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# Potential of finely ground limestone powder to benefit ultra-high performance concrete mixtures

Jedadiah F. Burroughs [a,*], Jay Shannon [a], Todd S. Rushing [a], Kevin Yi [b], Quinn B. Gutierrez [b], Danny W. Harrelson [a]

[a] Geotechnical and Structures Laboratory, U.S. Army Engineer Research and Development Center, 3909 Halls Ferry Rd, Vicksburg, MS 39180, USA
[b] Department of Civil and Mechanical Engineering, United States Military Academy, Bldg. 752 Thayer Road, West Point, NY 10996, USA

## HIGHLIGHTS

- The use of limestone powder as an inert filler in UHPC is proposed.
- Limestone powder reduces health hazards associated with silica powder in UHPC.
- Minor replacements of cement with limestone do not negatively affect performance.
- Full replacement of silica powder does not significantly impact UHPC performance.

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## ABSTRACT

Ultra-high performance concrete is a specialized class of cementitious materials notable for very high compressive strength, increased toughness, and improved durability relative to conventional concrete. Due to its high cement content and the use of very finely ground crystalline silica, ultra-high performance concrete has a greater cost and $CO_2$ footprint than normal concrete. Potential health concerns can also arise due to repeated inhalation of crystalline silica powder during production. Finely ground limestone powder was hypothesized as a potential filler to help minimize these negative aspects when used as either a partial or full mass replacement of similarly sized cement and silica powder. Limestone powder was shown to have a positive effect on fresh properties of the composite including mixing time and workability. Small replacements of cement were shown to have beneficial effects on compressive strength. Scanning electron microscopy showed that limestone powder replaced unreacted materials without significantly impacting hydration reactions.

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## 1. Introduction

Ultra-high performance concrete (UHPC) is defined by American Concrete Institute (ACI) Committee 239 as "concrete that has a minimum specified compressive strength of 150 MPa (22,000 psi) with specified durability, tensile ductility and toughness requirements" in which "fibers are generally included to achieve specified requirements" [1]. UHPC is a low water-to-cement (w/c) ratio composite generally composed of cement, fine siliceous aggregates, silica fume, various chemical admixtures, and reinforcing fibers, with cement paste representing approximately 1/3 of the matrix volume in most cases. Due to the high cementitious materials content and optimized particle packing, UHPC is beneficial for use in many cases where high compressive strengths, increased durability, and increased tensile capacity are required, such as in bridge decks and girders [2,3].

As with any material, certain drawbacks exist with the production of UHPC. The high cement content used in mixture designs increases the cost and the carbon footprint of the material as cement production expels higher amounts of $CO_2$ than the other materials in UHPC. Therefore, decreasing the amount of cement used decreases the cost and the carbon footprint of the material. Finely ground limestone, or limestone powder, has been used to reduce the $CO_2$ footprint of concretes, generally through the intra-grinding of limestone with cement clinker in the production of portland cement to produce portland-limestone cement (PLC) [4,5]. Limestone powder has been successfully used in conjunction with portland cement as an accelerator [6–9], particularly when used in high-volume fly ash mixtures [10]. Limestone powder

* Corresponding author.
*E-mail address:* Jedadiah.F.Burroughs@usace.army.mil (J.F. Burroughs).

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has been shown to have similar retarding effects to gypsum when added to raw cement clinker [11]. Other studies have shown the benefits of blending limestone powders with cements prior to use [4,12]. One of the most common uses for limestone powder cited in open literature is as a mineral admixture to improve workability [13–15]. Other uses for limestone powder include reducing the expansion associated with sulfate attack [16] and decreasing the cumulative amount of heat produced during hydration [17].

Bentz et al. [18] studied the effects of the addition of limestone powder to ordinary portland cement (OPC) concrete on early age properties such as setting time and heat of hydration as well as compressive strength development. Results showed that the addition of 10% very fine limestone powder by volume as a replacement for OPC decreased the setting time and had little to no effect on the early age strength development when compared to a reference material. Minor reductions in strength were seen at 28 days age [18]. Liu et al. also showed this replacement threshold for limestone powder for nominally 150 MPa mixes [19]. Other studies have suggested that increases in compressive strength can be seen with up to 40% replacement of cement [14].

There has been significant discussion in the published literature concerning whether or not limestone powder is reactive. Liu et al. [19] showed that hydration products such as $C_3A{\cdot}CaCO_3{\cdot}11H_2O$ and $C_3A{\cdot}3CaCO_3{\cdot}32H_2O$ were present in samples containing limestone powder that were cured at high temperature. These phases were not present in similar samples without limestone, suggesting that limestone powder has some reactivity when mixed with cement paste. Other studies show limestone powder to be inert [9,17] with accelerating benefits caused by nucleation effects rather than reactivity [18]. Still other studies suggested that limestone powder is reactive at low addition rates, while also providing a filler effect at higher addition rates [20]. The potential for limestone reactivity can be further increased by decreasing temperatures [21].

Of possibly greater concern are the potential long-term effects of repeated exposure to aerosolized very fine crystalline silica that is included in some UHPC mixture designs. Inhalation of these materials through prolonged exposure can lead to silicosis, a fibrosis of the lungs that can lead to shortness of breath, persistent cough, and lung lesions that can be fatal in certain cases. Crystalline silica is also carcinogenic to humans according to the International Agency for Research on Cancer [22]. The potential for inhalation is not limited simply to production of the material, as crystalline silica inhalation has been shown to be a potential health hazard in the removal of existing concrete as well [23]. According to the National Institute for Occupational Safety and Health (NIOSH), no chronic health issues are associated with repeated exposures to ground limestone. Ground limestone products that contain more than 3% quartz can cause similar health issues to those related to silica powder [24].

The two forms of crystalline silica generally used in the production of UHPC are fine silica sand and silica powder. Fine silica sand is a natural whole grain silica that is marketed as many different products based on the gradations that are required. In UHPC, all silica sands used are generally smaller than 500 μm in diameter. Silica powder (also called quartz flour or silica flour) is a finely ground form of silica that is generally much finer than silica sand and sometimes angular in shape compared to the rounded sand particles. It is much easier for silica powder to become aerosolized than fine silica sand; therefore, silica powder is of greater concern for inhalation.

Considering the cost, environmental, and health issues, it was hypothesized that finely ground limestone could be used as either full or partial replacement for cement and silica powder in UHPC with little to no detriment to the composite properties of the material. The study presented herein was a preliminary investigation into the effects of the use of limestone powder on the mixing time, rheology, and compressive strength of UHPC. Image analysis using scanning electron microscopy was also used to further analyze the utility of limestone powder in UHPC.

## 2. Materials and methods

### 2.1. Constituents

American Petroleum Institute (API) class H oil well cement was used in this study rather than OPC due to its coarser grinding, typically higher dicalcium silicate ($C_2S$) content, and lack of tricalcium aluminate ($C_3A$), which allows for slower and more thorough hydration. Inert fine silica sand (d < 500 μm) and silica powder were used as the primary fine aggregates in this study. Elkem ES900W silica fume, or microsilica, was used as a very fine-grained supplementary cementitious material in all mixtures. A polycarboxylate high-range water-reducer (HRWR) was used to improve the workability of the material without significantly altering the w/c ratio of the composite.

The limestone powder used in this study was CalCarb™ R2 Ground Calcium Carbonate. This product was chosen as the commercially available product with an average particle size most similar to both the cement and silica powder used in this study. Similar particle sizes were of utmost importance for this study because optimal particle packing was necessary for UHPC to reach the desired mechanical properties. Further characterization information is shown in Table 2. Due to the negligible amount of $C_3A$ present in the cement used, the additional phases documented by Liu et al. [19] formed from the reaction of limestone powder with $C_3A$ at high temperature were not expected to be formed, further supporting the hypothesis that the limestone powder would serve as an inert filler. Nominal average particle sizes ($D_{50}$) and densities (ρ) of all powder constituents are given in Table 1.

### 2.2. Mixture proportions

The baseline mixture proportion used in this study is shown in Table 3. Fibers were not included as no tensile or flexural properties were considered, and all batching was performed on a mass basis. For comparison, 10% replacement of cement by volume equated to roughly 8.5% replacement of cement by mass when replacing cement by limestone powder. In order to determine the

**Table 1**
Physical properties of powder constituents.

| Material | $D_{50}$ (μm) | ρ (kg/m$^3$) |
|---|---|---|
| Cement | 18 | 3150 |
| Silica Sand | 300 | 2650 |
| Silica Powder | 16 | 2650 |
| Silica Fume | – | 2200 |
| Limestone Powder | 25–30 | 2700 |

**Table 2**
Typical chemical properties of CalCarb™ R2 Ground Calcium Carbonate (adapted from Mississippi Lime) [25].

| $CaCO_3$ | 98.5% |
|---|---|
| $MgCO_3$ | 0.6% |
| $SiO_2$ | 0.6% |
| $Al_2O_3$ | 0.05% |
| $Fe_2O_3$ | 0.04% |
| $H_2O$ | 0.03% |

**Table 3**
Baseline Mixture Proportion.

| Material | Proportion (mass) |
|---|---|
| Cement | 1.000 |
| Silica Sand | 0.967 |
| Silica Powder | 0.277 |
| Silica Fume | 0.389 |
| HRWR | 0.017 |
| Water | 0.208 |

**Table 4**
Experimental Design on Mass Basis. CR and SPR represent the amount of cement and silica powde replacement respectively.

| Batch Number | 0% CR | 5% CR | 10% CR | 15% CR |
|---|---|---|---|---|
| 0% SPR | B1 | B5 | B9 | B13 |
| 33% SPR | B2 | B6 | B10 | B14 |
| 67% SPR | B3 | B7 | B11 | B15 |
| 100% SPR | B4 | B8 | B12 | B16 |

upper threshold for the use of limestone powder as a cement replacement, the three dosage rates for limestone powder considered were 5%, 10%, and 15% by mass.

To validate cement element interpretation, scanning electron photomicrographs were obtained for cut surfaces of the hardened UHPC samples by using a FEI Nova NanoSEM 630 scanning electron microscope (SEM). Elemental maps, shown in Fig. 1, were generated using a Bruker solid-state EDX detector. Ferrite ($C_4AF$) was seen easily in the elemental maps by its blue and yellow color. Ferrite was most commonly attached to belite ($C_2S$). Alite ($C_3S$) and aluminate ($C_3A$) phases were seen in the red over green and red-blue particles in the elemental maps, respectively. Limestone particles (not present in Mixture B1) appeared as a dark non-uniform red. Elementally, calcium is shown in red, silicon is shown in green, aluminum is shown in blue, and iron is shown in yellow.

### 2.3. Experimental design

With no prior research concerning the replacement of silica powder with limestone powder in UHPC, the three dosages considered for silica powder replacement were 33%, 67%, and 100% by mass. This allowed for the experimental design to be modeled as a $4 \times 4$ factorial with 0% replacement of both cement and silica powder as the reference batch. This experimental design resulted in 16 distinct batches to be studied as shown in Table 4.

Statistical analyses were completed using the two-way analysis of variance (ANOVA) model shown in Eq. (1), where $X_{ijk}$ represented the measured property of a sample, $\mu$ represented the grand mean for all samples, $\alpha_i$ represented the treatment effect for silica powder replacement percentage $i$, $\beta_j$ represented the treatment effect for cement replacement percentage $j$, and $\varepsilon_{ijk}$ represented error. Statistical analysis for compressive strength was performed using averages per batch due to the inherent variability of UHPC; therefore, interaction effects were not considered due to a limited number of degrees of freedom. This assumption was seen as appropriate because inert filler was being added as replacement for two different materials rather than reactive elements being added. A standard significance level of $\alpha = 0.10$ was chosen for all analyses due to the inherent variability with this class of material.

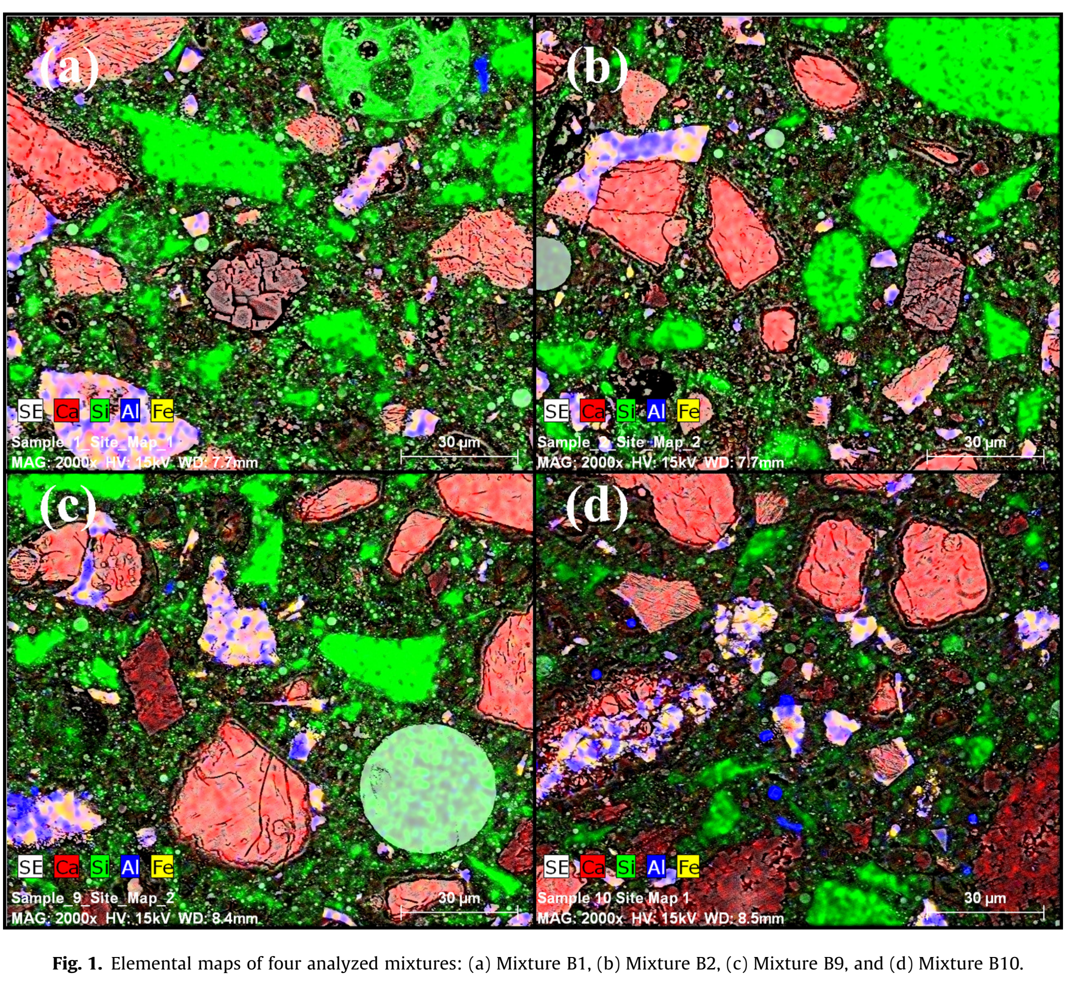


**Fig. 1.** Elemental maps of four analyzed mixtures: (a) Mixture B1, (b) Mixture B2, (c) Mixture B9, and (d) Mixture B10.

$$X_{ijk} = \mu + \alpha_i + \beta_j + \varepsilon_{ijk} \quad (1)$$

### 2.4. Mixing time

All batching was performed in the same manner. First, all dry ingredients were added to an 11.4 L Hobart Legacy® Countertop mixer. All mixing was performed with an attachment speed of 33 RPM. Dry materials were blended together for two minutes prior to the addition of the premixed liquid constituents. As soon as the liquid mixture was added to the dry materials, a timer was started to measure the time required for the mixture to "break over" or wet out. Breaking over was defined as the time at which the material progressed from being in a granular state through a fluid state to a more fluid state suitable for placing. Mixing time was rounded to the nearest whole minute.

### 2.5. Workability

Workability was measured in accordance with ASTM C1437 [26]. This test method determines the flow of a material as a percentage of spread from an initial cone diameter. Because UHPC contains no coarse aggregate, it meets the definition to be classified as a mortar for use with this test method. The flow cone was filled approximately half full and tamped 20 times to increase consolidation. The remaining volume of the flow cone was then filled and tamped an additional 20 times to ensure compatibility between the two layers. Excess material above the top of flow cone was struck off using a straight edge, and the flow cone was then removed. The flow table was then raised and dropped 25 times in 15 s. After the drops were completed, the diameter of the resulting spread was measured at four distinct spots using specially designed calipers. The sum of these four diameters represented the percentage of spread caused by the energy of the 25 drops.

### 2.6. Strength testing

Compressive strength testing was performed in accordance with ASTM C109 [27]. Fifty millimeter cubic samples were used for testing. Molds were filled approximately half full and rodded lightly before adding the next lift of material. The filled molds were then placed on a vibrating table for approximately 30 s to increase consolidation. Samples were stored in a room temperature (23°C), 100% humidity environment for seven days (samples were demolded on day two). Samples were then submerged in a 90°C water bath for further curing for six days. After the full 13 days of curing, samples were allowed to cool to room temperature for 24 h and tested at 14 days. The experience of the research team has shown no difference in testing similar samples at 14 and 28 days. A Tinius Olson load frame capable of applying 1950 kN of force was used to test samples. Load and displacement data were recorded for each sample, with the compressive strength reported as the peak load reached divided by the initial cross-sectional area.

## 3. Results and discussion

In Figs. 2, 4, and 6, for a given level of cement replacement, the corresponding raw data points represent all four levels of silica powder replacement. Conversely, in Figs. 3, 5, and 7, for a given level of silica powder replacement, the corresponding raw data points represent all four levels of cement replacement.

### 3.1. Mixing time

The effects of limestone powder replacement on the mixing time of UHPC are shown in Figs. 2 and 3. The data in Fig. 2 suggest that there was a minor relationship between the amount of cement replacement and the required mixing time, with increasing cement replacement resulting in reduced mixing time. Fig. 3 shows that a trend toward shorter mixing times was also observed when limestone powder was used to replace silica powder rather than cement. In both cases, the use of limestone powder decreased the required mixing time for UHPC. The results in Fig. 2 show that increasing the amount of cement replaced decreased the variability in the measured mixing times, and slightly decreased average mixing times. Fig. 3 shows that this effect was also consistent with increasing the amount of silica powder replaced. Because the limestone powder used was slightly coarser than both the cement and silica powder it replaced, the amount of surface area required to wet was smaller. Furthermore, the reduction in cement content resulted in an increase in the effective dose of HRWR, which could lead to reduced mixing time.

As can be seen from the results of the statistical analysis shown in Table 5, both treatment factors were statistically significant.

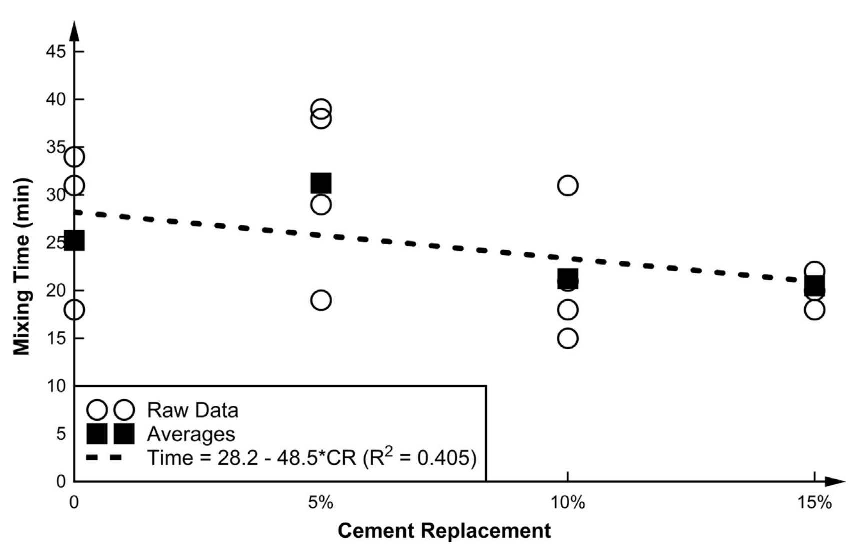


**Fig. 2.** Measured required mixing time to break over as a function of the percent mass replacement of cement with similarly sized limestone powder. Raw data points represent all four levels of silica powder replacement at each cement replacement level.

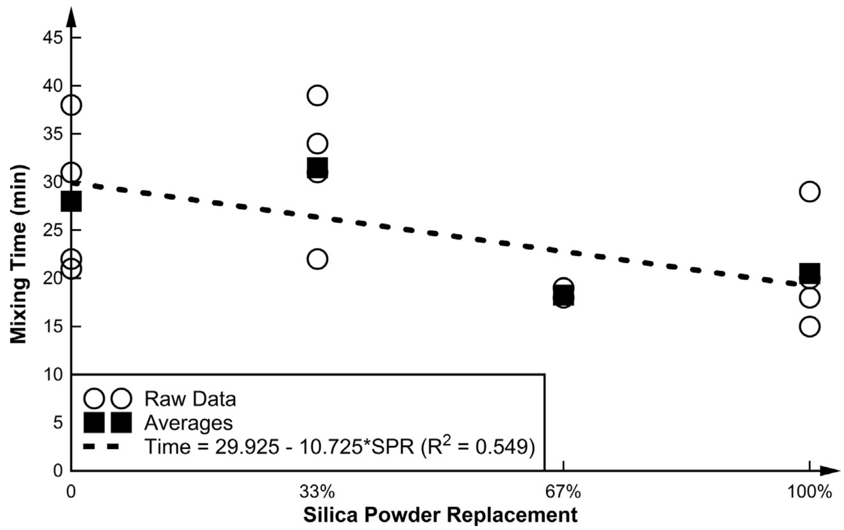


**Fig. 3.** Measured required mixing time to break over as a function of the percent mass replacement of silica powder with similarly sized limestone powder. Raw data points represent all four levels of cement replacement at each silica powder replacement level.

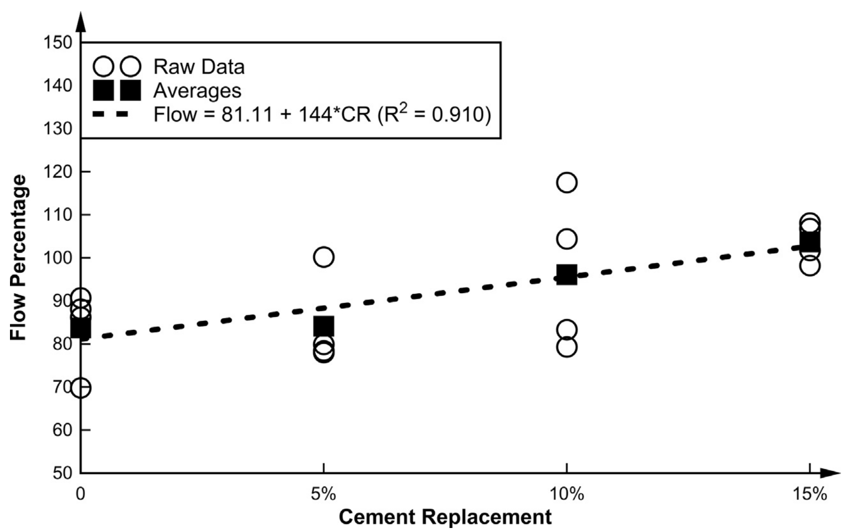


**Fig. 4.** Measured flow percentage as a function of the percent mass replacement of cement with similarly sized limestone powder. Raw data points represent all four levels of silica powder replacement at each cement replacement level.

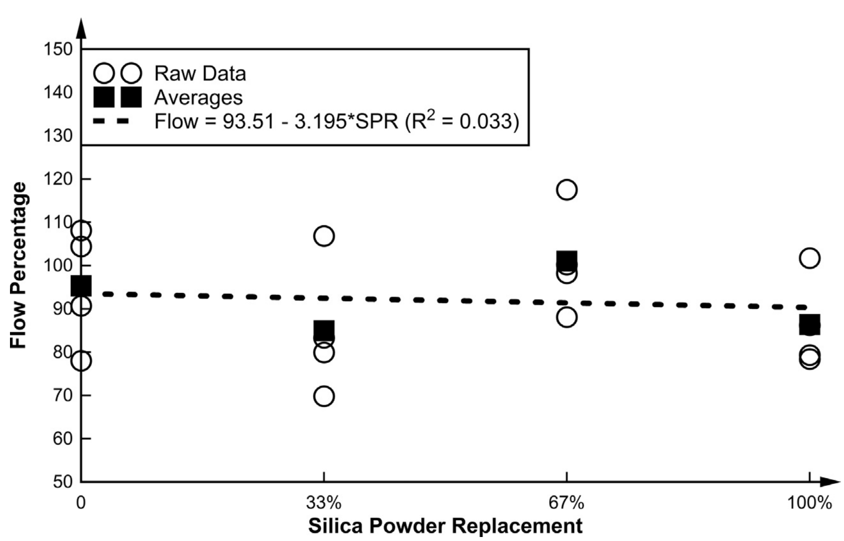


**Fig. 5.** Measured flow percentage as a function of the percent mass replacement of silica powder with similarly sized limestone powder. Raw data points represent all four levels of cement replacement at each silica powder replacement level.

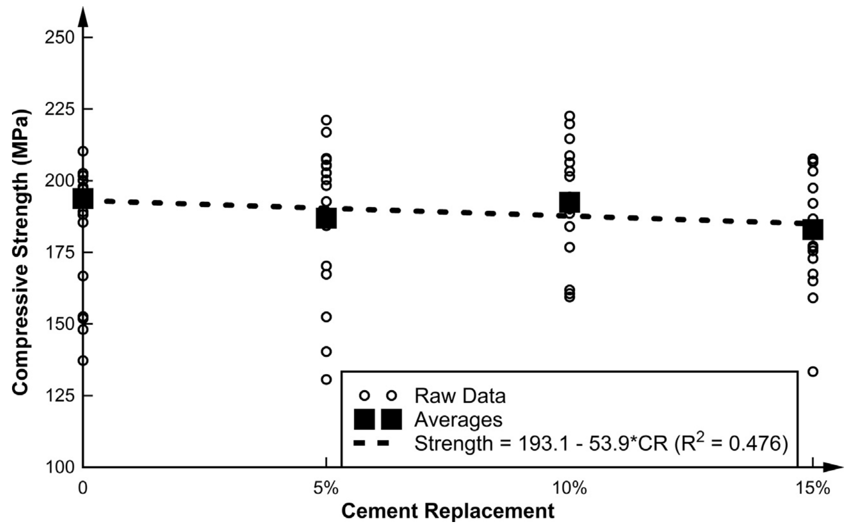


**Fig. 6.** Measured 14-d concrete compressive strength as a function of percent mass replacement of cement with similarly sized limestone powder. Raw data points represent all four levels of silica powder replacement at each cement replacement level.

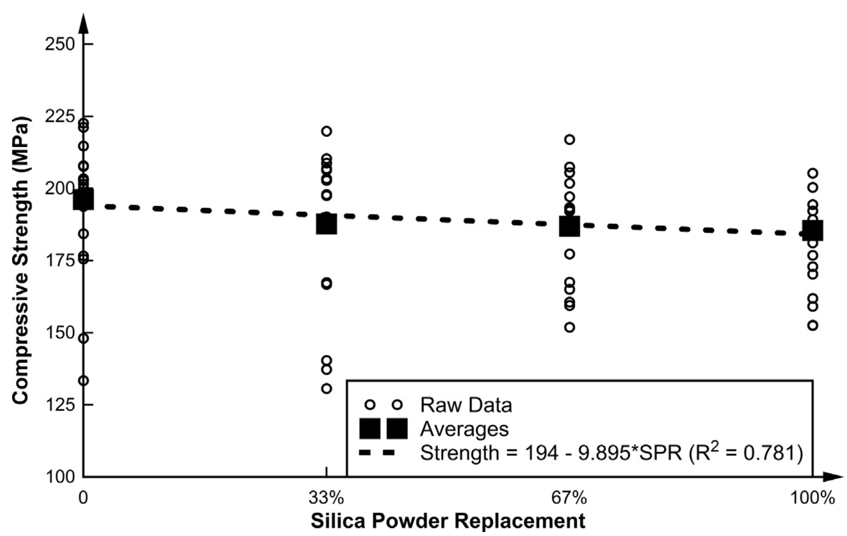


**Fig. 7.** Measured 14-d concrete compressive strength as a function of the percent mass replacement of silica powder with similarly sized limestone powder. Raw data points represent all four levels of cement replacement at each silica powder replacement level.

**Table 5**
ANOVA Table for Mixing Time.

| Source of Variation | SS | DOF | MS | F | P-value |
|---|---|---|---|---|---|
| Silica Powder Replacement | 465.19 | 3 | 155.06 | 8.40 | 0.006 |
| Cement Replacement | 290.69 | 3 | 96.90 | 5.25 | 0.023 |
| Error | 166.06 | 9 | 18.45 | | |
| Total | 921.94 | 15 | | | |

These results indicated that using limestone powder to replace both cement and silica powder in the levels studied statistically lowered the measured mixing time. This statistical analysis was in agreement with practical interpretations presented above.

### 3.2. Workability

The effects of using limestone powder on the measured workability of UHPC are shown in Figs. 4 and 5. Fig. 4 illustrates that there was a strong relationship between the amount of cement replaced with limestone powder and the measured workability, with workability increasing with increasing cement replacement. This trend did not continue when limestone powder was used to replace silica powder as shown in Fig. 5. Silica powder replacement showed a weak relationship between variables and no noticeable trends. A likely explanation for the observed effects on workability was that the effective dose of HRWR was modified when cement was replaced by limestone powder, but not when silica powder was replaced. Because it is designed to act on reactive cement particles, HRWR is normally dosed as fluid ounces per hundredweight of cement. Partial replacement of cement with inert limestone powder, at a constant addition rate of HRWR, resulted in a higher effective dosage of HRWR in these mixtures and consequently, greater workability. In contrast, when the inert silica powder was replaced with inert limestone powder, there was no change in the effective dose of HRWR in these mixtures, and workability was generally unaffected.

As can be seen from the results of the statistical analysis shown in Table 6, the replacement of cement with limestone powder factor was statistically significant while the replacement of silica powder was not. This statistical analysis indicated that the use of

**Table 6**
ANOVA Table for Workability.

| Source of Variation | SS | DOF | MS | F | P-value |
|---|---|---|---|---|---|
| Silica Powder Replacement | 691.69 | 3 | 230.56 | 2.16 | 0.162 |
| Cement Replacement | 1139.12 | 3 | 379.71 | 3.56 | 0.060 |
| Error | 958.83 | 9 | 106.54 | | |
| Total | 2789.64 | 15 | | | |

**Table 7**
ANOVA Table for Compressive Strength.

| Source of Variation | SS | DOF | MS | F | P-value |
|---|---|---|---|---|---|
| Silica Powder Replacement | 278.70 | 3 | 92.90 | 0.23 | 0.876 |
| Cement Replacement | 305.25 | 3 | 101.75 | 0.25 | 0.861 |
| Error | 3703.82 | 9 | 411.54 | | |
| Total | 4287.77 | 15 | | | |

limestone powder to replace cement has a significant impact on the measured workability. The analysis for cement replacement is in good agreement with the results shown in Fig. 4; however, the statistical analysis indicated that a stronger trend should have been expected between silica powder replacement and workability in Fig. 5.

### *3.3. Strength testing*

The effects of the use of limestone powder on the 14-day compressive strength of the composite material are shown in Figs. 6 and 7. Fig. 6 shows that small replacements of cement with limestone powder had little effect on compressive strength. Overall, trends between cement replacement and compressive strength were not strong ($R^2$ of 0.48). Variability in compressive strength results was larger for samples with 5% and 10% replacement than either 0% or 15% replacement. The results indicate that replacing some of the cement with limestone powder in the studied UHPC did not cause significant detriment to the compressive strength.

Fig. 7 shows that the addition of limestone powder as replacement for silica powder had a slight negative effect on average compressive strengths for any percentage of limestone powder used; however, the loss of compressive strength was less than 6% of the control mixtures, even at 100% replacement of silica powder. This equates to a 1600 psi (11 MPa) reduction in compressive strength. As the limestone powder was slightly coarser than the silica powder, the reduction in compressive strength could be attributed to suboptimal particle packing. Interestingly, the middle replacement rates again exhibited more variability than either the 0% or 100% replacement mixtures.

As can be seen from the results of the statistical analysis shown in Table 7, neither treatment factor was statistically significant. These results indicated that using limestone powder to replace either cement or silica powder in the levels tested did not have a significant impact on the measured compressive strength, which further supported the hypothesis of this study. Given the very high P-values ($P > 0.85$ in both cases), it was reasonable to modify Eq. (1) as shown in Eq. (2).

$$X_{ijk} = \mu + \varepsilon_{ijk} \tag{2}$$

This modified ANOVA model indicated that the compressive strength of any sample using any combination of the studied treatment factors can be statistically explained as the sum of the overall grand mean of all samples tested and random variation/error. The use of limestone powder in place of cement or silica powder had no statistical effect on the measured compressive strength of any of the 16 mixtures studied.

## 4. Conclusions

The present study showed that limestone powder can be used effectively as a filler in ultra-high performance concrete. The following conclusions were drawn from the data presented:

1. Using limestone powder as partial replacement of cement and partial or full replacement of silica powder slightly decreased the mixing time required to produce UHPC.
2. Partial replacement of cement has a strong positive effect on the workability of the composite. Replacement of silica powder has little to no effect on the workability of the composite.
3. Compressive strength trends were not significant statistically or practically. In general, a slight downward trend was observed with both cement and silica powder replacement. The analysis showed that some amount of cement, silica powder, or both can be replaced in the studied UHPC without significant detriment to compressive strength. There did appear to be the potential of optimized mixtures in which marginally greater compressive strengths might be obtained.
4. Considering the similar, or in the case of mixing time improved, performance of the UHPCs tested, full replacement of silica powder with limestone powder appears to be a good alternative to eliminate the threat of silicosis. Likewise, replacement of a partial amount of cement with limestone powder would reduce total cost and $CO_2$ emissions associated with the UHPC's production, without significantly reducing strength.

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