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Improving silica fume cement by using silane

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Abstract

Silica fume cement was improved by using silane, which was introduced by either coating silica fume particles with silane or using silane as an admixture. Both methods of silane introduction similarly enhanced the workability, tensile strength and compressive strength, but the latter method resulted in lower compressive ductility, lower damping capacity, more drying shrinkage, lower air void content, higher density, higher specific heat and greater thermal conductivity, mainly due to the network of covalent coupling among the silica fume particles. © 2000 Elsevier Science Ltd. All rights reserved.

Keywords: Silica fume; Workability; Compressive strength; Tensile properties; Cement

1. Introduction

Silica fume is a very fine non-crystalline silica produced by electric arc furnaces as a by-product of the production of metallic silicon or ferrosilicon alloys. It is a powder with particles having diameters 100 times smaller than Portland cement, i.e., mean particle size between 0.1 and 0.2 μm. The SiO_2 content ranges from 85% to 98%. Silica fume is pozzolanic.

Silica fume used as an admixture in a concrete mix has significant effects on the properties of the resulting material. These effects pertain to the strength [1–8], modulus, ductility, vibration damping capacity [9–11], sound absorption [12], abrasion resistance [13–15], air void content [11,16], shrinkage [17–20], bonding strength with reinforcing steel [21–26], permeability [1,3,27–32], chemical attack resistance [33–36], alkali–silica reactivity reduction [37–40], corrosion resistance of embedded steel reinforcement [41–44], freeze–thaw durability [14,45], creep rate [46], coefficient of thermal expansion (CTE) [46], specific heat [46,47], thermal conductivity [46,47], and degree of fiber dispersion in mixes containing short microfibers [48–50]. In addition, silica fume addition degrades the workability of the mix [1,2,27,28,51]. Silica fume is becoming an increasingly common admixture in cement-based materials [52–55].

Surface treatment of silica fume with sulfuric acid prior to incorporation in a cement matrix results in mortars exhibiting increases in tensile strength by 12%, tensile modulus by 72%, tensile ductility by 57%, abrasion resistance by 20%, loss tangent by 30–80%, flexural storage modulus by 80–120%, and flexural loss modulus by 160–300%, relative to the values obtained by using as-received silica fume [56].

The use of silane coupling agents is common for enhancing the bond between a ceramic filler and a polymer matrix [57] because the epoxy structure at the end of the silane molecule allows coupling to an epoxy matrix. However, silane coupling agents are seldom used for enhancing the bond between a ceramic filler and a cement matrix [51]. In spite of the difference in chemical structure between cement and polymers, silane coupling agents are effective for both types of matrices. The effectiveness of silane for cement is due to the reactivity of its molecular ends with –OH groups and the presence of –OH groups on the surface of both silica and cement. Moreover, the hydrophilic nature of silane is expected to improve the workability of the mix and to enhance the dispersion of admixtures in the mix.

Silane can be introduced to silica fume cement in two ways: (i) as a coating on silica fume (i.e., coating the silica fume with silane prior to using the silica fume), and (ii) as an admixture (i.e., adding the silane directly into the cement

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mix). This work is an investigation of the effects of these two methods of silane introduction to silica fume cement and provides a comparison of the effects of these two methods of silane introduction.

2. Experimental methods

The cement used was Portland cement (Type I) from Lafarge (Southfield, MI). The silica fume (Elkem Materials, Pittsburgh, PA, EMS 965) was used in the amount of 15% by weight of cement. No aggregate (whether fine or coarse) was used for cement pastes. The fine aggregate used for mortars was natural sand (all passing #4 US sieve, 99.9% SiO_2); the particle size analysis of the sand is shown in Fig. 1 of Ref. [58]; no coarse aggregate was used; the sand/cement ratio was 1.0. The water/cement ratio was 0.35 for both cement pastes and mortars. A water-reducing agent (TAMOL SN, Rohm and Haas, Philadelphia, PA; sodium salt of a condensed naphthalenesulphonic acid) was used in the amount of 2% by weight of cement for cement pastes and in the amounts indicated for mortars.

The silane coupling agent used for coating silica fume was a 1:1 (by weight) mixture of Z-6020 ($H_2NCH_2CH_2NHCH_2CH_2CH_2Si(OCH_3)_3$) and Z-6040 ($OCH_2CHCH_2OCH_2CH_2CH_2Si(OCH_3)_3$) from Dow Corning (Midland, MI). The amine group in Z-6020 serves as a catalyst for the curing of epoxy and consequently allows the Z-6020 molecule to attach to the epoxy end of the Z-6040 molecule. The trimethylsiloxy ends of the Z-6020 and Z-6040 molecules then connect to the –OH functional group on the surface of silica fume. The silane was dissolved in ethylacetate. Surface treatment of silica fume was performed by immersing the silica fume in the silane solution, heating to 75°C while stirring, and holding at 75°C for 1 h, followed by filtration and drying. After this, the silica fume was heated in a furnace at 110°C for 12 h. The water-reducing agent was used in the amount of 1% by weight of cement for cement pastes with as-received silica fume and 0.2% by weight of cement for pastes with treated silica fume.

The silane used as an admixture was aqueous amino vinyl silane (Hydrosil 2781, Sivento, Piscataway, NJ), chosen due to its stability in aqueous systems. In contrast, the silane used for coating silica fume is not sufficiently stable in aqueous systems. The silane admixture was used in the amount of 0%, 0.20%, 0.50%, 0.75%, 1.0%, 1.5%, and 2.0% by weight of cement. The corresponding amount of water-reducing agent was 1.0%, 0.10%, 0.10%, 0.05%, 0.05%, 0.025%, and 0.025% by weight of cement, respectively. The more the amount of silane used, the less was the amount of water-reducing agent needed to maintain workability. The surfactant, used in the amount of 1% by weight of cement to help distribute the silane, was polyoxyethylene lauryl ether, $C_{12}H_{25}(OCH_2CH_2)_nOH$, from Aldrich Chemical, Milwaukee, WI. The defoamer (Colloids, Marietta, GA, 1010) was used in the amount of 0.13 vol.%.

A rotary mixer with a flat beater was used for mixing. The cement, silica fume, sand (if applicable) and water-reducing agent were mixed at a low speed. Water, silane (if applicable), surfactant (if applicable) and defoamer (if applicable) were mixed and then the mixture was added to the cement mix and mixed for 10 min at a high speed. After pouring into molds, an external vibrator was used to facilitate compaction and decrease the amount of air bubbles. The samples were demolded after 24 h and then cured in air at room temperature and a relative humidity of 100% for 28 days.

For compressive testing according to ASTM C109-80, cement paste specimens were prepared by using a 2 × 2 × 2 in. (5.1 × 5.1 × 5.1 cm) mold. Compression testing was performed using a hydraulic material testing system (MTS). The cross-head speed was 1.27 mm/min. Dog-bone-shaped specimens of the dimensions shown in Fig. 1 of Ref. [59] were used for tensile testing. They were prepared by using molds of the same shape and size. Tensile testing was performed using a screw-type mechanical testing system (Sintech 2/D). The displacement rate was 1.27 mm/min. During compressive or tensile loading up to fracture, the strain was measured by the cross-head displacement in compressive testing or by a strain gage in tensile testing. Six specimens of each composition were tested.

Dynamic mechanical testing (ASTM D4065-94) at controlled frequencies (0.2, 1.0 and 2.0 Hz) and room temperature (20°C) were conducted on cement pastes under flexure using a Perkin-Elmer Model DMA 7E dynamic mechanical analyzer. Measurements of tan δ and storage modulus were made simultaneously as a function of temperature at various constant frequencies. The heating rate was 2°C/min, which was selected to prevent any artificial damping peaks that may be caused by higher heating rates. The specimens were in the form of beams (120 × 8 × 3 mm) under three-point bending, such that the span was 115 mm. The loads used were all large enough so that the amplitude of the specimen deflection was from 6.5 to 9 μm (over the minimum value of 5 μm required by the equipment for accurate results). The loads were set so that each different type of specimen was always tested at its appropriate stress level. Six specimens of each type were tested.

The thermal conductivity (in W/m K) was given by the product of the thermal diffusivity (in cm^2/s), specific heat (in J/g K) and density (in g/cm^3). For measuring the thermal diffusivity, the laser flash method was used. In this method, a pulsed laser (Coherent General, MA) and a computer with Labtech software and data acquisition board were used. The cement paste specimen was in the form of a disc with diameter 13 mm and thickness 2 mm. Sample preparation for laser diffusivity measurement involved: (i) polishing both sides of the sample, (ii) coating both sides of the sample with gold for thermal contacts, and (iii) coating one of the sides (the side on which the laser beam would hit) with carbon (for avoiding reflection of the laser beam, since carbon is black). The temperature of the specimen at the side without carbon coating was measured after the laser flash as a function of time by using a thermocouple. From the temperature vs. time curve, the thermal diffusivity was calculated. Four specimens of each type were tested.

A Perkin-Elmer Differential Scanning Calorimeter (DSC-7) with UNIX Specific Heat Software was used for measuring the specific heat of cement pastes. A three-curve analysis method was used; it involved obtaining DSC sample, baseline, and reference material data. Sapphire was selected as a reference material. The specimen was in the form of a disc of diameter 6 mm and thickness 1 mm. Four specimens of each type were tested.

The density of cement pastes was measured by weight and volume (dimensions) measurements. Four specimens of each type were tested.

The air void content of cement pastes was measured by using ASTM method C185-95. Three specimens of each composition were tested.

Table 1
Workability of mortar mix

Silica fume	Water-reducing agent/cement (%)	Slump (mm)
Plain	0	[a]
With untreated silica fume	0	150
With untreated silica fume	1	186
With untreated silica fume	2	220
With treated silica fume	0	194
With treated silica fume	0.2	215
With silane and untreated silica fume	0	197
With silane and untreated silica fume	0.2	218

[a] Too large to be measured.

Drying shrinkage measurement was conducted on cement pastes as a function of curing age on samples of size 280 × 25.4 × 25.4 mm. A stainless steel contact point was positioned at each end along the length of each sample, such that the exterior tips of the two contact points were outside the sample and the interior tips of the two contact points were inside the sample and were separated by a distance of 250 mm, which was the gage length for drying shrinkage strain measurement. The measurement was conducted in accordance with ASTM Method C490-93a.

The workability (more exactly the consistency) of the mortar mixes was tested by measuring the slump in accordance with ASTM C143-90a method.

3. Results and discussion

3.1. Workability

Table 1 shows that the silane introduction using either method causes the silica fume mortar mix to increase in workability (slump). With silane (by either method of introduction) and no water-reducing agent, the workability is better than the mix with as-received silica fume and water-reducing agent in the amount of 1% by weight of cement. With silane (by either method) and water-reducing agent in the amount of 0.2% by weight of cement, the workability is almost as good as the mix with as-received silica fume and water-reducing agent in the amount of 2% by weight of cement.

3.2. Mechanical properties

As shown in Table 2 for cement pastes at 28 days of curing, the tensile strength, tensile ductility, compressive strength and compressive modulus are increased and the compressive ductility is decreased by the addition of silica fume (15% by weight of cement) which has not been surface-treated. The tensile strength and compressive strength are further increased and the compressive ductility is further decreased when silane-treated silica fume is used. On the other hand, the tensile modulus is essentially

Table 2
Mechanical properties of cement pastes at 28 days of curing

	Plain	With untreated silica fume[a]	With treated silica fume[a]	With silane[b] and untreated silica fume[a]
Tensile strength (MPa)	0.91 ± 0.02	1.53 ± 0.06	2.04 ± 0.06	2.07 ± 0.05
Tensile modulus (GPa)	11.2 ± 0.24	10.2 ± 0.7	11.5 ± 0.6	10.9 ± 0.5
Tensile ductility (%)	0.0041 ± 0.00008	0.020 ± 0.0004	0.020 ± 0.0004	0.021 ± 0.0004
Compressive strength (MPa)	57.9 ± 1.8	65.0 ± 2.6	77.3 ± 4.1	77.4 ± 3.7
Compressive modulus (GPa)	2.92 ± 0.07	13.6 ± 1.4	10.9 ± 1.8	15.8 ± 1.6
Compressive ductility (%)	1.72 ± 0.04	0.614 ± 0.023	0.503 ± 0.021	0.474 ± 0.015

[a] 15% by weight of cement.
[b] 0.2% by weight of cement.

Table 3
Dynamic flexural properties of cement pastes at a flexural (three-point bending) loading frequency of 0.2 Hz

	Loss tangent (tan δ, ±0.002)	Storage modulus (GPa, ±0.03)	Loss modulus (GPa, ±0.02)
Plain	0.035	1.91	0.067
With untreated silica fume[a]	0.082	12.71	1.04
With treated silica fume[a]	0.087	16.75	1.46
With silane[b] and untreated silica fume[a]	0.055	17.92	0.99

[a] 15% by weight of cement.

[b] 0.2% by weight of cement.

not affected by the silica fume addition. The use of both silane (0.2% by weight of cement) and untreated silica fume (as two admixtures, last column of Table 2) enhances the tensile strength, compressive strength and compressive modulus, but decreases the compressive ductility, relative to the paste with untreated silica fume and no silane. The effects of using treated silica fume (next to the last column of Table 2) and of using the combination of silane and untreated silica fume are quite similar, except that the compressive modulus is higher and the compressive ductility is lower for the latter due to the network of covalent silane coupling among the silica fume particles in the latter case.

The use of silane as an admixture which is added directly into the cement mix involves slightly more silane material but less processing cost than the use of silane in the form of a coating on silica fume. Both methods of silane introduction result in increases in the tensile and compressive strengths. The network attained by the admixture method of silane introduction does not result from the silane coating method, due to the localization of the silane in the coating, which nevertheless provides chemical coupling between silica fume and cement. The network, which is formed from the hydrolysis and polymerization (condensation) reaction of silane during the hydration of cement, also causes the ductility to decrease.

3.3. Vibration damping capacity

As shown in Table 3, the vibration damping capacity, as expressed by the loss tangent under dynamic three-point flexural loading at 0.2 Hz, is significantly increased by the addition of silica fume which has not been surface-treated. The use of silane-treated silica fume increases the loss tangent slightly beyond the value attained with untreated silica fume. The use of silane and untreated silica fume as two admixtures decreases the loss tangent to a value below that attained by using untreated silica fume alone, but still above that for plain cement paste.

The ability of silica fume to enhance the damping capacity is due to the large area of the interface between silica fume particles and the cement matrix and the contribution of interface slippage to energy dissipation. Although the pozzolanic nature of silica fume makes the interface rather diffuse, the interface still contributes to damping. The silane covalent coupling introduced by the silane surface treatment of silica fume can move during vibration, thus providing another mechanism for damping and enhancing the loss tangent. The network introduced by the use of silane and untreated silica fume as two admixtures restricts movement and therefore reduces the damping capacity relative to the case with untreated silica fume alone. Nevertheless, the use of the two admixtures enhances the damping capacity relative to the plain cement paste case, as even less movement is possible in plain cement paste.

The storage modulus (Table 3) is much increased by the addition of untreated silica fume, is further increased by the use of silane-treated silica fume, and is still further increased by the use of silane and untreated silica fume as two admixtures. The increase in storage modulus upon addition of untreated silica fume is attributed to the high modulus of silica compared to the cement matrix. The enhancement of the storage modulus by the use of silane-treated silica fume is due to the chemical coupling provided by the silane between silica fume and cement. The further enhancement of the storage modulus by the use of silane and untreated silica fume as two admixtures is due to the network of covalent coupling among the silica fume particles.

The loss modulus (Table 3) is the product of the loss tangent and the storage modulus. As vibration reduction requires both damping and stiffness, both loss tangent and storage modulus should be high for effection vibration reduction. Hence, the loss modulus serves as an overall figure-of-merit for the vibration reduction ability. The

Table 4
Drying shrinkage strain (10^{-4}, ±0.015) of cement pastes at 28 days

Plain	4.98
With untreated silica fume	4.41
With treated silica fume	4.18
With silane and untreated silica fume	4.32

Table 5
Air void content (%, ±0.02) of cement pastes

Plain	2.32
With untreated silica fume	3.73
With treated silica fume	3.26
With silane and untreated silica fume	3.19

Table 6
Density (g/cm^3, ±0.02) of cement pastes

Plain	2.01
With untreated silica fume	1.72
With treated silica fume	1.73
With silane and untreated silica fume	1.97

loss modulus is much increased by the addition of untreated silica fume and is further increased by the use of silane-treated silica fume. However, the use of silane and untreated silica fume as two admixtures decreases the loss modulus to a value below the paste with untreated silica fume alone, due to the decrease in the loss tangent. As a result, the use of silane-treated silica fume gives the highest value of the loss modulus.

3.4. Drying shrinkage

The addition of untreated silica fume to cement paste decreases the drying shrinkage (Table 4). This desirable effect is due to the prestressing effect of silica fume, which restrains the shrinkage. The use of silane-treated silica fume in place of untreated silica fume further decreases the drying shrinkage, due to the hydrophylic character of the silane-treated silica fume and the formation of chemical bonds between silica fume particles and cement. The use of silane and untreated silica fume as two admixtures also decreases the drying shrinkage, but not as significantly as the use of silane-treated silica fume.

3.5. Air void content and density

The air void content of cement paste (Table 5) is increased by the addition of untreated silica fume. Along with this effect is a decrease in density (Table 6). Both effects are related to the reduction in drying shrinkage. The introduction of silane by either method decreases the air void content, but the values are still higher than that of plain cement paste. The use of silane and untreated silica fume as two admixtures increases the density to a value almost as high as that of plain cement paste, due to the network of covalent coupling among the silica fume particles.

3.6. Specific heat

A high value of the specific heat is valuable for improving the temperature stability of a structure and to retain heat in a building. The specific heat (C_p, Table 7) is increased by the addition of untreated silica fume. The use of silane-treated silica fume in place of untreated silica fume further increases the specific heat, though only slightly. The effect of untreated silica fume is due to the slippage at the interface between silica fume and cement. The effect of the silane treatment is due to the contribution of the movement of the covalent coupling between silica fume particles and cement. The use of silane and untreated silica fume as two admixtures greatly increases the specific heat, due to the network of covalent coupling among the silica fume particles contributing to phonons.

Table 7
Specific heat (J/g K, ±0.001) of cement pastes

Plain	0.736
With untreated silica fume	0.782
With treated silica fume	0.788
With silane and untreated silica fume	0.980

Table 8
Thermal conductivity (W/m K, ±0.07) of cement pastes

Plain	0.53
With untreated silica fume	0.35
With treated silica fume	0.33
With silane and untreated silica fume	0.61

3.7. Thermal conductivity

Concrete of low thermal conductivity is useful for the thermal insulation of buildings. On the other hand, concrete of high thermal conductivity is useful for reducing temperature gradients in structures. The thermal stresses that result from temperature gradients may cause mechanical property degradation and even warpage in the structure. Bridges are among structures that tend to encounter temperature differentials between their top and bottom surfaces. In contrast to buildings, which also encounter temperature differentials, bridges do not need thermal insulation. Therefore, concrete of high thermal conductivity is desirable for bridges and related structures.

The thermal conductivity (Table 8) is decreased by the addition of untreated or silane-treated silica fume, due to the interface between silica fume particles and cement acting as a barrier against heat conduction. However, the thermal conductivity is increased by the use of silane and untreated silica fume as two admixtures, due to the network of covalent coupling enhancing heat conduction through phonons.

4. Conclusion

Two methods of silane introduction, namely silane in the form of a coating on silica fume particles and silane in the form of an admixture, were found to enhance the workability of silica fume mortar similarly and increase the tensile and compressive strengths of silica fume cement paste similarly. However, the latter method gives silica fume cement paste of lower compressive ductility, lower damping capacity, more drying shrinkage, lower air void content, higher density, higher specific heat, and greater thermal conductivity. These differences are mainly due to the network of covalent coupling among the silica fume particles in the latter case.

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