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# Influence of silica flour–silica fume combination on the properties of high performance cementitious mixtures at ambient temperature curing

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## HIGHLIGHTS

- Silica flour–Silica fume combinations improved the early age compressive strength.
- Increase in fineness of silica flour improved the early-age strength of HPCM.
- Combinations of silica fume and silica flour reduced permeability of HPCM.
- Higher fineness and dosage of silica flour increased drying shrinkage of HPCM.
- RCP and permeable voids of HPCMs showed a good correlation.

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## ABSTRACT

The compressive strength, chloride ion permeability, bulk density, volume of permeable voids, and drying shrinkage of high performance cementitious mixtures (HPCM) containing both silica fume (SFU) and silica flour (SFL) were studied under ambient-temperature curing. SFU was used as cementitious material in addition to cement; SFL was used as filler material replacing part of the fine aggregate. The use of SFL in combination with SFU increased the early-age compressive strength of HPCM, but decreased the later-age compressive strengths. Based on the results of both thermo-gravimetric analysis and loss on ignition tests, this phenomenon was attributed to the improved hydration of cementitious materials by SFL at early ages, but inhibited continued hydration at later ages. The addition of SFL combined with SFU resulted in further reduction of chloride ion permeability and the volume of permeable voids. The addition of SFL slightly increased the drying shrinkage. Among SFLs with different fineness, the finest SFL which densified the microstructure of HPCM most resulted in the highest drying shrinkage. For HPCM cured under ambient-temperature the use of SFU alone will suffice when used at sufficient dosages; and the addition of SFL along with SFU presents advantages mostly in improving the early-age compressive strengths.

## 1. Introduction

Silica fume (SFU) and silica flour (SFL) are common ingredients used in the formulations for high performance cementitious mixtures (HPCM) [1–7]. If cured at elevated temperature, sometime together with elevated pressure, HPCM utilizing SFU and SFL can present compressive strength over 150 MPa and superior durability [1–3]. HPCM with such high compressive strength is often referred to as ultra-high performance concretes (UHPC) [4–6].

SFU is a by-product of the production of elemental silicon or alloys containing silicon [7]. SFU can densify the microstructure of both the bulk paste and the interfacial transition zone (ITZ) between paste and aggregate of hardened cementitious mixture [7–10]. The micro-filler effect and the pozzolanic effect of SFU are believed to be responsible for the modified microstructure [7–10]. Accelerated pozzolanic reaction of SFU has been observed when cementitious mixture is cured at 90 °C [1].

SFL is produced by grinding quartz. Studies have shown that at a temperature of 250 °C, SFL with mean particle size of 10 μm can react with amorphous hydration products of Portland cement to form crystalline hydrates, such as xonotlite [1–3]. The resulting cementitious compounds with $CaO/SiO_2$ ratio equal to or less than 1.0 exhibited higher compressive strength and lower permeability

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[1–3]. However, SFL is an inert material under ambient temperature. Its fine particulate nature physically improves the gradation of the aggregate and reduces the permeability of concrete [11–14]. Moreover, some studies showed that these fine particles also promote the rate of cement hydration, and increase the early-age strength of cement paste [12–14]. In these studies, fine inert silica was incorporated into concrete either as mineral admixture replacing part of the cement, or as fillers replacing part of the fine aggregate. The test results under ambient temperature curing showed significantly higher compressive strength of mixture with proper amount of inert silica than the control mixture at early ages, but insignificant difference in compressive strength between them at later ages. Studies on the heat evolution of cement hydration found that the higher compressive strength at early ages was because of the accelerated hydration [14]. It was believed that the accelerated cement hydration in the presence of inert silica was due to the acceleration in the precipitation of portlandite [12–14]. Specifically, the particles of fine inert materials provided a large amount of substrates surface for portlandite to crystallize at early stages of cement hydration [12–14].

Significant research on the properties of HPCM has been conducted when SFU and SFL have been used together and cured at elevated temperatures [1–6]. The roles of SFU and SFL at elevated temperature have been well studied [1–3]. Regardless of the superior properties of HPCM at elevated temperature curing, researchers have recognized the need for ambient temperature cured HPCM which is usually mixed with steel fiber to develop UHPC [4–6]. UHPC having superior workability, mechanical and durability properties has been successfully prepared utilizing SFU and SFL at ambient temperature curing [6]. However, there is still limited knowledge on the mechanism by which the combinations of SFU and SFL impact the mechanical and durability properties of HPCM or UHPC at early and later ages.

Typically, UHPC mixtures contain reinforcing fibers, which significantly enhance the mechanical properties of the mixture. A previous study by the authors has revealed that the use of steel fibers can improve the measured compressive strength by about 20%, compared to the mixture without steel fibers [15]. However, to study the fundamental properties of the mortar phase of the UHPC, it is undesirable to include the fibers in the mixture. This investigation focuses on the effect of SFU and SFL combination under ambient temperature on the properties of HPCM without reinforcing fibers. In particular, the compressive strength, rapid chloride ion penetration (RCP), bulk density, volume of permeable voids, and drying shrinkage of HPCM containing SFU and SFL were investigated. The variables studied include SFU content, SFL content, and fineness of SFL. Thermo-gravimetric analysis (TGA) and loss on ignition tests (LOI) were conducted to investigate the effect of silica flour on the relative degree of hydration of HPCM.

## 2. Experimental program

### 2.1. Materials

For the experimental study, a Type III Portland cement meeting ASTM C150 specification was used. The cement had $C_3S$, $C_2S$, $C_3A$ and $C_4AF$ of 58%, 15%, 7% and 11%, respectively. A low LOI (0.22%) SFU with an off-white color was used as a supplementary cementitious material. Three types of SFL with different fineness (denoted as SFL1, SFL2 and SFL3) were used as filler materials, substituting part of the fine aggregate. Table 1 lists the physical and chemical properties of cement, SFU and SFL. The fine aggregate used in this study was a natural siliceous sand meeting the ASTM C33 gradation specification. The gradation of fine aggregate is shown in Table 2. The specific gravity, water absorption, and fineness modulus of the fine aggregate were 2.62%, 0.30%, and 2.65%, respectively. The LOI of sand is 0.445%. The high-range water-reducing admixture (HRWRA) was a polycarboxylic ether in powder form, therefore needing no adjustments in the final mix water content to account for any water addition resulting from the use of liquid admixtures.

**Table 1**
Physical and chemical properties of materials.

| Materials name | Type III cement | SFU | SFL | | |
|---|---|---|---|---|---|
| | | | Min-U-Sil 5 (SFL1) | Min-U-Sil 30 (SFL2) | Sil-Co-Sil (SFL3) |
| Specific gravity | 3.15 | 2.2∼2.3 | 2.65 | 2.65 | 2.65 |
| Specific surface area ($m^2$/kg) | 543[a] | 20,000[b] | 5000[b] | 1900[b] | 910[b] |
| Particle size (μm) | – | 0.15[c] | 1.6[d] | 8.0[d] | 40–250[e] |
| Passing 325 mesh (%) | 98.8 | 99.8 | 99.996 | 99.7 | 79% |
| LOI (%) | 1.342 | 0.22 | 0.443 | – | – |
| $SiO_2$ (%) | 20.4 | 92.0 | 99.2 | 99.5 | 99.5 |
| $Fe_2O_3$ (%) | 3.5 | – | 0.035 | 0.024 | 0.022 |
| $Al_2O_3$ (%) | 5.0 | – | 0.3 | 0.3 | 0.3 |
| $TiO_2$ (%) | – | – | 0.02 | 0.01 | 0.01 |
| CaO (%) | 64.4 | <1.0 | 0.03 | 0.02 | 0.02 |
| MgO (%) | 1.0 | – | 0.01 | 0.01 | 0.01 |
| $Na_2O_{eq.}$ (%) | 0.49 | – | 0.02 | 0.02 | 0.02 |
| $SO_3$ (%) | 3.5 | <2.0 | – | – | – |

[a] Blaine's surface area.
[b] BET-Gas adsorption surface area.
[c] Average particle size.
[d] Medium particle size.
[e] Maximum particle size.

**Table 2**
Gradation of fine aggregate.

| Sieve | Percent passing |
|---|---|
| 9.5-mm | 100.0 |
| 4.75-mm | 99.8 |
| 2.36-mm | 97.1 |
| 1.18-mm | 82.0 |
| 600-μm | 41.9 |
| 300-μm | 14.0 |
| 150-μm | 0.5 |
| 75-μm | 0.1 |

### 2.2. Mixture proportions and specimens preparation

The proportions of the materials in the mixtures were designed to study the effect of SFU and SFL combination on the compressive strength and the durability of HPCM. Table 3 shows the relative proportions of the ingredients in each of the HPCM mixtures.

As this table shows, the first HPCM is the control which does not contain SFU or SFL, and its w/cm is 0.225. The next three HPCMs, SFU-10%, SFU-20% and SFU-30% have SFU content at 10%, 20%, and 30% by weight of cement, respectively, but do not contain SFL. It should be noted that SFU was used in addition to cement and not as a replacement of cement in this study, and the w/cm of HPCMs was held constantly at 0.225. The effect of SFU addition on the properties of HPCMs was determined by comparing the performance of these HPCMs with that of the HPCM C-0.225. The remaining 5 HPCMs contain both SFU and SFL, with the content of former (SFU) held constant at 10% by weight of cement in all of them. In order to study the effect of SFL content on the properties of HPCM, three mixtures that contained SFL1 were designed and identified as SFL1-10%, SFL1-20% and SFL1-30%. These mixtures contained SFL1 at a dosage of 10%, 20% and 30% by mass replacement of fine aggregate. To study the effect of fineness of SFL at a given content, HPCM mixtures SFL1-20%, SFL2-20% and SFL3-20% were designed, wherein the SFL content was fixed at 20% by mass replacement of fine aggregate. However, the fineness of SFL was varied as indicated by the use of three types of SFL, i.e. SFL1, SFL2 and SFL3, with SFL1 being the finest of the three SFLs and SFL3 being the coarsest. The w/cm of all HPCM mixtures with SFL and SFU were held constant at 0.225. The mass ratio of filler materials to cementitious materials was 1.25 throughout the study. In order to have fresh mixtures with at least 150% flow in accordance with ASTM C1437, the dosage of HRWRA was kept at 1.25% by weight of cementitious materials. Table 4 shows the detailed proportions for 1 $m^3$ of each of the HPCMs investigated in this study.