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LONG-TERM EFFECT OF SILICA FUME ON THE PRINCIPAL PROPERTIES OF LOW-TEMPERATURE-CURED CERAMICS

Bertil Persson
Lund Institute of Technology, Division of Building Materials, Lund University,
Lund, Sweden

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ABSTRACT

This article outlines an experimental and numerical study of the long-term interaction between silica fume and Portland cement in low-temperature-cured ceramics such as concrete subjected to air, water and sealed curing. For this purpose about 2000 kg of eight qualities of concrete were studied at 4 different ages, each over a period of 90 months. Half of the concretes contained 10% silica fume. Parallel studies of strength, heat of hydration, hydration and internal relative humidity were carried out. New and original results and analyses of the interaction between Portland cement and silica fume related to compressive strength, split tensile strength, hydration and internal relative humidity are presented. The project was carried out between 1989 and 1996. © *1997 Elsevier Science Ltd*

Introduction

Reports have been presented over the last few years dealing with the decrease of strength in low-temperature-cured ceramics such as concrete over time due to content of silica fume (1). Most of the observations have been explained by different moisture conditions in the concrete when the compressive tests were carried out (2). The decrease of split tensile strength compared to compressive strength in a concrete with silica fume has been related to the pronounced basic (autogenous) shrinkage that occurred in a concrete with silica fume (3). Finally, the development of hydration differs substantially between concretes with and without silica fume (4). To complete the pozzolanic reaction between silica fume and Portland cement about 16% silica fume is required calculated on the basis of the reacted amount of cement (5). At low water-cement ratio, $w/c < 0.30$, some cement is left unreacted which thus means that a surplus of silica fume exists to complete the pozzolanic reaction provided that 10% silica fume was added as calculated from the cement content. Since no additional water is consumed during the pozzolanic reaction, no additional chemical shrinkage occurs (6). The additional autogenous shrinkage is explained by the extended depression in the pore water when silica fume is used (6).

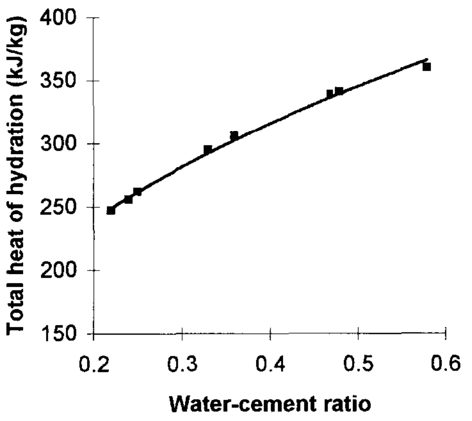

FIG. 1.
Total heat of hydration versus w/c.

Previous Research on the Effect of Silica Fume on Properties of Low-Temperature-Cured Ceramics

Hydration

An attempt has been made to estimate the ultimate heat of hydration and the rate of hydration of low-temperature-cured ceramics (concrete) as affected by w/c, Figure 1, (7, 8). The following correlation was obtained for total heat of hydration, Q (kJ/kg):

$$Q = 445\cdot(w/c)^{0.4} \quad (1)$$

Superplasticiser, s.p., is normally required when silica fume, s.f., is added to the concrete, especially at low w/c. Addition of s.p. affects hydration and temperature rise. Figures 2 and 3 show the retarding effect (9, 10). Figure 4 shows a substantial delay of rate of heat of hydration due to the effect of the added s.p. (11).

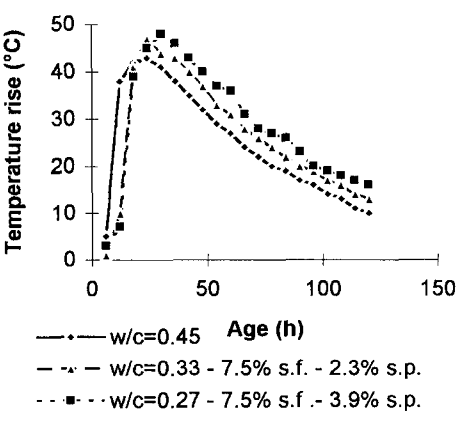

FIG. 2.
Temperature rise versus age.

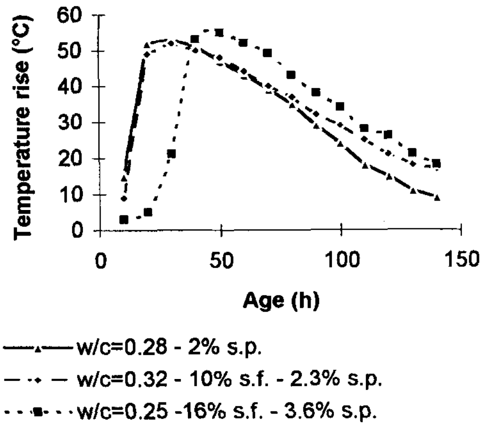

FIG. 3.
Temperature rise versus age.

Strength

The efficiency factor of silica fume, k_s, on properties of concrete is defined in Eq. 1, (4). Figure 5 shows efficiency factors around $k_s = 7$ when 5–10% silica fume was added to the concrete (12), according to literature and experiments.

$$(w/c)_{effective} = w/(c + k_s \cdot s) \qquad (2)$$

c denotes the cement content (kg/m^3)
k_s denotes the efficiency factor of silica fume according to equation (2)
s denotes the cement content (kg/m^3)
w denotes the cement content (kg/m^3)

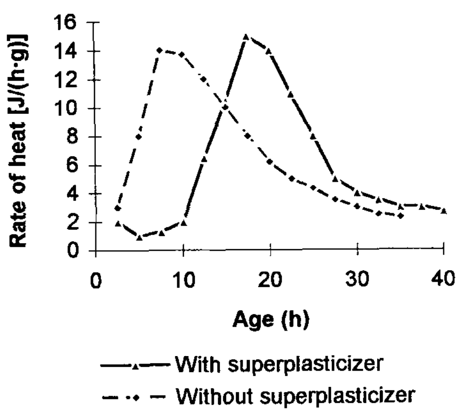

FIG. 4.
Rate of heat versus age.

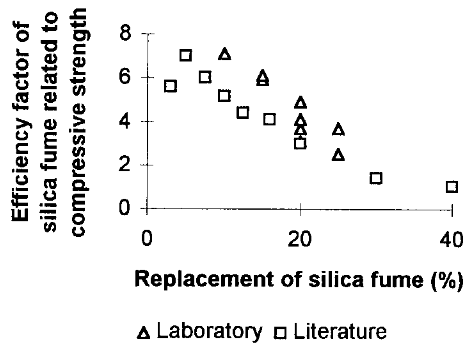

FIG. 5.
Efficiency factor of silica fume versus replacement of silica fume.

Especially when the addition of silica fume, s.f., was supplemented by quartzite filler, q.f., the efficiency factor of silica fume related to the strength, k_S, of the concrete became high. Figure 6 shows strength versus w/c of concrete with and without s.f. (13).

Experimental

Material and Preparation of Specimens

Table 1 shows the chemical composition of the cement (4). Table 2 shows the number of specimens that were studied, in all 1854 specimens. Table 3 shows the mix proportions of the tested concretes (4). Figure 7 gives the grading curves of all material in the fresh concrete, i.e. a linear-logarithmic distribution of particles. The low-temperature-cured ceramic (concrete) was poured in the shape of a disc, 1 m in diameter and 0.1 m thick. The surface was sealed with 2 mm epoxy resin. The specimens consisted of drilled cores with a diameter of 40 mm and a length of 80 mm. The ends of the cores were carefully ground before testing. The heat of hydration was studied on 50 kg concrete in the shape of a cylinder cast in a remaining mould of steel-sheeting (14).

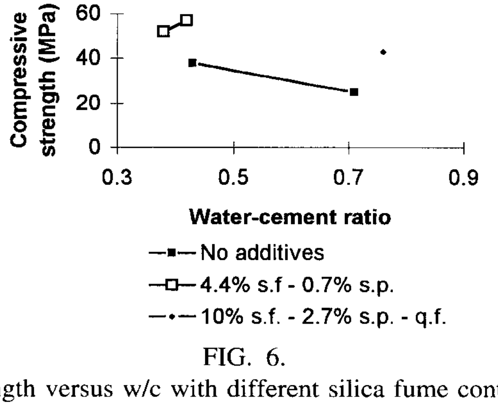

FIG. 6.
Strength versus w/c with different silica fume content.

TABLE 1
Chemical Composition of Cement

Analyzed properties (%):	
CaO	64.6
SiO_2	21.8
Al_2O_3	3.34
Fe_2O_3	4.39
MgO	0.84
K_2O	0.62
Na_2O	0.07
Alkali	0.48
SO_3	2.23
CO_2	0.14
Free CaO	1.13
Mineralogical properties (%):	
C_2S	22.5
C_3S	53.0
C_3A	1.42
C_4AF	13.4
Physical properties:	
Ignition losses	0.63%
Blaine	325 m^2/kg
Density	3180 kg/m^3

Methods

About 900 cores were studied related to strength. During testing interlayers of hardboard were used. About 230 measurements of ∅ in the concrete were carried out. High-capacity probes were used. The measurement period was 22 h. The probes were calibrated according to ASTM E 104-85 (15) within 5 days from the measurement. Hydration was studied by 648 measurements of weight losses during ignition between 105°C and 1050°C. Compensation was made for losses of weight of the different materials (16). The heat of hydration was obtained by semi-adiabatic experiments (17).

TABLE 2
Number of Measurements (m = months)

Parameter	1 m	3 m	5 m	15 m	90 m
f_c	144	144	72	144	144
f_{sp}	72	72	–	72	72
Hydration	144	144	72	144	144
∅	72	72	–	72	18
Total	432	432	180	432	378

TABLE 3
Mix Proportions of Tested Concretes (kg/m^3 Dry Material)

Littera	1	2	3	4	5	6	7	8
Quartzite sandstone 8–12 mm	1358	1306	1306	1214	1158	1150	1153	1145
Natural gravel 0–8 mm	525	630	549	723	730	846	825	812
Cement, c, low-alkaline, 325 m^2/kg	484	456	476	400	389	303	298	299
Silica fume, s, granulated, 17.5 m^2/g	48	–	48	–	39	–	30	–
Superplasticiser, naphthalene sulphonate	13.3	8.84	7.78	3.35	3.07	3.01	2.13	–
Water-cement ratio, w/c	0.22	0.25	0.24	0.33	0.36	0.47	0.48	0.58
Air content (%)	0.95	1.5	0.8	1.4	1.1	1.1	0.95	0.75
28-day strength (cylinder, MPa)	112	96	115	74	92	56	66	38
90-day strength (cylinder, MPa)	133	107	124	90	103	66	77	45
450-day strength (cylinder, MPa)	143	130	142	96	106	75	78	49

Result and Analyses

Self-Desiccation, ∅

Figure 8 shows the self-desiccation, ∅, during sealed curing as a function of the water-cement ratio (18). The results coincided well with research carried out by others (19–21). Figure 9 shows the efficiency factor (estimated according to Eq. 2) of silica fume related to self-desiccation, k_{se}, versus the water-cement ratio. Figure 9 gives a calculation of the efficiency factor related to self-desiccation (22):

$$k_{se} = 17.2\cdot(0.004\cdot t - 1)\cdot(w/c) - 0.026\cdot t + 9.2 \ \{\text{limits of age: } 28 < t < 450 \text{ days}\} \quad (3)$$

k_{se} denotes the efficiency factor at self-desiccation

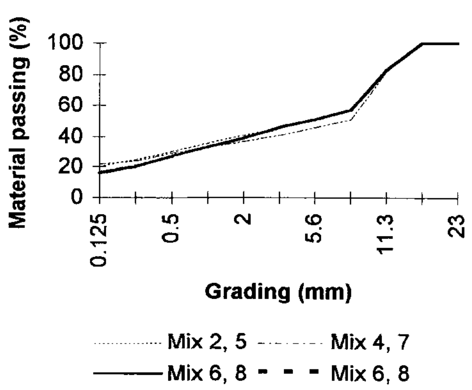

FIG. 7.
Grading of particles in fresh concrete.

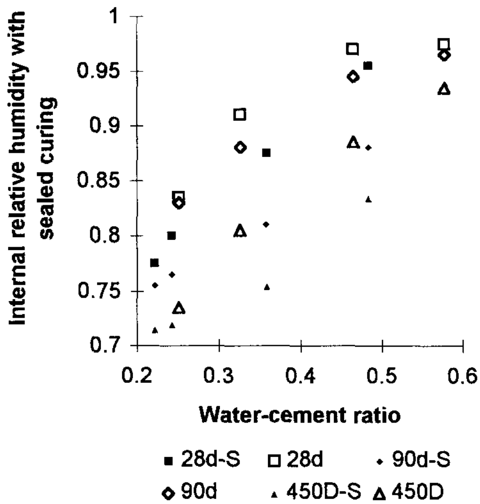

FIG. 8.

∅ after self-desiccation versus w/c. Symbols: d = days' age; S = 10% silica fume.

t denotes age (days)
w/c denotes the water-cement ratio

Compressive Strength

Figure 10 shows the compressive strength of concrete with sealed curing as a function of the water-cement ratio (4). Figure 11 shows the efficiency factor, k_{sc}, related to compressive strength as a function of the water-cement ratio (4). From Figure 11 the following correlation was calculated:

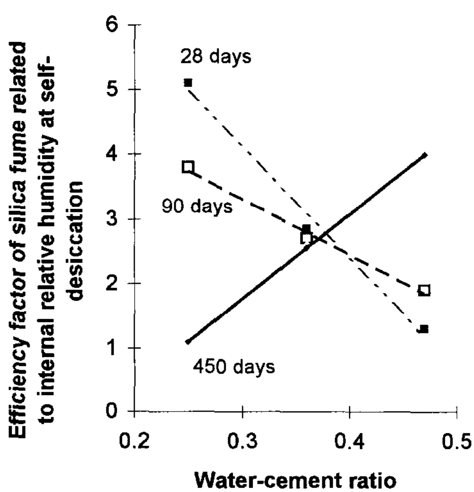

FIG. 9.

Efficiency factor of 10% silica fume related to self-desiccation. ∅ = relative humidity.

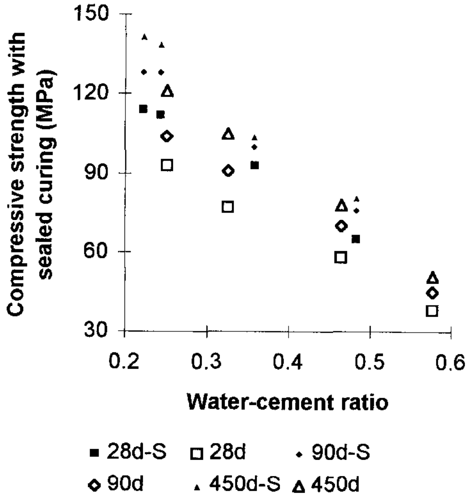

FIG. 10.

Compressive strength versus w/c. Symbols: d = days' age; S = 10% silica fume.

$$k_{sc} = 112 \cdot t^{-0.40} \cdot e^{(0.00073 \cdot t - 5.23) \cdot w/c} \; \{\text{limits of age: } 28 < t < 450 \text{ days}\} \qquad (4)$$

e denotes the exponential natural logarithm

k_{sc} denotes the efficiency factor related to compressive strength

t denotes age (days)

w/c denotes the water-cement ratio

The efficiency factor, k_{sc}, related to compressive strength decreased substantially over time. Tests at seven years' age have shown an even lower efficiency factor, k_{sc}, for silica fume. After 7 years the efficiency of silica fume on compressive strength became zero or slightly

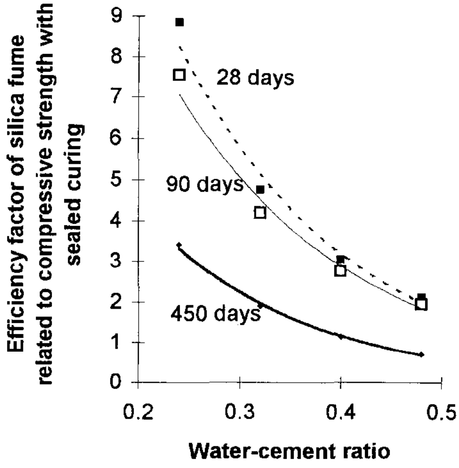

FIG. 11.

Efficiency factor of 10% silica fume related to compressive strength. The age is indicated.

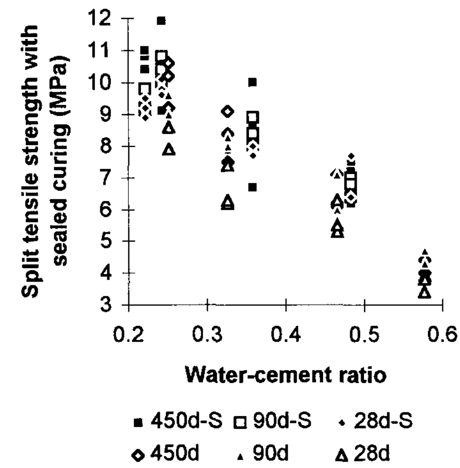

FIG. 12.
Split tensile strength versus w/c. Symbols: d = days' age; S = 10% silica fume.

negative (23). The reason for this was probably the self-desiccation of the concrete that was pronounced at early age in concretes with silica fume (3), thus almost stopping the hydration.

Split Tensile Strength

Figure 12 shows the split tensile strength with sealed curing as a function of the water-cement ratio (4). Figure 13 shows the efficiency factor of silica fume related to split tensile strength, k_{st} as a function of the water-cement ratio. Between 1 month's and 3 months' age a sudden drop of the efficiency factor of silica fume related to split tensile strength was observed. This observation most probably was related to a pronounced autogenous shrinkage that occurs in concretes with silica fume (3, 20, 21). The autogenous shrinkage exceeded the ultimate

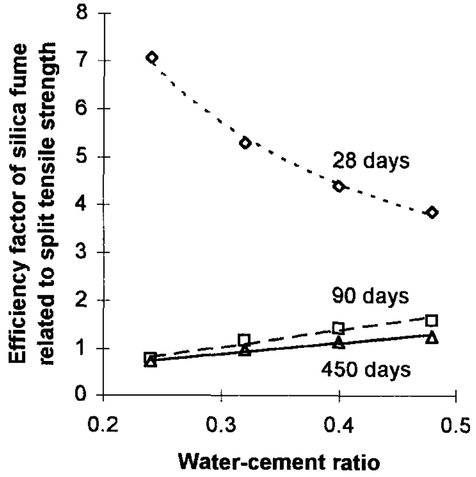

FIG. 13.
Efficiency factor of 10% silica fume related to split tensile strength.

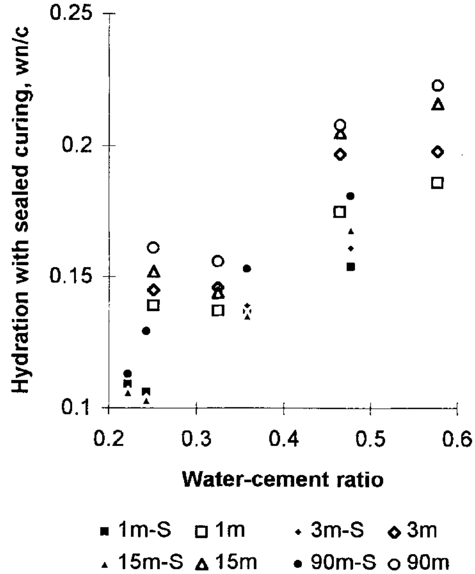

FIG. 14.
Hydration versus w/c. Symbols: m = months' age; S = 10% silica fume.

tensile strain of the concrete which most probably caused internal cracking (3). This assumption was confirmed by field observations (1). From Figure 13 the following correlations were calculated:

$$k_{st} = 12.4 \cdot e^{-2.5 \cdot w/c} \; \{t = 28 \text{ days}\} \tag{5}$$

$$k_{st} = (3.6 - 0.0031 \cdot t) \cdot (w/c) + 0.0005 \cdot t \; \{\text{limits of age: } 90 < t < 450 \text{ days}\} \tag{6}$$

After 15 months' age the efficiency factor of tensile strength remained at $k_{st} = 1$, at least until 90 months' age (23).

e denotes the exponential natural logarithm
k_{st} denotes the efficiency factor of silica fume related to split tensile strength
t denotes age (days)
w/c denotes the water-cement ratio

Hydration

The hydration of concrete related to w/c differed greatly between concretes with and without s.f., Figure 14. A great part of the calcium hydroxide in the concrete was consumed in the pozzolanic reaction. The efficiency factor of 10% silica fume, k_{wn}, related to hydration thus became negative, Figure 15. From Figure 15 the following equation for the efficiency factor of 10% silica fume, k_{wn}, related to hydration was calculated:

$$k_{wn} = 0.043 \cdot (\ln(t) + 30) \cdot \ln(w/c) - 0.006 \cdot t \cdot (1 - 0.01 \cdot t) - 0.71 \; \{\text{limits of age: } 1 < t < 90 \text{ months}\} \tag{7}$$

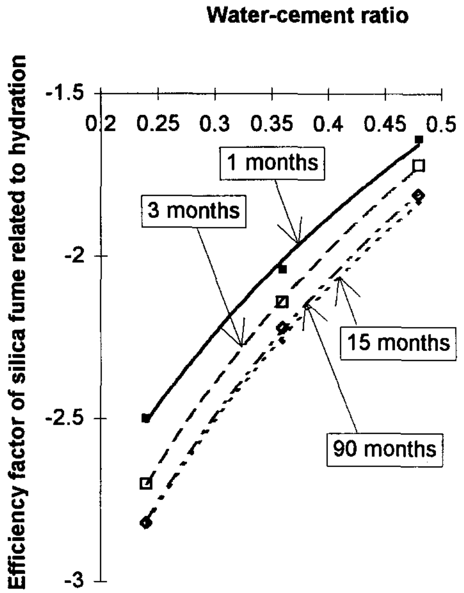

FIG. 15.
Efficiency factor of 10% silica fume related to hydration versus w/c.

k_{wn} denotes the efficiency factor of 10% silica fume related to hydration
ln(t) denotes the natural logarithm of the age of the concrete, t (months)
ln(w/c) denotes the natural logarithm of the water-cement ratio
t denotes age (months)
w/c denotes the water-cement ratio

Heat of Hydration

Figure 16 shows the heat of hydration versus time obtained in the experiments (14). The temperature rise was affected by w/c, addition of silica fume, s.f. and/or superplasticiser, s.p. (14) and cp., Figures 1–4 above. S.f. did not significantly affect the total heat shown in Figure 17. With the strength held constant, addition of 10% silica fume as calculated from the cement content did not affect the maximum temperature rise (of great importance related to the risk of cracking), Figure 18.

These results are of great interest from a practical point of view. As a consequence the addition of 10% silica fume to the concrete did increase the strength but did not increase the heat of hydration or the maximum temperature rise in the same size of construction. The following equation of the heat of hydration after 200 h, Q_{200}, was obtained (kJ/kg):

$$Q_{200} = 370 \cdot (w/c)^{0.37} \quad (8)$$

w/c denotes the water-cement ratio
Q_{200} denotes the heat of hydration after 200 h (kJ/kg)

Eq. 8 is similar to Eq. 1, cp. above.

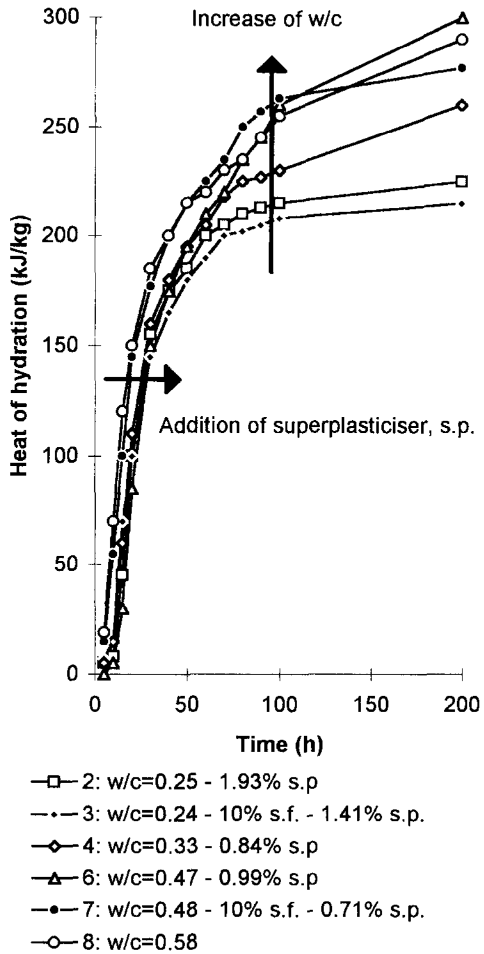

FIG. 16.
Heat of hydration versus time.

Summary and Conclusions

Based on the literature studies, on experimental studies related to hydration, structures and strength of about 1800 cores made of low-temperature-cured ceramics (concrete) and, finally on 8 semi-adiabatic tests on the heat release of concrete, the following conclusions were drawn:

1) Initially the effect of silica fume was very high on both self-desiccation and strength.
2) The effect of 10% silica fume as calculated from the cement content (estimated on the basis of concrete content) on self-desiccation decreased with w/c at 28 days' age (reversed effect at 450 days' age).
3) The efficiency factor of 10% silica fume as calculated from the cement content on strength decreased with w/c and with age.
4) The effect of 10% silica fume as calculated from the cement content on split tensile

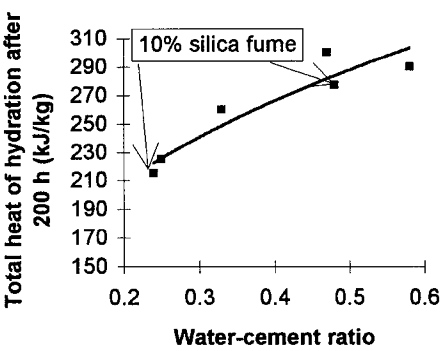

FIG. 17.
Heat of hydration versus w/c.

strength dropped after 28 days' age which may be an effect of the pronounced autogenous shrinkage that occurs in concrete with silica fume.

5) A negative efficiency factor of 10% silica fume as calculated from the cement content related to hydration was obtained since the calcium hydroxide in the concrete was consumed by the pozzolanic reaction.
6) Combined with superplasticiser, silica fume retarded the hydration process substantially.
7) The total heat of hydration in the concrete increased with w/c.
8) At a constant strength level the temperature rise was the same, with and without silica fume.

Acknowledgment

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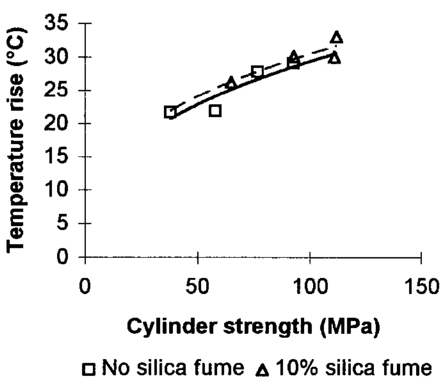

FIG. 18.
Maximum temperature rise.

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