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Investigations on the performance of silica fume-incorporated cement pastes and mortars

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Abstract

Studies on the performance of cementitious products with silica fume (SF) are very important, as it is one of the inevitable additives to produce high-performance concrete (HPC). In this study, some experimental investigations on the influence of SF on various preliminary properties of cement pastes and mortars are reported. The properties included specific gravity and normal consistency (NC) of cement and air content and workability of mortar with different SF contents. Pozzolanic and chemical reactions of SF have been studied on setting times, soundness and shrinkage of cement pastes. Further, strength developments in compression and tension in cement mortars have also been studied at various SF contents. SF was varied from 0% to 30% at a constant increment 2.5/5% by weight of cement. Test results show that the SF changes the behavior of cement pastes and mortars significantly. It has been observed that the water–binder (w/b) (cement + SF) ratio seemed to play an important role for the performance of the products with higher SF contents. NC, soundness and drying shrinkage of cement pastes and the strength of mortar increase as the SF content increases, while the initial setting times of cement pastes and the air content and workability of mortar decrease as the SF content increases. However, hardly any influence has been observed on the final setting times of cement pastes. The early age hydration reactions of C_3A and C_3S increase with the addition of SF. The optimum SF content ranges between 15% and 22%.

Keywords: Air content; Cement paste; Mortar; Setting times; Silica fume; Soundness

1. Introduction

The structure of cement paste depends on the chemical composition, water–cement ratio, rate of hydration and the type of industrial admixtures. The significance of cement composition on its strength has been investigated extensively [1–3]. It appears that this effect is a rather complex phenomenon and varies with factors like mutual ratios of clinker materials, the amount and form of the added calcium sulfate and the presence of minor oxides. Use of silica fume (SF), as a cement replacement material, has been reported for producing high-performance concrete (HPC) [4]. It has been reported that SF in concrete/mortar is an efficient pozzolanic material, which results in more discontinuous and impermeable pore structure than that of the plain cement paste [5]. Addition of SF enhances the rate of cement hydration at early hours due to the release of OH^- ions and alkalis in to pore fluids. SF accelerates both C_3S and C_3A hydration during the first few hours [6]. It enables to provide nucleating sites to hydration products like lime, CSH and ettringite [7]. SF tends to affect the pattern of crystallization and degree of orientation of CH crystals at the aggregate surface during the first few days of cement hydration [8]. Calcium silicate hydrates (CSH) play a vital role in influencing the characteristics of cement paste. The compressive strength of cement pastes decreases as the SF content increases at low water–binder (w/b) ratios (0.25). However, plain cement pastes exhibit higher strength and greater strength development after 28 days [9]. At higher w/b ratio (0.45), with 30% SF, the pastes exhibit higher strength between 1 and 180 days.

The higher strength developments with SF are due to pore size refinement and matrix densification, reduction in content of $Ca(OH)_2$ (CH) and cement paste-aggregate interfacial refinement [9–11]. Hydration proceeds faster in pastes with SF due to both $Ca(OH)_2$ and nonevaporable water contents at the early ages of 3 and 7 days. However, hydration reactions in mortar terminate earlier. After 28 days, nonevaporable water content continuous to increase significantly in plain cement concrete [12]. However, the

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nonevaporable water content decreases in the age between 90 and 550 days [6,13]. It has been reported that the optimum SF content ranges between 15% and 20% [14,15]. Sabir [16] reported that the strength development in concrete with CSF is higher in the range of 12% to 28%. Lower compressive strengths have been reported at 3 days and higher strengths at 7 and 28 days with SF mortars [17].

2. Pozzolanic reaction of SF

As a pozzolana, SF reacts with calcium hydroxide (CH) liberated by the hydrolysis of C_3S and C_2S of Portland cement. The CH constitutes about 20–25% of the volume of the hydration products and CH crystals grow in solution. Due to their morphology, they are relatively weak, brittle and not cementitious. Cracks can easily propagate through regions populated by CH crystals, especially at the aggregate cement paste matrix interface. The percentage of CH consumed by SF is represented as an index of the pozzolanic activity of SF. It has been reported that due to high pozzolanic action of SF, about 25% of SF consumes most of the liberated $Ca(OH)_2$ at about 28 days.

The reaction process is given by the following equations:

$$2C_3S + 6H \rightarrow C_3S_2H_3 + 3CH \quad (1)$$

Portland cement

$$2C_2S + 4H \rightarrow C_3S_2H_3 + CH \quad (2)$$

SF

$$3CH + 2S \rightarrow C_3S_2H_3 \quad (3)$$

3. Experimental program

3.1. Materials

Ordinary Portland cement was used for the study. The physical properties of the cement are shown in Table 1. SF produced in the manufacturing of Ferro silicon alloy with the chemical composition as shown in Table 2 partially replaced the cement. Specific gravity and specific surface of the SF were 2.05 and 16,000 m^2/kg, respectively. Natural river sand was used with the fraction of sand passing through 1.18 mm sieve and retaining on 0.60 mm. The specific gravity and the bulk density of sand were 2.68 and 1584 kg/m^3, respectively. Potable water available in the laboratory was used. No water reducing agents were used.

Table 1
Physical properties of cement

Property	Result
Fineness of cement (%)	2.65
Normal consistency (%)	31.5
Setting times (min)	
a. Initial	135
b. Final	335
Specific gravity	3.168
Compressive strength (N/mm^2)	
a. @ 3 days	21.06
b. @ 7 days	30.14
c. @ 28 days	48.74

Table 2
Chemical composition of SF

Chemical compound	Result (%)
Silica (SiO_2)	84–86
Alumina oxide (Al_2O_3)	1.00
Iron oxide (Fe_2O_3)	2.0–3.5
Silica + Alumina + Iron oxide ($SiO_2 + Al_2O_3 + Fe_2O_3$)	87–90.5
Calcium oxide (CaO)	1.0–1.5
Loss on ignition	4–7

3.2. Preparation of test specimens

The normal consistency (NC) and setting times of cement pastes were determined using Vicat apparatus with different SF contents. Specific gravity of cement pastes containing various SF contents was investigated using Le Chatlier flask. Soundness/or expansions of cement pastes were determined by Le Chatlier method. Compressive strength of mortar was obtained on cube specimens of 70.7 mm at different ages. The water quantity was calculated by [(NC/4) + 3]% of combined weight of cement with SF and sand. The beam specimens, $40 \times 40 \times 160$ mm, were used for the determination of tensile strength of mortar with w/b ratio 0.50. Mortar bar specimens, $25 \times 25 \times 250$ mm, were used to determine the drying shrinkage of cement at the age of 28 days with w/b ratio 0.50. Workability of the mortar was determined using flow table test at w/b ratios 0.45 and 0.50. The cement–sand ratio in the mortars was 1:3 throughout the program. Table 3 shows different mixes, cement pastes or mortars with different SF contents used.

4. Experimental results and discussion

4.1. Specific gravity of cementitious material

Fig. 1 shows the variation of specific gravity of cement with SF. It demonstrates that the specific gravity of cement decreases as the SF content increases. It could be understood that the addition of lighter material to given cement changes the weight per unit volume. It has been noticed that the specific gravity of plain cement was 3.18 while it decreased to 2.78% at 30% SF. Using the experimental data from

Table 3
Mixes with different SF contents for the evaluation of various properties of cement pastes and mortars

S. No	Mix designation	SF (%)	Specific gravity[a]	Normal consistency[a] (%)	Air content[b] (%)	Workability[b]	Setting times[a]	Expansion[a]	Drying shrinkage[a]	Compressive strength[b]	Tensile strength[b]
1	MSF-0	0	◆	◆	◆	◆	◆	◆	◆	◆	◆
2	MSF-I	5	◆	◆	◆	◆	◆	◆	◆	◆	◆
3	MSF-II	10	◆	◆	◆	◆	◆	◆	◆	◆	◆
4	MSF-III	15	◆	◆	◆	◆	◆	◆	◆	◆	◆
5	MSF-IV	17.5	–	◆	–	◆	–	–	–	◆	◆
6	MSF-V	20	◆	◆	◆	◆	◆	◆	◆	◆	◆
7	MSF-VI	22.5	–	◆	–	◆	–	–	–	◆	◆
8	MSF-VII	25	◆	◆	◆	◆	◆	◆	◆	◆	◆
9	MSF-VIII	27.5	–	◆	–	◆	–	–	–	◆	◆
10	MSF-IX	30	◆	◆	◆	◆	◆	◆	◆	◆	◆

◆Indicates that the corresponding SF content was used for the mix.
[a] Cement paste.
[b] Mortar.

various cement pastes with different SF contents, an empirical equation (Eq. (4)) has been proposed to evaluate the specific gravity (S_c) with SF content. The regression coefficient is .985.

$$S_c = 3.144 - 0.013(\mathrm{SF}, \%) \quad (4)$$

4.2. Normal consistency

The amount of water required to produce a standard cement paste to resist a specified pressure is known as normal or standard consistency. In other words, it is the limit of water at which the cement paste resists the penetration of standard plunger (1-mm diameter) under a standard loading up to a distance of 5 or 7 mm from the base of Vicat apparatus. The consistency of cement depends on the type and its fineness. More water is required in cements with higher fineness values. As the SF is finer than the cement, the specific surface increases as the SF content increases. The standard consistency of pure cement paste was 31.50%; while at 30% SF, the consistency was 44.25%. Fig. 2 shows the variation of consistency of cement at different SF contents. It shows that the consistency of cement increases as the SF content increases. As much as 40% of additional water requirement was observed for cement pastes containing 20–30% SF. An equation (Eq. (5)) has been proposed to evaluate the NC of cement with SF. The regression coefficient is equal to .98.

$$\mathrm{NC} = 32.40 + 0.4(\mathrm{SF}, \%) \quad (5)$$

4.3. Air content of mortar

The durability of concrete depends upon the permeability of mortar. The amount of air present in the mortar influences the strength of the product. It has been reported that the strength decreases as the air content or permeability increases. Fig. 3 shows the variation of air content with SF content in mortar. It indicates that the air content decreases as the SF content increases. As the SF is finer than the cement particles, the finer particles of SF fill the gap between cement particles resulting in impermeable microstructure of the cement paste. The microfilling effect of SF is one of the important factors for the development of dense concretes with very high strength. At 0% SF, the air content was 2%, whereas it decreased to 1% with 30% SF. A small amount of air content significantly decreases the strength of mortar. Using the experimental data on various mortars, an equation (Eq. (6)) has been proposed

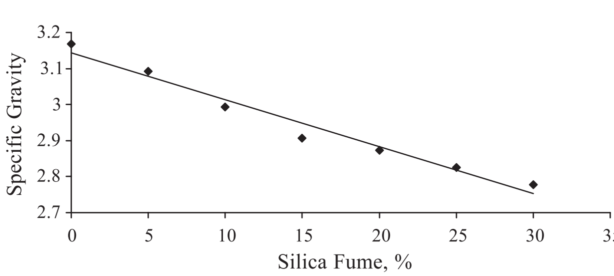

Fig. 1. Variation of specific gravity of cement with SF at different percentages.

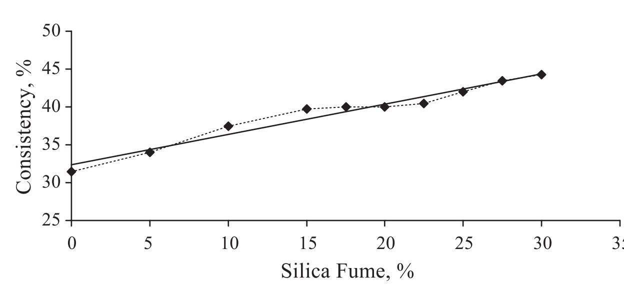

Fig. 2. Variation of consistency of cement pastes containing different percentages of SF.

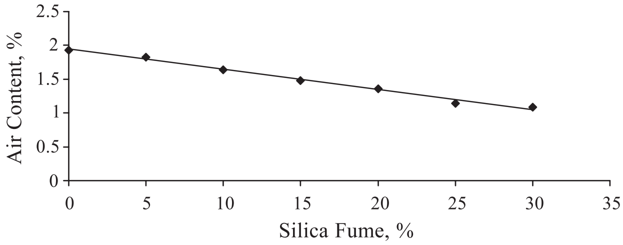

Fig. 3. Variation of air content in cement mortar with the addition of different SF contents.

to evaluate the air content (a_c) of mortars at different SF contents. The regression analysis shows a regression coefficient of .995.

$$a_c = 1.944 - 0.03(SF,\%) \quad (6)$$

4.4. Workability/flowability of mortar

Workability of mortar slightly decreases as the SF content increases. This has been due to the higher specific surface of SF, which needs more water for complete hydration and for workability. Fig. 4 demonstrates the variation of workability as percentage flow at w/b ratios 0.45 and 0.50 with different SF contents. The effect of condensed SF on the rheology of fresh mortar is generally viewed as a "stabilizing effect." In other words, the addition of very fine particles to a mortar/concrete tends to reduce segregation and bleeding tendencies. Without SF, the finest particles in mortar are those of Portland cement. Since the sand particles are bigger than the cement particles, the latter act as stabilizers by reducing the dimensions of channels through which bleed water rises to the surface of mortar. When very fine particles of SF are added to the mortar, the size of flow channels further reduced because these fine particles are able to adjust their positions to occupy the empty spaces between cement particles. Due to increase in the number of contact points between solid particles, the cohesiveness of mortar mixture greatly improved when SF is present. In fact, the presence of too much SF in mortar (>10% by weight of cement) tends to make the mixture stiff. The addition of small amounts of SF does not require the use of extra water or super plasticizers. However, with higher dosages of SF, the workability of mortar has been reduced. Fig. 4 shows the variation of workability as percentage flow on flow table at w/b ratios 0.45 and 0.50 with different SF contents.

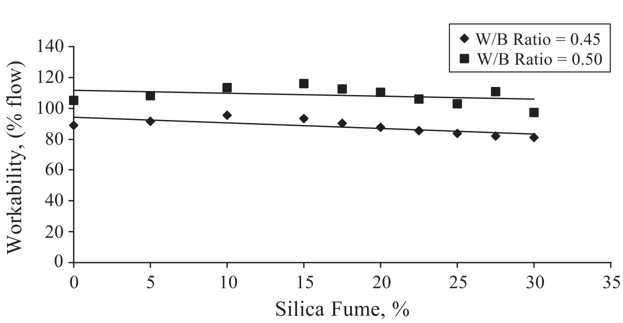

Fig. 4. Variation of workability of cement mortars with SF at different contents.

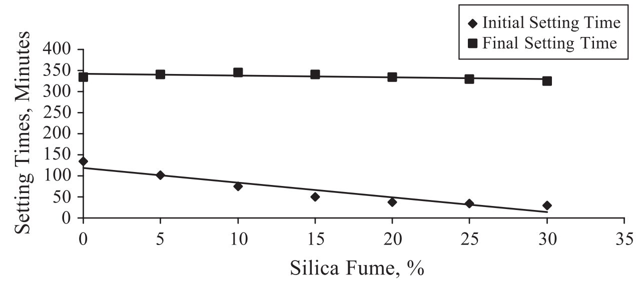

Fig. 5. Variation of setting times of cement pastes with different percentages of SF addition.

4.5. Setting times

The ability of cement to make fluid concrete to hardened state is represented by the time of setting. The fineness of the cement and its chemical composition influences the setting times of concrete. Fig. 5 shows the variation of setting times with the addition of SF in cement pastes. It has been observed that the initial setting time decreases as the SF content increases. At smaller contents, the setting times of cement paste did not affect much. However, at higher SF contents, the initial setting time significantly decreases. At 30% SF, the initial setting time has been only 30 min. It indicates that if higher contents were added, it would have been produced with cement pastes with false setting. The final setting times seem to be not influenced by the SF. The pozzolanic action of SF seems to be very active at early hours of hydration. Cheng Yi and Feldman [6] reported that SF accelerates the hydration of C_3A and C_3S reaction at the early hours of hydration. According to Swamy [18], the addition of SF in small amount (about 10% by weight of cement) to cement composites either has no significant effect or alter the time of set very slightly. Further, no significant changes have been observed in the initial setting times, whereas a remarkable influence of SF was observed on final set. However, in the present program, the final setting times were hardly influenced by the SF.

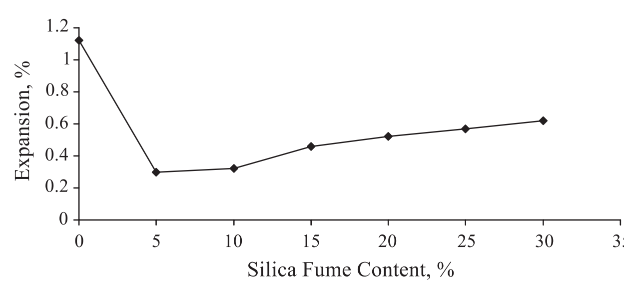

Fig. 6. Variation of soundness of cement pastes with SF content.

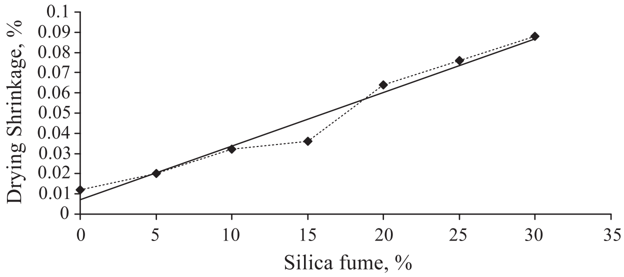

Fig. 7. Variation of drying shrinkage of mortar with SF content.

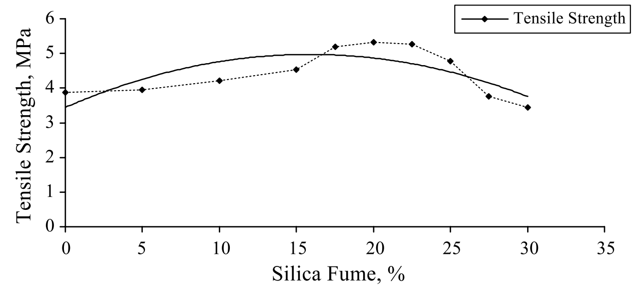

Fig. 9. Variation of tensile strength of mortar with SF.

4.6. Soundness of cement

The resistance against volume changes of cement is measured by the soundness. Fig. 6 shows the variation of soundness of cement paste as percentage expansion with SF content. It has been generally observed that the soundness of cement increases or the expansion decreases with the addition of SF. The pure cement paste exhibited very high expansion than those with SF. The cement paste obtained with SF shows very good resistance against volume changes. Fig. 7 illustrates the variation of drying shrinkage of mortar with SF in various mortars. It has been observed that drying shrinkage increases as the SF content increases. The data from drying shrinkage tests by many researchers show that the short-term drying shrinkage of cementitious composites increases with the addition of SF. But the test results on long-term drying shrinkage indicate that there has been no significant effect of SF addition. The mortars and concretes containing SF seem to experience with the same drying shrinkage in the long run as those did without SF. However, the presence of coarse aggregate in concrete could restrain the shrinkage stress produced with the addition of SF.

4.7. Strength of mortar

The strength of mortar with SF increases up to a certain SF content beyond which the strength was decreased. It shows that between 17.5% and 22.5%, higher strengths have been achieved at any age of mortar. Similar trends have been noticed in compression and tension. The variations of compressive strength of mortar at the age of 3, 7, 28 and 90 days with SF content are shown in Fig. 8. Fig. 9 shows the variation of tensile strength with SF content at the age of 28 days. It indicates that the optimum SF content to produce higher strength products seems to range between 17.5% and 22.5%. The optimum SF content ranges between 15% and 20% [14,15]. Recently, Sabir [16] reported that the strength development in concrete with CSF is higher with SF content in the range of 12% to 28%. The internal mechanism in the cementitious products can be understood through the following. Three mechanisms have been responsible for the strength enhancement by SF: (i) strength enhancement by pore size refinement and matrix densification, (ii) strength enhancement by reduction in the content of $Ca(OH)_2$ (CH) and (iii) strength enhancement by cement paste-aggregate interfacial zone refinement [9–11]. The transformation of large pores into finer pores, i.e., pore size refinement as a result of pozzalanic reaction, plays an important role in enhancing the strength of Portland cements. The presence of SF in the Portland cement mixes causes considerable reduction in the volume of large pores at all ages and is therefore instrumental in enhancing the compressive strength. But the exact mechanism by which the process of pore refinement takes place in SF Portland cement pastes is not completely understood. In the presence of SF, the pozzolanic reaction reduces the CH content, which leads to increase the strength. SF changes the orientation of CH crystals in the zone, resulting in less microcracking at the transition zone [8].

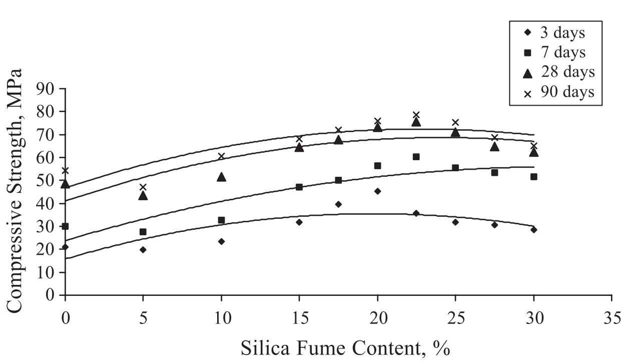

Fig. 8. Variation of compressive strength of mortars with SF content (%).

5. Conclusions

From the above observations, the following conclusions can be drawn on the influence of SF on various properties of cement pastes and mortars. Specific gravity, air content and workability decrease as the addition of SF increases. The SF seemed to be an efficient pozzolanic material. It activates the constituents of cement in the early hours of hydration. The air content has been reduced due to its microfilling effect, which may lead to increase the compressive strength. Quick setting of cement results with higher SF contents. The positive trends have been observed with SF on strength of mortars and soundness. The drying shrinkage of mortar increases as the SF content increases. The optimum SF content for achieving higher strength of mortars ranges between 15% and 22%. The results are different than those commonly attained, along with the additions of superplasticizer as the flowability of mortars gets affected significantly.

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