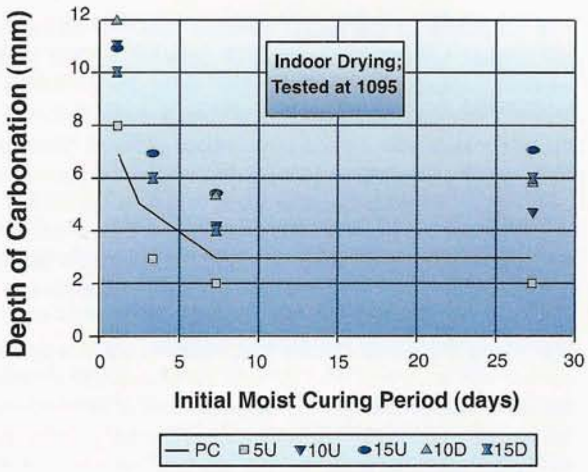

Figure 4(a):Variation in depth of carbonation with initial moist curing period for the concretes dried indoors

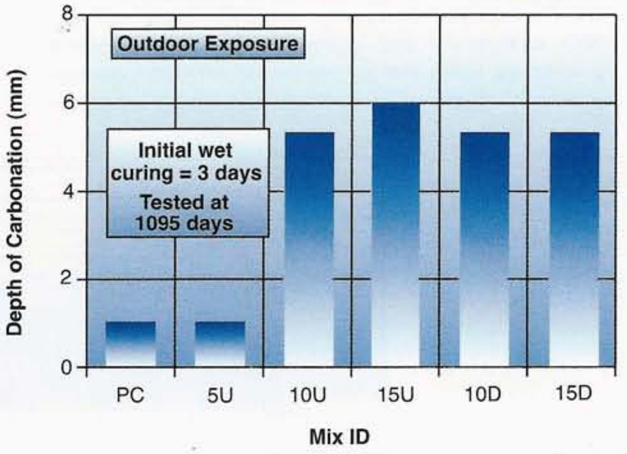

Figure 4(b): Depth of carbonation of the samples exposed outdoors

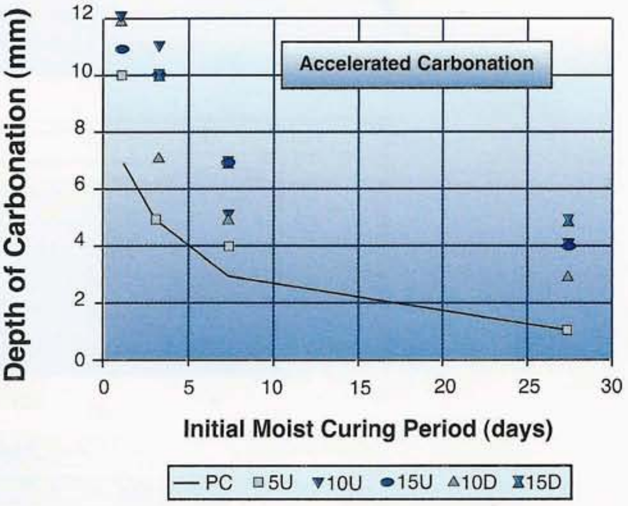

Figure 4(c): Effect of initial wet curing period on the depth of carbonation of the different concretes exposed in the accelerated carbonation chamber

The magnitude of the results obtained from the accelerated carbonation tests is similar to that obtained for the indoor exposed samples. This indicates that, given the conditions of testing, the accelerated carbonation gives a useful indication of the carbonation after 3 years under indoor exposure conditions. Also, ignoring the OPC and 5% CSF concretes, the accelerated carbonation chamber gives a result which is equivalent to approximately 15 years of outdoor exposure. This is based on the carbonation rate equation : $x = D.t^{1/2}$, where x is the depth of carbonation at time t and D is the carbonation coefficient.

Importantly, the results indicate that, replacing ordinary portland cement with more than 5% CSF with the objective of achieving the same 28-day strength, increases the rate of carbonation in the resulting concrete. This is in spite of the fairly similar fluid transport characteristics of the surface sections of the concretes, in terms of the water sorptivity results obtained at 28 days after casting. The implication here is that the effect of the CSF in consuming $Ca(OH)_2$ during hydration is large enough to cause an acceleration in the carbonation rate because of the lower amount of $Ca(OH)_2$ available for the carbonation reaction. This agrees with the results obtained by Skjolsvold(8).

4.4 SOFT WATER ATTACK

The results of the mass loss of the samples exposed in the accelerated soft water attack facility for the brushed and unbrushed conditions are shown in Figures 5(a) and 5(b) respectively. Note that the vertical scales on these figures are different and that, as expected, the concretes showed a lower mass loss under the unbrushed condition.

In general, the CSF concretes showed very similar behaviour to that of the OPC concrete. However, the OPC concrete shows an unexpected behaviour in that the samples which were water cured for 28 days before exposure, gave significantly higher mass loss results than the companion samples which were cured for 1 and 3 days only. This behaviour was noted for both the brushed and unbrushed conditions. Some of the CSF concretes also show this trend but the increases are small in comparison with that of the OPC concrete. The reason for this behaviour is unclear at this stage and, while this result may well be anomalous, it does appear to warrant further and more fundamental investigation.

In considering the CSF concretes only, there is no clear trend regarding the relative resistance of the concretes containing the different CSF types with different replacement ratios. Also, there is a marginal benefit in extending the initial wet curing period

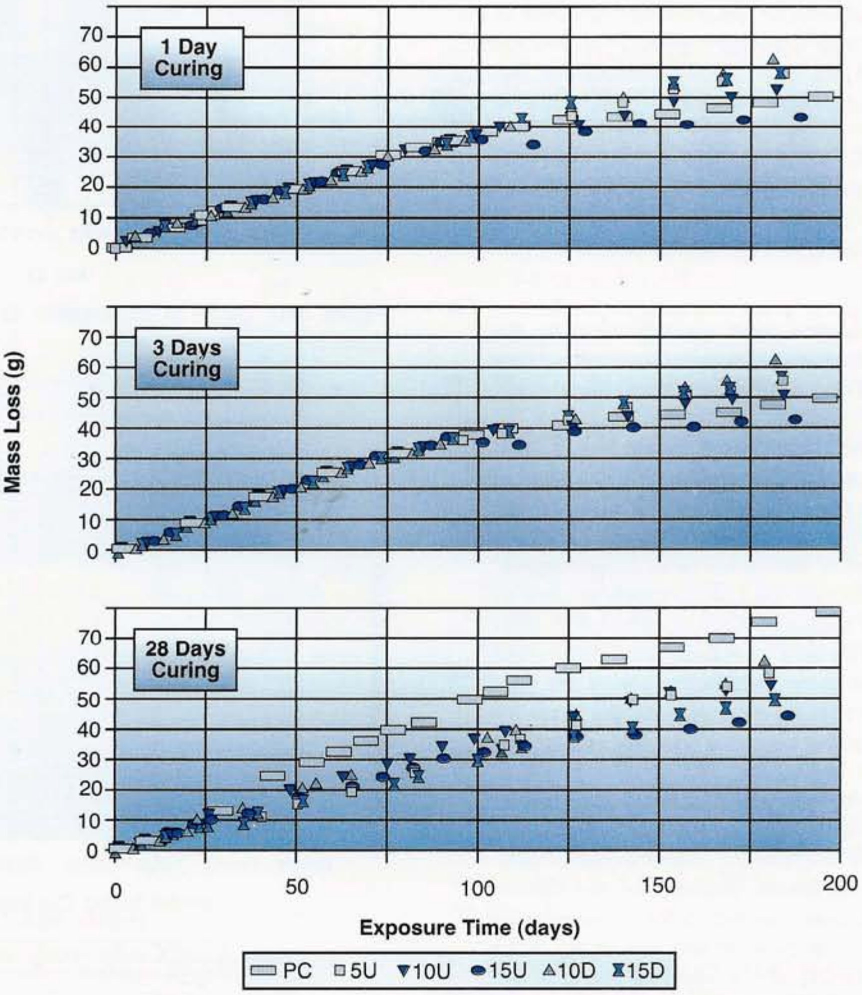

Figure 5(a): Mass loss of the samples exposed to soft water attack and regularly brushed, for the three periods of initial wet curing.

from 1 to 3 days, while no further benefit is derived in extending the wet curing period to 28 days. This comment relates to concretes allowed to dry in the protected environment of the laboratory. The situation may be different if the concretes were dried outdoors and exposed to the elements. Furthermore, given the conditions of this test, the consumption of $Ca(OH)_2$ in the hydration of CSF does not appear to make the resulting concrete more susceptible to attack by soft water. Here again, this is likely to derive from the effects of CSF on both the physical microstructure of the binder paste as well as the chemical nature of the hydration products.

5. CONCLUSIONS

1. Using CSF as a cement replacement with the objective of achieving a similar 28-day compressive strength has the effect of significantly reducing the rate of strength gain after 28 days. This rate reduction increases as the CSF replacement is increased from 5% to 10%. The concretes with 15% CSF replacement showed similar rates of strength gain during this period as the concretes containing 10% CSF replacement.

2. The addition of CSF at cement replacements up to 15% results in concrete in which the surface zone has a similar or lower water sorptivity after 1 day of moist curing than an equivalent ordinary portland cement concrete. This difference in the fluid transport properties of the surface zone becomes insignificant for wet curing periods of longer than 1 day and at depths greater than 20 mm below the concrete surface.

3. At a replacement level of 5%, the carbonation resistance of CSF concrete is similar to that of an equivalent ordinary portland cement concrete. At replacement levels of 10% and 15%, the CSF concretes show significantly higher rates of carbonation than the ordinary portland cement concrete.

4. Given the high strengths of the concretes tested in this investigation, the resistance of the concrete to soft water attack is largely unaffected by CSF replacement up to 15% under both brushed and unbrushed conditions. However, the OPC concrete showed an anomalous behaviour in that the mass loss of the samples wet cured for 28 days before exposure to the soft water was significantly higher than that for the samples water cured for 1 and 3 days. This aspect requires further investigation.

5. This investigation showed no significant difference in the performance of the densified and undensified CSF concretes in terms of strength, surface water sorptivity or resistance to carbonation and soft water attack. ●

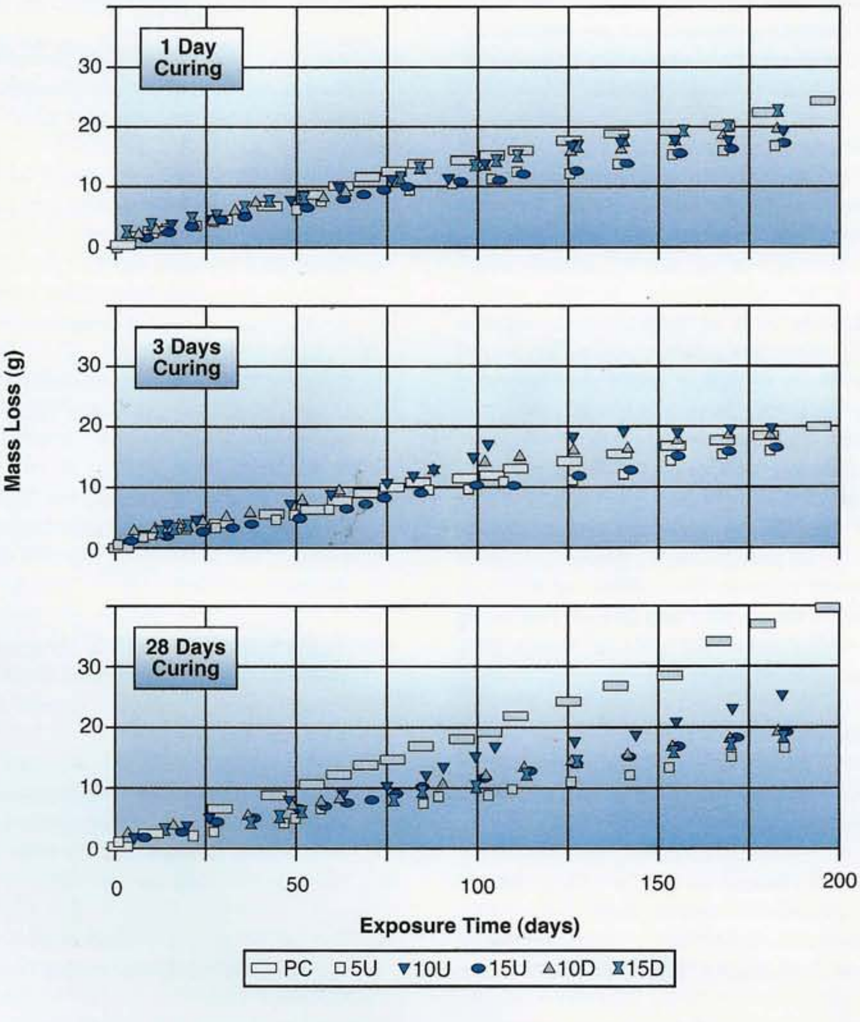

Figure 5(b): Mass loss of the samples exposed to soft water attack in an unbrushed condition, for the three periods of initial wet curing.

Prof Yunus Ballim

Nathan Ananmalay

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