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Influence of high volumes of silica fume on the rheological behavior of oil well cement pastes

A B S T R A C T

Specialized classes of concrete, such as ultra-high performance concrete, use volumes of silica fume in concrete that are higher than those in conventional concrete, resulting in increased water demand and mixing difficulty. This study considered the effects of eight different silica fumes in three dosages (10%, 20%, 30%) with three w/b (0.20, 0.30, 0.45) on rheological behavior as characterized by the Herschel-Bulkley model. Results indicated that the specific source of silica fume used, in addition to dosage and w/b, had a significant effect on the rheological behavior. As such, all silica fumes cannot be treated as equivalent or be directly substituted one for another without modification of the mixture proportion. The rheology of cement pastes is significantly affected by the physical properties of silica fume more so than any chemical effects.

1. Introduction

Silica fume is a very fine pozzolanic material composed primarily of amorphous silicon dioxide (SiO_2), which is a byproduct of the ferrosilicon industry [1]. Silica fume has been reported to improve the properties of cementitious materials since the 1950s [2,3]. Much of the published research concerning the use of silica fume focuses on the effects of silica fume on the hydration of cementitious materials, with different researchers suggesting that the beneficial influences of silica fume are due to both physical [4] and chemical effects [5,6]. Other researchers have shown silica fume to be beneficial in reducing alkali-silica reactions [7] and calcium oxychloride production and damage [8].

In general, most concretes that contain silica fume have dosages between 5% and 10% by mass of cement [2]. Much higher dosages of silica fume are being used in more specialized classes of concrete, such as ultra-high performance concrete (UHPC). These classes of concrete have had several names, many of which can be loosely grouped under the UHPC umbrella including high-performance fiber reinforced cement composite (HPRCC), reactive powder concrete (RPC), and ultra-high performance fiber-reinforced concrete (UHPFRC) [9]. Russell and Graybeal [9] showed mixture proportions for six different UHPCs, with silica fume representing 20–30% of the total binder used. This increase in the amount of silica fume used presents new challenges that have yet to be fully investigated. While increased silica fume dosages improve mechanical performance in materials such as UHPC, the detrimental effects of silica fume, such as increased water demand and mixing difficulties, have also become more pronounced.

Previous research [10] has shown that differences can exist when multiple silica fume sources are used. This can result in radical changes in composite performance of UHPC depending on the silica fume source. In that study, some silica fumes could not be successfully incorporated into the UHPC baseline matrix. This inability to adequately mix these sources into the UHPC baseline matrix suggested that the choice of silica fume had a significant effect on the rheological properties of a cementitious material.

Many researchers previously studied the effects of silica fume on concrete rheology. Plank et al. [11] showed that certain polycarboxylate superplasticizers were not compatible with some silica fumes. Vikan and Justnes [12] studied the effects of silica fume dosages up to 14% on cementitious pastes and showed that sodium naphthalene superplasticizers actually had an adverse effect on pastes containing silica fume. They also showed that the rheology of cement paste could be correlated with the chemical and physical properties of cement, while suggesting that those findings could be extended in cement pastes with constant silica fume dosage [13].

Senff et al. [14] showed that the relationship between the amount of silica fume, the superplasticizer dosage, and the flow properties were not linearly related and were better described using the Herschel-Bulkley rheological model. Mei et al. [15] also suggested using the Herschel-Bulkley model and showed that low dosages (less than 8%) of silica fume had little effect on concrete slump, whereas higher dosages resulted in a more pronounced effect. Ferraris [16] showed that the Bingham plastic model was widely used to describe the rheology of traditional concrete, but that more advanced models, such as the Herschel-Bulkley model, were necessary to describe the rheology of more specialized classes of concrete.

Shahriar and Nehdi [17] showed that different chemical admixtures affect the rheological behavior of neat oil well cement slurries differently depending on temperature and w/c. Additional rheological models have been developed to better describe the rheological behavior of oil well cement slurries containing various mineral nanoparticles [18,19].

The vast majority of the published literature focuses on the effects of a single silica fume source on the performance of a cementitious mixture. The results are then used to draw conclusions as to how all silica fumes might affect mixture performance. There is a knowledge gap in the literature as to whether various silica fume sources similarly affect cement paste rheology, especially at high silica fume dosages (more than 10% of total binder). The work presented herein addresses this knowledge gap by answering the following questions:

- Does changing silica fume sources affect the rheological characteristics of a cement paste?
- If so, does this effect become more pronounced at high silica fume dosages?
- Does densification affect the rheological characteristics of a cement paste?
- Can raw material properties for various silica fumes be used to predict the changes in rheological characteristics of a cement paste?

2. Materials

2.1. Constant mixture components

A Class H cement (American Petroleum Institute [API]) was used as the primary cementitious material in this study. Class H cement was chosen because it is commonly used in ultra-high strength and ultra-high performance cementitious composites. Due to low tricalcium aluminate (C_3A) content and coarser grain size, Class H cement reacts more slowly than traditional Type I

portland cement, which increases the amount of calcium hydroxide (CH) available in solution for pozzolanic reaction with silica fume.

Two liquid components were used in this study: distilled water and a high-range water reducing admixture (HRWRA). Distilled water was chosen to minimize the effects of traditional tap water minerals on the rheology of cement paste. A commercially available HRWRA specifically design for use with low water-to-cement (w/c) materials was used for this study. The manufacturer's recommended dosage was 1–5% HRWRA by mass of cementitious materials. To fit within these recommendations, 3% HRWRA by mass of combined cementitious materials (cement and silica fume) was chosen as a constant HRWRA dosage rate.

2.2. Silica fumes

Eight silica fumes were chosen for the study as shown in Table 1. All eight are commonly used in ultra-high strength and ultra-high performance cementitious composites. It is hypothesized that the use of silica fume will increase the amount of calcium silicate hydrate (C-S-H) produced through pozzolanic reaction and refine the pore structure of the composite through physical filler effects. Although commercially marketed as silica fumes, three of the eight tested fumes failed to meet ASTM C1240 [20], due to a low silicon dioxide content; a minimum of 85.0% SiO_2 is required to meet ASTM C1240. Both SF5 and SF8 displayed high amounts of CaO compared to the other six fumes, which is atypical for silica fumes. All chemical composition data were collected using a wavelength-dispersive X-ray fluorescence spectrometer (WDXRF).

Of the eight silica fumes considered, three (SF1, SF2, and SF4) were undensified/raw fumes, whereas the other five were all densified fumes. The physical properties of the eight silica fumes are shown in Table 2. Bulk density was determined by modifying ASTM D4254 [21], whereby a smaller standard mold was used to reduce the amount of material needed for testing. Due to a very limited supply of SF8, the bulk density of this material was not measured.

The results in Table 1 indicate that all densified silica fumes have higher levels of carbon impurities than the undensified fumes, as evidenced by each material's respective loss on ignition (LOI), measured in accordance with ASTM C311 [22]. Densified fumes have, on average, 3% higher levels of carbon impurities when compared to undensified fumes. According to ASTM C1240, the LOI for silica fumes cannot exceed six percent. Once again, SF8 did not meet this specification. The differences between the undensified and densified silica fume LOIs suggest the densification process is possibly imparting impurities to the materials.

Silica fumes were physically characterized by using laser diffraction to determine particle size and the Brunauer-Emmett-Teller (BET) method for specific surface area. Due to the agglomerated nature of the particle (Fig. 1), dispersion of the particles was required prior to particle size analysis. Disaggregation of the

Table 1
Silica fume chemical characterization data.

Silica Fume	SiO_2	CaO	LOI
SF1	92.3	1.5	0.9
SF2	96.0	0.3	0.9
SF3 ¹	83.5	0.9	1.9
SF4	97.8	0.3	0.9
SF5 ¹	70.0	19.5	5.1
SF6	90.5	0.6	2.4
SF7	94.4	0.6	1.9
SF8 ^{1,2}	68.4	18.0	8.9

¹ Silica fume fails to meet ASTM C1240 due to low SiO_2 content.

² Silica fume fails to meet ASTM C1240 due to high LOI.

Table 2
Silica fume physical characterization data.

Silica Fume	Specific Surface Area (m ² /g)	G _s (g/cm ³)	d _{50,BET} (nm)	d _{50,LD} (nm)	ρ _b (kg/m ³)
SF1	18.1	2.37	140	205	485
SF2	29.3	2.23	92	113	371
SF3	22.0	2.30	119	98	775
SF4	29.4	2.23	91	79	407
SF5	25.7	2.23	94	111	787
SF6	24.0	2.26	110	80	731
SF7	22.8	2.21	119	78	771
SF8	19.8	2.34	126	98	N/A

Fig. 1. (a) Agglomerated silica fume particle approximately 500 μm in diameter; (b) composed of thousands of smaller particles, many with diameters less than 1 μm.

agglomerated particle produced thousands of smaller particles. Specimens were dispersed in a sodium carbonate solution commonly used for clay dispersion by using a 700 W sonicator with a 12-mm probe for 10 min and then allowed to disperse another three minutes under 100% sonication from the particle size analyzer. This method was chosen because it gave the most consistent results of all dispersion techniques attempted.

Specific surface areas, as shown in Table 2, were determined using nitrogen adsorption techniques. By utilizing true specific gravities, as determined by using ASTM D854 [23], the average particle size can be determined from specific surface area measurements. Because SF5 and SF8 were potentially reactive with water due to their high levels of CaO, the specific gravities of these two fumes were calculated in accordance with ASTM C1240 using kerosene rather than water. Average particle sizes, as determined by BET and by laser diffraction, are also in Table 2. The discrepancies between these two measurements are attributed to specimen variability and incomplete dispersion during laser diffraction measurements.

3. Methods

3.1. Experimental design

The study was conducted as an $8 \times 3 \times 3$ factorial considering three different treatment factors: silica fume (SF1-SF8), water-to-binder (w/b) (0.20, 0.30, 0.45), and the percentage of total materials dry mass represented by silica fume (10%, 20%, 30%). These treatment factors are shown in Table 3 as factors A, B, and C, respectively. For example, for a 30% mixture, 300 g of silica fume would be added to 700 g of cement to create 1000 g of dry materials. This factorial experimental design resulted in 72 distinct combinations to be tested.

Analysis of variance (ANOVA) was used to determine the statistical significance of each treatment factor and first-order interactions. Second-order interactions were not considered. The ANOVA model used is shown in Eq. (1). A significance level (α) of 0.05 was chosen because measured standard errors were low.

$$X_{ijkl} = \mu + \alpha_i + \beta_j + \gamma_k + (\alpha\beta)_{ij} + (\alpha\gamma)_{ik} + (\beta\gamma)_{jk} + \varepsilon_{ijkl} \quad (1)$$

where,

- X_{ijkl} = measured property;
- μ = average of all specimens;
- α_i = effect of silica fume i ;
- β_j = effect of w/b j ;
- γ_k = effect of silica fume percentage k ; and
- ε_{ijkl} = error term.

3.2. High-shear mixing

All composites were mixed by using a commercial laboratory blender with an attached variable speed controller. Initially, all water, HRWRA, and silica fume were blended on the lowest setting until an approximately homogenous paste was achieved. After the silica fume had been incorporated, all mixing followed the protocol illustrated in Fig. 2 described as follows:

- All cement was added slowly with the blender on the lowest setting over two minutes.
- The rotational speed of the blender was increased to 4000 RPM over 30 s.
- The paste was blended at 4,000 RPM for 60 s.
- The paste was allowed to rest for 30 s.
- The rotational speed of the blender was increased to 10,000 RPM over 30 s.

Table 3
ANOVA table for rate index.

Source	DF	SS	MS	F	P
A = Fume type	7	0.11	0.02	7.62	0.00
B = w/b	2	1.97	0.98	460.97	0.00
C = Fume amount	2	0.82	0.41	191.83	0.00
AB	14	0.09	0.01	3.09	0.01
AC	14	0.06	0.00	1.91	0.07
BC	4	0.22	0.06	26.09	0.00
Error	28	0.06	0.00	—	—
Total	71	3.33	—	—	—

Fig. 2. High-shear mixing protocol.

VI. The paste was blended at 10,000 RPM for 30 s.

Because the rheostat used to control the blender was analog rather than digital, exact rotational speeds could not be achieved; thus, all blending was performed at the approximate rotational speeds listed in the high-shear mixing protocol.

3.3. Rheological testing

After the completion of the mixing protocol, paste specimens were added to a rheometer equipped with a cup and paddle analogous to traditional concentric cylinders. According to ASTM C1749 [24], the shear stress and shear rate of a cement paste can be calculated from data collected using a rotational rheometer with concentric cylinders using Eqs. (2) and (3), respectively.

$$\dot{\gamma} = \frac{R_2 \Omega_1}{R_2 - R_1} \quad (2)$$

$$\tau = \frac{\Gamma}{2\pi R_1^2 L} \quad (3)$$

where,

- L = cylinder length, m
- R_1 = inner cylinder radius, m
- R_2 = outer cylinder radius, m
- $\dot{\gamma}$ = shear rate, s^{-1}
- τ = shear stress, Pa
- Γ = torque, Nm
- Ω_1 = rotational speed of inner cylinder, rad/s

A Newtonian fluid of known viscosity was used to calculate a virtual inner radius represented by the paddle attachment. The cup was fitted with a cage insert to minimize wall effects. Material was added until the vanes of the cage insert were completely submerged. The material was allowed to rest in the cup for 30 s to allow it to fully fill all voids and then pre-sheared at $10 s^{-1}$ for 30 s to ensure specimen homogeneity. After pre-shearing, the specimen underwent a loading and unloading cycle, loading from 0 to $60 s^{-1}$ at a rate of $1 s^{-1}/s$ and then unloading from 60 to $0 s^{-1}$ similarly. In total, each test lasted three minutes, meaning all testing was completed before the pastes reached 10 min of age. The unloading curve was used for all rheological characterization, as has been described elsewhere [13]. Specimen loading and unloading curves are shown in Fig. 3.

Specimen data were fit to the Herschel-Bulkley model shown in Eq. (4). Consistency index is analogous to viscosity in most commonly known rheological models for Newtonian and Bingham plastic fluids. In other words, a higher consistency paste can be described as being more viscous. Rate indices greater than one indicate that the material is shear thickening, whereas rate indices less than one indicate shear-thinning behavior. In the case where $n = 1$, the Herschel-Bulkley model reduces to the Bingham plastic model. Practically, pastes with rate indices approaching two are easier to mix than those with rate indices closer to one. As the rate index falls below one, mixing the paste becomes much more difficult.

$$\tau = \tau_0 + k\dot{\gamma}^n \quad (4)$$

where,

Fig. 3. Specimen loading and unloading curves.

- τ = shear stress, Pa
- τ_0 = yield stress, Pa
- k = consistency index, Pa·s ^{n}
- $\dot{\gamma}$ = shear rate, s⁻¹
- n = rate index, unitless

While the raw data collected during rheological testing were able to be fit well with the three-parameter Herschel-Bulkley model, there are certain drawbacks when using the model [13]. First, the fitting process can result in a slightly negative yield stress being given as a rheological parameter, which has no physical interpretation. To account for this, the yield stress has to be bounded at 0 Pa. Second, the consistency index has units of Pa·s ^{n} , with the rate index varying from specimen to specimen. As a result, each consistency index is given in slightly different units, making direct comparison between results impossible. Due to these drawbacks, only rate index results are discussed in this paper.

4. Experimental results

Rate index results for each silica fume tested are shown in Fig. 4. In all cases, increasing the amount of silica fume decreased the rate index; similarly, an increase in w/b also resulted in increased rate indices. For the case of 30% SF2 with a w/b of 0.20, the paste exhibited shear thinning behavior with a rate index of 0.96, with all

Fig. 4. Rate index results.

other tested combinations exhibiting shear thickening behavior. Previous research has shown that shear thickening behavior can be readily observed in cement pastes containing higher dosages of HRWRA, with increased dosages of silica fume leading to a reduction in the shear thickening effect [25].

The ANOVA table for rate index is shown in Table 3. Using the previously denoted α , all three treatment factors, the interaction between fume type and w/b, and the interaction between fume amount and w/b were statistically significant.

These measurements were obtained with the cement pastes at very early ages (less than 10 min from combination of cement and water), and it is unlikely that chemical reactions are responsible for the observed behavior variations. Instead, it is likely the silica fumes' different physical characteristics that best explain the behavior. The given average particle sizes, whether measured from laser diffraction or calculated from specific surface area and specific gravity, only represent a single silica fume characteristic do not fully describe the distribution of particles as a whole. Additionally, data observations indicate there is not any strong dependency between the densification state and the measured rate indices.

Specific surface area measurements can be used to explain the observed rheological differences. As specific surface area increases, the measured rate index generally decreases, especially at low w/b. It is hypothesized that this response is caused by a decrease in the thickness of the lubricating layer of water between particles. For equivalent amounts of water, the lubricating layer of water around each particle becomes thinner as surface area increases. Due to the decrease in lubrication, the composite becomes much more difficult to mix. In mixtures with high w/b, these effects are minimized due to excess water, suggesting a critical lubrication layer thickness exists beyond which surface area no longer controls the rheological behavior. This is further suggested by the coefficients of determination (R^2) shown in Fig. 4, as the variance explained by surface area variations increases as the w/b decreases.

Additional research is needed to investigate this critical lubricating layer thickness, but the hypothesis is supported by findings from Chen et al. [26], who showed that this lubrication layer thickness could be used to describe increased flow behavior in high w/b (greater than 0.60) composites containing condensed silica particles.

5. Rheological model

5.1. Model development

Multiple linear regression was used to develop an empirical model relating specific surface area, w/b, and silica fume percentage to rheological behavior (Eq. (5)). Initially, all three treatment factors and both significant first-order interactions determined from ANOVA were included in the regression model (Eq. (6)). Upon development of the multiple linear regression equation model, statistically insignificant components were removed from the equation (Table 4), leaving three significant factors in the regression model (Eq. (7)). The resulting regression equation was then

Table 4
Significance and regression coefficients.

Coefficient	Eq. (5)	Eq. (6)	Eq. (7)
β_1	1.90	2.14	2.04
β_2	-0.010 (P = 0.00)	-0.020 (P = 0.01)	-0.0089 (P = 0.00)
β_3	-0.33 (P = 0.00)	-0.33 (P = 0.57)	—
β_4	-0.018 (P = 0.00)	-0.030 (P = 0.00)	-0.0369 (P = 0.00)
β_5	0.036 (P = 0.01)	0.036 (P = 0.11)	—
β_6	-0.00050 (P = 0.07)	—	—
β_7	0.052 (P = 0.00)	0.052 (P = 0.00)	0.0755 (P = 0.00)

plotted through the 72 raw data points (Fig. 5), showing a very strong correlation ($R^2 = 0.88$). By using a regression equation relating measured and predicted indices, it can be shown that Eq. (7) will tend to underpredict $n > 1.568$ and overpredict $n < 1.568$. The standard error between the predicted and measured rate indices was 0.072.

$$n = \beta_1 + \beta_2 SSA + \beta_3 \left(\frac{w}{b}\right) + \beta_4 P_{SF} + \beta_5 SSA \left(\frac{w}{b}\right) + \beta_6 SSA (P_{SF}) + \beta_7 \left(\frac{w}{b}\right) (P_{SF}) \quad (5)$$

$$n = \beta_1 + \beta_2 SSA + \beta_3 \left(\frac{w}{b}\right) + \beta_4 P_{SF} + \beta_5 SSA \left(\frac{w}{b}\right) + \beta_7 \left(\frac{w}{b}\right) (P_{SF}) \quad (6)$$

$$n = \beta_1 + \beta_2 SSA + \beta_4 P_{SF} + \beta_7 \left(\frac{w}{b}\right) (P_{SF}) \quad (7)$$

5.2. Model validation

In order to validate the proposed empirical model, 10 trial cement-silica fume paste combinations were determined by random number generation, specimens prepared, and requisite measurements made to compare the measured response with model predictions. These trial combinations are shown in Table 5.

The measured rate indices of the 10 validation mixtures compared with the predicted values from Table 5 are shown in Fig. 6. There appears to be a strong correlation ($R^2 > 0.9$) between the predicted and the measured indices using the Eq. (7) model. The measured rate indices for nine of the 10 validation mixtures fell within 5% of the predicted value with the 10th predicted rate index only 5.2% less than measured.

Fig. 5. Rheological model fit.

Table 5
Model validation mixtures with predicted rate indices.

Trial	Silica Fume	w/b	P _{SF}	n _{predicted}
T1	SF5	0.24	10	1.62
T2	SF7	0.20	24	1.31
T3	SF2	0.34	11	1.66
T4	SF4	0.29	17	1.52
T5	SF2	0.25	17	1.47
T6	SF8	0.40	26	1.69
T7	SF1	0.44	14	1.83
T8	SF3	0.33	10	1.73
T9	SF7	0.29	27	1.43
T10	SF4	0.29	29	1.34

Fig. 6. Model validation mixtures.

6. Conclusions

Many conclusions can be drawn from this study. First, changing silica fume source has a statistically significant effect ($P < 0.01$) on the rheological properties of oil well cement pastes. Secondly, the effect of changing silica fume sources becomes more pronounced at higher silica fume dosages. Silica fume dosage has a statistically significant effect ($P < 0.01$) on the rheological properties of oil well cement pastes. Thirdly, densification does not significantly affect the rheological properties of oil well cement pastes. Lastly, specific surface area can be used, along with mixture proportions such as w/b and silica fume dosage, to predict the rheological characteristics of oil well cement pastes.

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14. ABSTRACT Specialized classes of concrete, such as ultra-high-performance concrete, use volumes of silica fume in concrete that are higher than those in conventional concrete, resulting in increased water demand and mixing difficulty. This study considered the effects of eight different silica fumes in three dosages (10%, 20%, 30%) with three w/b (0.20, 0.30, 0.45) on rheological behavior as characterized by the Herschel-Bulkley model. Results indicated that the specific source of silica fume used, in addition to dosage and w/b, had a significant effect on the rheological behavior. As such, all silica fumes cannot be treated as equivalent or be directly substituted one for another without modification of the mixture proportion. The rheology of cement pastes is significantly affected by the physical properties of silica fume more so than any chemical effects.					
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