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Mechanical Properties and Frost Resistance of Silica Fume Concrete

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Abstract

Freeze–thaw tests were carried out on air-entrained and non-air-entrained concrete prisms containing different dosages of condensed silica fume (CSF). Six concrete mixes were made incorporating 0, 5 and 10% CSF as partial replacements for OPC. The performance of the concrete prisms exposed to 210 cycles of freezing and thawing was assessed from weight, length, resonance frequency and pulse velocity measurements of the test specimens before and after freezing and thawing. Tests were also conducted to determine the compressive and flexural strengths and the static modulus of elasticity. Although the control concrete gave better durability factors (92%) than those obtained for the CSF concrete (85%), the physical appearance of the CSF prisms exhibited less scaling. © 1997 Elsevier Science Ltd. All rights reserved.

Keywords: Silica fume, concrete, durability, freeze–thaw tests.

INTRODUCTION

In recent years, many workers have shown considerable interest in research aimed at energy conservation in the concrete industry. This is partly being accomplished by the use of less energy intensive cementitious materials such as fly ash, slags, condensed silica fume (CSF) and more recently metakaolin. The need for more durable concrete in particular with improved resistance to freeze–thaw exposure has resulted in a number of investigations on CSF concrete. These investigations included studies of air pore system characteristics[1–3], ice formation and pore structure[4–8], freeze–thaw tests with and without de-icing salts[2,6,7,9] and chemical structure[10–12].

Although concrete with high strength may be produced using CSF[13], the enhanced strength cannot always guarantee adequate performance against freezing and thawing. Several studies, including that by Sorenson[9], indicated that air entrainment is necessary to ensure good freeze–thaw resistance of CSF concrete. Furthermore, the response of CSF concrete to the number of freezing and thawing cycles may be different in that, at a given low number of cycles, the deterioration may be marginal, but deterioration may accelerate later. It has been suggested[14] that test specimens should be subjected to more cycles of freezing and thawing than normally prescribed.

Earlier work by the author[15] showed that exposure to 35 cycles of freezing and thawing results in only slight reductions in the durability factors of CSF concrete as compared to those of the control concrete. Furthermore, it was reported[15] that there was no noticeable difference in the physical appearance of the CSF and control concrete specimens. This paper reports the results of exposure to 210 freeze–thaw cycles. The control concrete mix was that usually adopted for in-situ paving. The OPC in the control mix was partially replaced by up to 10% CSF. The performance of the concretes was assessed from measurements of weight, length, resonant frequency and pulse velocity carried out at weekly intervals. At the termination of the freeze–thaw tests, the prism specimens were subjected to flexural strength tests. The broken portions of the prisms were

Table 1. Physical properties and chemical composition of cement (OPC) and condensed silica fume (CSF)

	OPC	*CSF*
Physical properties		
Particle size (mm)	15	0.15
Surface area (m^2/kg)	350–390	15000–20000
Bulk density (kg/m^3)	1300–1400	200–300
Specific gravity	3.14	2.2
Composition (%)		
Silicon dioxide (SiO_2)	20.9	92.0
Ferric oxide (Fe_2O_3)	2.2	1.2
Aluminium oxide (Al_2O_3)	4.5	0.7
Calcium oxide (CaO)	64.0	0.2
Magnesium oxide (MgO)	2.3	0.2
Sodium oxide (Na_2O) and pottasium oxide (K_2O)	0.88	2.0
Loss on ignition	1.0	—
Insoluble residue	0.5	—
Sulphuric anhydride (SO_3)	3.2	—

then tested for compressive strength by the equivalent cube method.

EXPERIMENTAL DETAILS

Materials

The cement was OPC complying with BS12: 1989. The physical properties and chemical composition of the OPC are given in Table 1.

The fine aggregate was natural sea-dredged sand from the Bristol Channel. The sieve analysis showed that the sand complied with grades C and M of BS 882: 1983. The grading and physical properties are given in Tables 2 and 3, respectively.

Twenty-millimetre maximum size crushed limestone supplied by a local quarry was used as the coarse aggregate. The grading and physical properties are given in Tables 2 and 3, respectively.

The CSF was supplied in a slurry form; the mass ratio of CSF solids to water was 1:1. The physical properties and chemical composition of the CSF particles are given in Table 1.

Table 2. Grading of fine and coarse aggregates

Coarse aggregate		*Fine aggregate*	
Sieve size (mm)	*Percentage retained*	*Sieve size (mm)*	*Percentage retained*
10	29.8	5 mm	2.4
5	93.4	2.36 mm	8.1
2.36	98.9	1.18 mm	21.8
		600 mm	50.2
		300 mm	89.9
		150 mm	99.8

Table 3. Physical properties of aggregates

	Coarse aggregate	*Fine aggregate*
Relative density (SSD)*	2.65	2.70
Water absorption (percentage of dry mass)	0.34	2.26

*SSD, saturated surface dry.

A polymeric sulphonate-based dark liquid superplasticizer was employed in the CSF mixes to compensate for the water demand of the CSF. The superplasticizer (Cormix SP1) does not contain chlorides and has a specific gravity of 1.14 at 20°C.

Cormix AE1 air-entraining agent, a dark brown liquid, was employed in some of the mixtures. This is a neutralized wood resin with no chloride content and conforms to ASTM designation C260 and BS 5075: part II: 1983 for air entraining admixtures. Its specific gravity is 1.034.

Definitions

In all the mixtures incorporating CSF, the water/binder ratio (w/b) is based on the total cementitious materials, i.e. OPC+CSF solids. In calculating w/b, allowance was made for the water included in the CSF slurry. The CSF content is defined as the mass ratio of CSF solids to the OPC particles employed in the control mixture (without CSF), expressed as a percentage.

Mix details

Several trial mixes were made in order to achieve the required slump (50 mm) and air content (5 ± 1 1/2 %). CSF has a high water demand, and to compensate for the loss in slump, a superplasticizer was used in all the mixtures containing CSF. The quantities used in the control mixture were as follows: cement, 330 kg/m^3; water, 165 kg/m^3; fine aggregate, 724 kg/m^3; coarse aggregate, 1181 kg/m^3. A total of six mixtures were prepared; three of the mixtures contained air-entraining agent at a dosage of 40 ml for every 50 kg of blend (OPC+CSF). The CSF mixtures were formulated on the basis of the control concrete with OPC replacements by CSF of 5% and 10%. Table 4 gives the details of the mixtures employed in the present work.

The fresh concrete was subjected to slump, compaction factor and air content tests in accordance with BS 1881: 1983 Parts 102, 103 and 106, respectively, and the results are shown in Table 5. It is seen that increasing CSF content led to reductions in the air content. This may be explained by the filler effect due to the very small particle size of CSF, being 100 times smaller than OPC particles.

Specimen preparation and testing

From each mixture, six 100-mm cubes; three of which were tested for compressive strength tests at 7 days and 28 days, three cylinders (150 mm diameter × 300 mm long) for 28 days for elastic modulus tests and two 100 mm × 100 mm × 500 mm long prisms for the freeze–thaw tests were prepared. All the samples were cast in steel moulds. The specimens were left to cure for 24 h and were then demoulded and cured in water at 20°C. The prism specimens were cured for 28 days before being subjected to freezing and thawing cycling. Additional 100 mm cube samples were prepared for water absorption tests.

Freeze–thaw exposure

The freeze–thaw chamber used in this investigation consists of refrigerating and heating equipment, which produces continuous freeze–thaw cycles with chamber temperatures in the range of ±20°C. The measured temperatures in the specimens during a full cycle changed from −13°C to +12°C. Figure 1 depicts typical temperature profiles in both the chamber and the samples over a 24 h period. Each specimen was placed in galvanized steel containers and surrounded by water at all times while in the freeze–thaw apparatus.

Dynamic modulus of elasticity

Frost damage in concrete is commonly assessed on the basis of the change in the modulus of elasticity. The modulus of elasticity of concrete may be determined by dynamic as well as static compression tests. The dynamic methods employ ultrasonic velocity and mechanical reso-

Table 4. Mix proportions

Mix no.	*Cement (kg/m³)*	*CSF (%)*	*Slurry (kg/m³)*	*AE1 (ml/m³)*	*SP1 (l/m³)*	*Water (kg/m³)*
1	330.0	0	0	0	0	164.9
2	330.0	0	0	264	0	164.9
3	313.7	5	33	0	1.13	147.6
4	313.7	5	33	264	1.13	147.6
5	297.2	10	66	0	1.34	131.1
6	297.2	10	66	264	1.34	131.1

Coarse aggregate: 1181 kg/m³; fine aggregate: 724 kg/m³.

Table 5. Properties of fresh concrete

Mix no.	*CSF (%)*	*Slump (mm)*	*Compaction factor*	*Air content (%)*
1	0	35–40	0.80–0.85	5.9
2	0	40–50	0.83–0.87	6.6
3	5	60–80	0.91–0.95	4.7
4	5	70–90	0.93–0.97	5.3
5	10	40–60	0.88–0.91	3.7
6	10	50–70	0.90–0.93	4.2

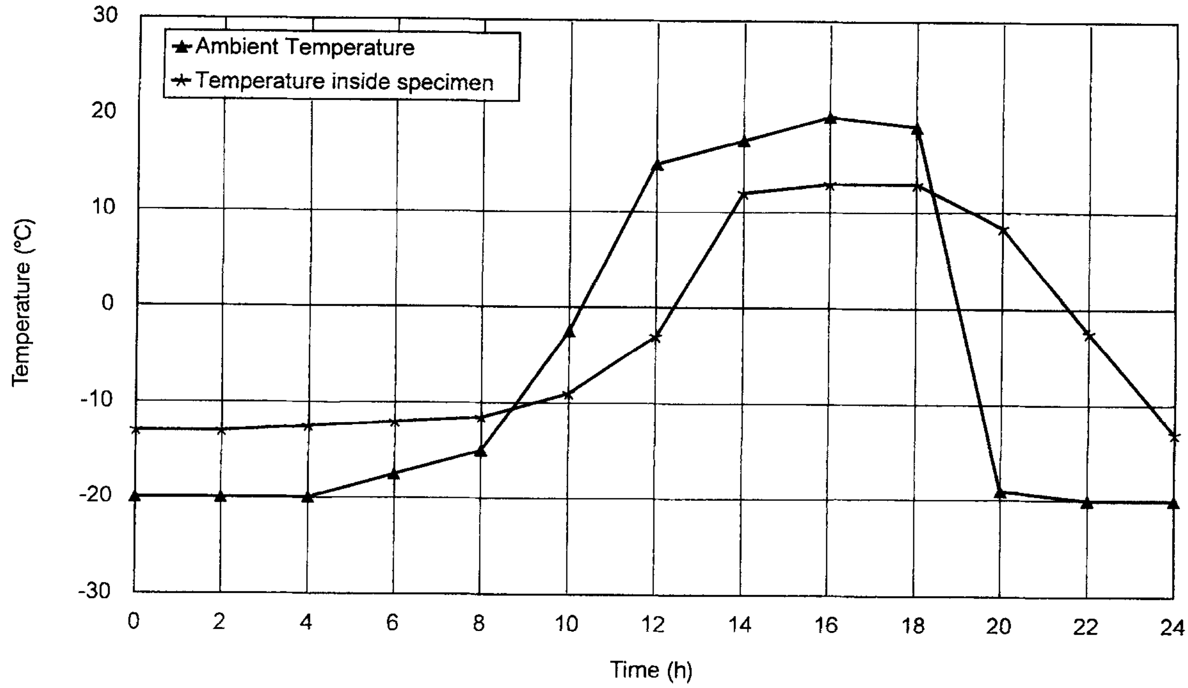

Fig. 1. Typical temperature profile over a full cycle of freezing and thawing.

nant frequency tests. Both these tests were employed in the present investigation.

Ultrasonic pulse velocity

In this test, the concrete samples are subjected to compression or shear wave pulses. Compression pulses of ultrasound with frequencies around 150 kHz were passed through the prisms by means of transmitting and receiving transducers. The travel time of the pulse through the specimen was obtained using electronic circuitry.

Fundamental longitudinal vibration

Immediately after the specified curing period, the prisms were tested for the fundamental longitudinal frequency in accordance with BS 1881: Part 209: 1990. The method is based on the principle that the resonance frequency of a concrete beam depends on the velocity of compression waves propagating through it. This measurement was carried out, with the prisms in a thawed condition, at intervals of seven cycles of freezing and thawing.

Durability factors

The freeze–thaw tests were conducted in accordance with procedure A of ASTM C666, details of which were given in Ref. 15. In this procedure, the specimens are subjected to freezing and thawing in water. The method is not intended to provide a quantitative measure of the length of service that may be expected from a specific type of concrete but is recommended as being adequate for use in determining the effects of variations in the formulations of the concrete on its resistance to freezing and thawing.

The durability factor (DF) is determined from the relative dynamic modulus of elasticity using the following expression:

$$DF = E_r N/M, \qquad (1)$$

where E_r is the relative dynamic modulus of elasticity at N cycles (as a percentage); N is the number of cycles at which E_r reaches the specified minimum value for discontinuing the test or the specified number of cycles at which the exposure is to be terminated, whichever is the smaller; and M is the specified number of cycles at which the exposure is to be terminated.

The relative dynamic modulus of elasticity E_r is determined from

$$E_{r,c} = (n_1/n)^2 \times 100, \qquad (2)$$

where $E_{r,c}$ is the relative dynamic modulus of elasticity after c cycles of freezing and thawing (as a percentage); n_1 is the fundamental frequency after c cycles of freezing and thawing;

and n is the fundamental frequency before freezing and thawing. The above calculation for the relative dynamic modulus of elasticity is based on the assumption that the weight and dimensions of the specimen remain constant throughout the test. This is clearly not true, and the E_r values thus determined can be considered adequate only for comparison between concretes of different formulations.

Water absorption tests

Initial surface absorption tests were conducted in accordance with BS 1881: Part 5: 1970. The specimens used were 100-mm cubes, and the tests were conducted after 28 days' curing in water at 20°C. Before testing, the specimens were dried in a well-ventilated oven at 110°C for 24 h. The specimens were then placed in an air-tight cooling cabinet until tested. The rate of flow of water into the concrete was determined at 10-min, 30-min, 1-h and 2-h intervals.

RESULTS AND DISCUSSION

Mechanical properties

It is now well known that 5–10% OPC replacements by CSF enhances the strength of concrete. Even higher dosages of CSF may be employed if suitable water-reducing agents are used[13]. CSF improves the concrete in two ways. First, due to its high pozzolanicity, it contributes to the hydration reaction between OPC and water by reacting with the calcium hydroxide to produce additional calcium silicate hydrate gel leading to enhancement in strength. This reaction is now well understood, and the results presented in Table 6 provide further evidence on the role played by CSF in strength development. These results show that CSF leads to significant increase in the 7-day and 28-day compressive strengths of both the air-entrained and non-air-entrained samples.

The second role played by CSF is that in altering the matrix structure of the concrete. Because of their extremely small particle size, the CSF particles occupy the voids between the cement grains, acting as a filler, reducing the porosity of the bulk cement matrix and resulting in a densified structure.

The flexural strength development of CSF concrete is similar to that for compressive strength. Carette and Malhotra[2], for example, found that the flexural strength at 28 days was higher than that of the control concrete. The present work shows an improvement in the flexural strengths for both the air-entrained and non-air-entrained samples (see Table 6).

The influence of CSF on the modulus of elasticity of concrete is not yet fully established, and the limited results available to-date are contradictory. For example, Sellevold[16] found that CSF did not influence the modulus of elasticity of concrete. Galeota *et al.* [17] concluded that the modulus of elasticity of normal-weight concrete increased with increasing CSF content. They reported that the increase was smaller for lightweight concrete. More recently, Sabir[13] found that the modulus of elasticity for concretes with strengths in the range 70–90 MPa (CSF contents up to 16%) was only slightly increased with increasing compressive strength. It was also found that marked reductions in the modulus of elasticity occurred as the compressive strength increased beyond 90 MPa with CSF contents greater than 16%. Alfes[18] also reported reductions in the modulus of elasticity of high strength concrete when 20% and 30% CSF contents were used. The addition of CSF to concrete reduces the porosity of the cement

Table 6. Mechanical properties

Mix no.	*Compressive strength (MPa)*			*Flexural strength (MPa)*		*Modulus of elasticity (GPa)*
	7 days	*28 days*	*After 210 cycles*	28 days	*after 210 cycles*	*28 days*
1	43.4	62.5	54.4	6.6	5.7	33.5
2	41.3	58.2	50.1	5.8	4.7	30.4
3	47.1	71.2	63.1	6.9	5.4	32.7
4	46.0	65.9	51.8	7.0	6.0	31.1
5	51.5	76.4	66.9	7.2	5.7	35.6
6	48.1	73.5	64.5	7.0	5.9	33.8

matrix, including that in the transition (interfacial) zone between aggregate and hydrated cement paste. This results in a more effective composite action where the aggregate's contribution to the overall modulus of elasticity is greater. In general, therefore, as with normal concrete, i.e. without CSF, an increase in the compressive strength of CSF concrete is accompanied by a smaller increase in the modulus of elasticity. The limited data available to date for normal strength CSF concrete, including those shown in Table 6, support this.

Freeze–thaw resistance

One of the first studies on the permeability of CSF concrete was that conducted by Markestad[19]. Replacement of 20% of the cement by CSF was found to result in a completely impermeable concrete in which the water-binder ratio was 0.89. Johansson[20] found that a 10% cement replacement by CSF results in a 50% reduction in the concrete's permeability. Although a 20% cement replacement resulted in a further reduction in the permeability, this was substantially less than double that obtained with 10% CSF. Gjorv[21] reported that the water permeability was greatly reduced when CSF was added to lean concrete mixtures. He also reported that the effect of CSF on the water permeability was small when the cement content was more than 400 kg/m^3.

Permeability tests on CSF concrete have also been made on samples cored from existing structures. Magge[22], for example, measured the permeability of samples cut from structures of age varying between 3 and 9 years. He found that the permeability increased with increasing wate-binder ratio and that for equal strengths, concretes containing CSF resulted in a significantly reduced permeability.

The general findings indicate that for low levels of CSF (5–10%) and at low concrete strength levels (up to 40 MPa), the CSF is more efficient in acting as a filler than as a pozzolan[23]. Figure 2 gives the results of the water absorption tests carried out in the present study. It was found that the air-entrained specimens did not behave significantly differently to the other specimens, and that reduced absorption was obtained with the CSF concrete. The reduction in the absorption of the concrete with CSF was about 30–35% of that obtained for the control concrete. This reduction is somewhat lower than that observed by other workers[14] for normal strength concrete and may be related to the level of strength developed and cement content used.

The frost resistance of concrete is greatly influenced by the water absorption capacity and

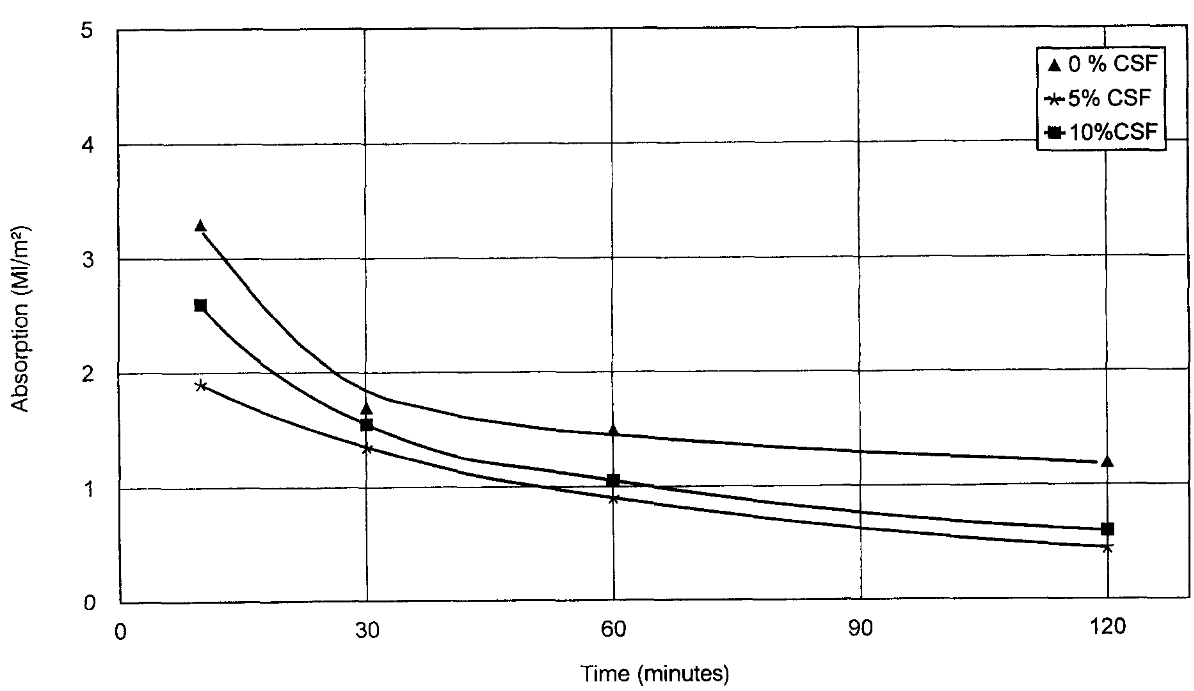

Fig. 2. Water absorption.

pore structure. In general, the addition of CSF results in pore refinement and increased watertightness of the cement matrix. The reduction of water penetration rate improves the durability of CSF concrete to frost attack. The refinement of the pore structure, however, leads to reduced permeability of the hydrated cement paste and can lead to retarded moisture migration through the cement matrix. This can lead to build-up of internal hydraulic pressure, which causes cracking in the concrete. The pore refinement due to CSF is also likely to result in the small voids being saturated leading to a reduction in the freeze–thaw resistance.

Several investigations have been carried out on the durability of CSF concrete exposed to freezing and thawing and, although considerable data are now available on this subject, some of the findings have been inconclusive and, indeed at times, contradictory. This is not surprising in view of the number of factors that have considerable influence on the performance of the concrete with respect to frost action. These include cement content, CSF replacement levels, water-binder ratio, and whether or not water-reducing and air-entrainment admixtures are employed. Furthermore, curing conditions and age and moisture states[24] of the concrete at the time of exposure are important considerations. The difficulties associated with the diversity of the parameters and conditions involved are compounded by the lack of agreement on the method and media of testing and the duration of exposure to freezing and thawing. In the main, the studies have been conducted following procedures suggested by ASTM and RILEM standards. These standards have been continuously revised, and there are currently two RILEM draft recommendations[25,26] for freeze–thaw resistance testing of concrete.

Despite the above difficulties, the evidence so far suggests that at low water-binder ratios (0.25–0.3) and low cement replacement levels (5–10%), CSF has a beneficial effect on frost resistance giving small reductions in the durability factors (<90) with increasing CSF content[14,27]. Higher CSF contents (20–30%) have detrimental effects over the range of water-binder ratios 0.35–0.55[2,28]. Air entrainment is generally found to be beneficial in CSF concrete[29], but there is evidence[5,9,30] to suggest that it is possible to produce CSF concrete that is frost-resistant without air-entrainment, provided that the water-binder ratio is low enough (0.3) and the CSF content is high enough (10% or more). CSF concrete may respond differently to freezing and thawing, as compared with normal concrete, in that the deterioration may be marginal at a low number of cycles, but may accelerate later. It has been suggested[14] that CSF concrete should be subjected to more cycles of freezing and thawing than normally prescribed.

The prism specimens tested here were subjected to a total of 210 freezing and thawing cycles. The tests were started after curing the specimens in water at 20°C for 28 days. After completion of the freeze–thaw tests, the prisms were tested for flexural strength. The equivalent cube test (BS 1881: Part 119: 1983) was then used to determine the compressive strength of the concretes. Table 6 gives the results for the flexural and compressive strengths. It can be seen that the exposure to freezing and thawing results in considerable reductions (10–20%) in the values obtained at 28 days.

The relative dynamic modulus of elasticity was determined at weekly intervals by measuring the resonance frequency of the prisms in the thawed condition. These were used in eqn (1) to determine the durability factors. Figure 3 shows the durability factors as a function of the number of freeze–thaw cycles. The durability factors for the control concrete (without CSF) throughout the test were above 92% with the air-entrained specimens showing slightly higher values. The CSF concretes, however, gave considerably reduced factors over those obtained for the control concrete. Furthermore, the air entrained in the CSF concrete appears to have a detrimental effect on the frost resistance. This is in contradiction to the normally observed behaviour, but on closer examination of the results, it was found that this effect is a reversal of what is observed at the earlier stages of exposure to freezing and thawing. To clarify this point, the results for the durability factors up to 42 cycles of freezing and thawing are replotted in Fig. 4. It can be seen that, over this range, air-entrainment leads to improved resistance in all the specimens, i.e. with or without CSF, although reduced factors were obtained for the CSF concretes. It is also interesting to note that, during early exposure, reductions in the durability factors are obtained with increasing CSF contents. This overall behaviour corresponds to that normally observed for CSF

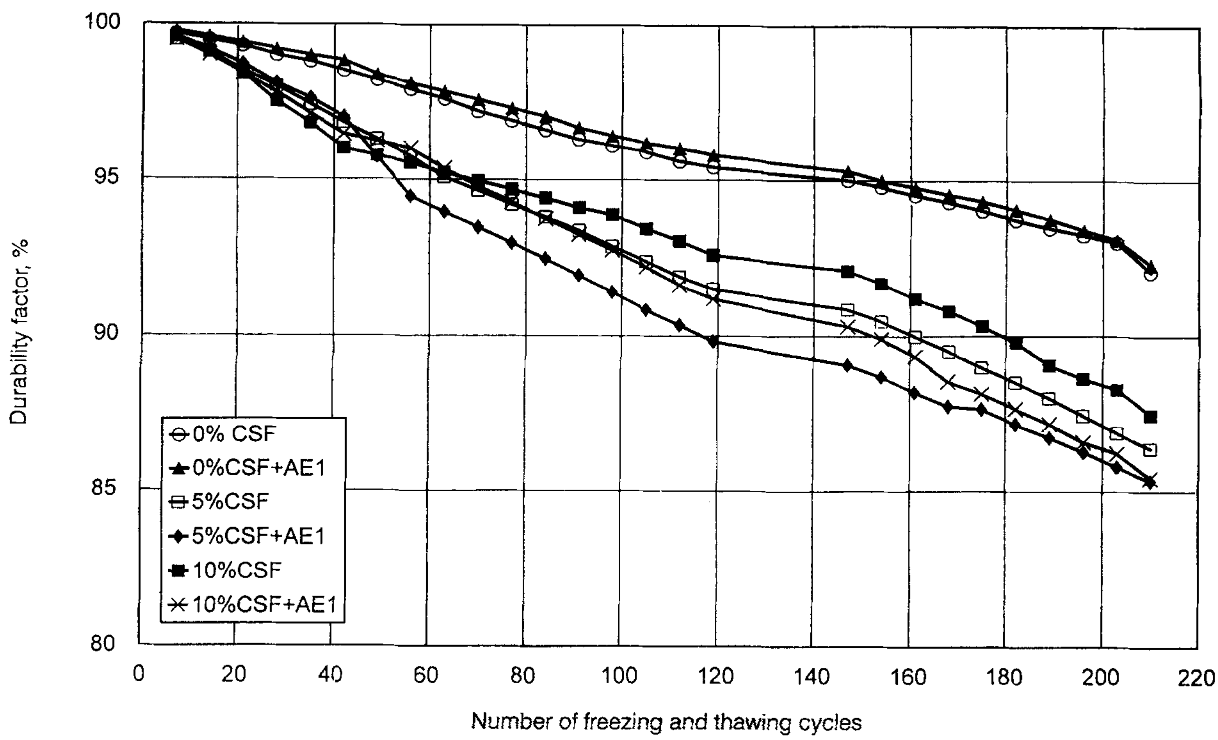

Fig. 3. Durability factors up to 210 cycles.

concrete which has traditionally been subjected to a relatively small number of cycles (30–40)[15]. The results obtained in the present study indicate that longer exposure to freezing and thawing leads to significant internal damage to the extent that air-entrainment results in a deleterious effect in the long term. The results also suggest that higher CSF contents may be beneficial for long-term frost resistance.

Measurements of weights, lengths and pulse velocities were carried out before exposure and after 42 and 210 cycles. These results, together with the durability factors, are shown in Tables 7 and 8. It can be seen that up to 42

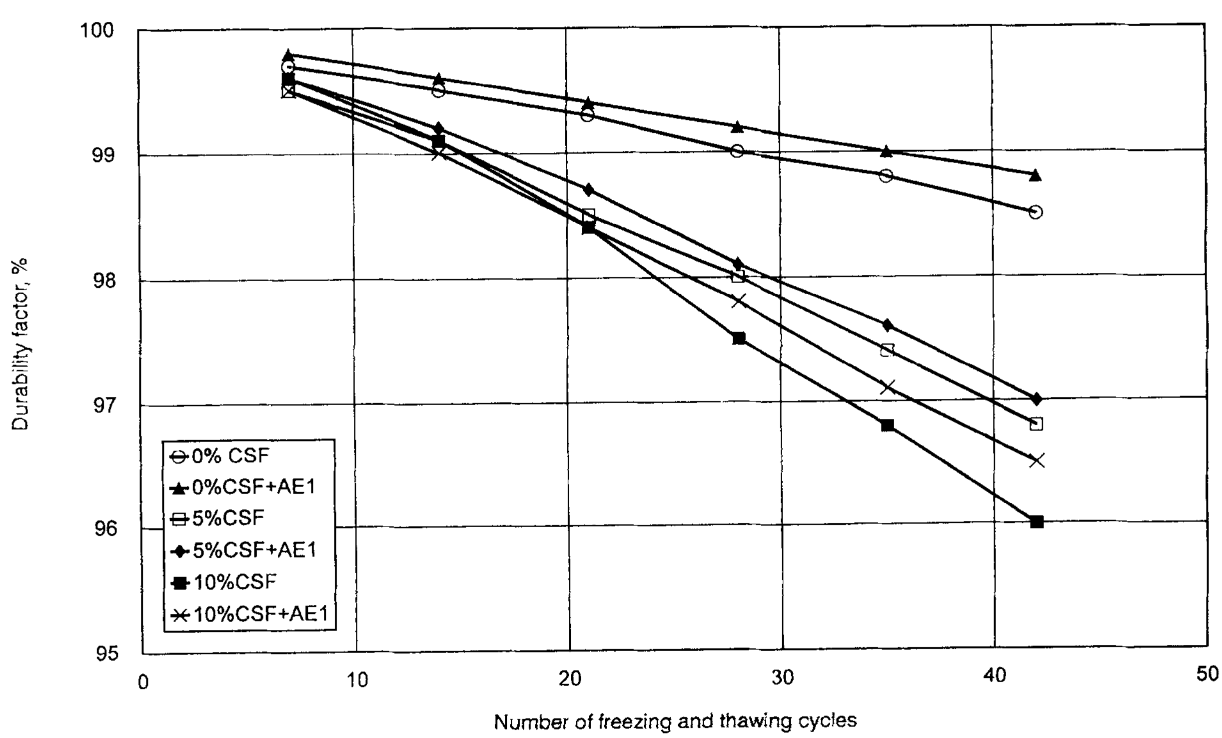

Fig. 4. Durability factors up to 42 cycles.

Table 7. Summary of test results after 42 cycles of freezing and thawing

Mix no.	Percentage change			Durability factor (%)
	Weight	Length	Pulse velocity	
1	−0.59	0.06	−1.07	98.5
2	−0.57	0.07	−1.15	98.8
3	−0.40	0.06	−0.98	96.8
4	−0.36	0.08	−0.92	97.0
5	−0.32	0.10	−0.87	96.0
6	−0.29	0.13	−0.81	96.5

Table 8. Summary of test results after 210 cycles of freezing and thawing

Mix no.	Percentage change			Durability factor (%)
	Weight	Length	Pulse velocity	
1	−3.54	0.56	−2.25	92.1
2	−5.36	0.26	−3.42	92.3
3	−0.69	0.05	−3.47	86.4
4	−1.89	0.42	−3.26	85.3
5	−0.64	0.10	−4.24	87.5
6	−1.25	0.34	−3.32	85.4

cycles of freezing and thawing (Table 7), increasing CSF content leads to improvements in the weight loss and pulse velocity reduction. Little expansion generally takes place, and this is found to increase with increasing CSF content. Table 8 shows that long-term exposure leads to a significant loss in weight and that CSF remains to be beneficial in this respect. There is also an increase in the expansion of the specimens and considerable reduction in the pulse velocities. This is in line with the reductions obtained in the relative dynamic modulus of elasticity, which resulted in the observed reductions in the durability factors.

CONCLUSIONS

The work described in the present investigation gives rise to the following conclusions:

(1) The employment of CSF (5–10%) in concrete leads to improved compressive and flexural strengths irrespective of whether or not air-entraining additives are used.

(2) The influence of CSF on the static modulus of elasticity is not fully understood. Although the available data for normal strength concrete point to a small increase in the modulus with increasing compressive strength, at higher strengths and higher CSF contents, the observed relationships are contradictory. CSF affects the porosity of the hydrated cement paste in a major way. At the lower CSF contents, the porosity is reduced, but there are indications[13] that at higher CSF contents (> 10%), there is an increase in porosity. The porosity at the aggregate–paste interface affects the composite action between the aggregates and hydrated cement paste. The development of the composite action may also be affected by the possibility[3,14] that, in CSF concrete, this interface is small compared with that in normal concrete. These factors, which clearly influence the modulus of elasticity of the concrete, need further study through microscopic examination and porosity measurements.

(3) Exposure to 210 cycles of freezing and thawing leads to reductions of 10–20% in the compressive and flexural strengths. The higher reductions are generally obtained in the concretes containing CSF.

(4) The incorporation of CSF in concrete leads to reduced durability factors over those obtained for the control concrete. On examination of the external surfaces of the specimens, however, the CSF concrete showed considerably less scaling than the control concrete.

(5) During the short term (up to 42 cycles) CSF reduces the weight loss and the rate of reduction in the pulse velocity. Although little expansion takes place, this is found to increase with increasing CSF.

(6) Significant loss in weight takes place during extended exposure to freezing and thawing, but CSF remains to be beneficial in this respect. The loss in weight is accompanied by an increase in expansion.

(7) Long term exposure leads to significant reductions in the pulse velocities, indicating considerable internal damage. This deterioration is also reflected by the observed reductions in the relative dynamic moduli of elasticity.

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