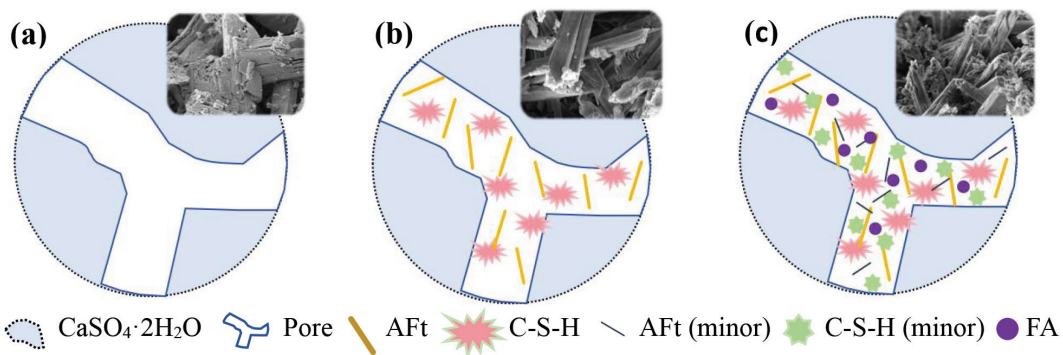


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MEETINGS

FEBRUARY 2026

10-12—1st International Conference on Mg-based cements, Murten, Switzerland, www.empa.ch/web/MgO2026

11-12—8th Annual Canadian Concrete Expo (CCE 2026), Toronto, ON, Canada, <https://canadianconcreteexpo.com>

16-18—Geo Week, Denver, CO, www.geo-week.com

18-19—Michigan Concrete Association (MCA) 2026 Annual Conference, Battle Creek, MI, www.miconcrete.org/annualconference

23-25—46th Annual Portland Cement Concrete Pavement Conference, Kansas City, MO, www.acpa.org/event/46th-annual-portland-cement-concrete-pavement-conference

26-27—2026 Decorative Concrete Expo, Canton, OH, www.deco-cretesupply.com/expo

FEBRUARY-MARCH 2026

28-2—NRMCA 2026 Annual Convention, Las Vegas, NV, www.nrmca.org/conferences-events/nrmca-annual-convention

MARCH 2026

1-3—2026 ASA Shotcrete Convention & Technology Conference, Santa Fe, NM, www.shotcrete.org/convention

4-5—ICC Central Europe 2026, Warsaw, Poland, www.iccx.org/exhibitions/central-europe

15-18—ICRI 2026 Spring Convention, Nashville, TN, www.icri.org/event/icri-2026-spring-convention

15-19—AMPP Annual Conference + Expo, Houston, TX, <https://ace.ampp.org/home>

19-20—1st International Conference on the Safe, Sustainable, and Swift Reconstruction of Ukraine, Lviv, Ukraine, www.circular-concrete.in.ua/swift-reconstruction-conference

23-27—Second Latin American Structural Engineering and Construction Conference 2026 (LATAM SEC 2026), Quito, Ecuador, www.isec-society.org/LATAM_SEC_2026

MARCH-APRIL 2026

30-1—International Conference on Digital Frontiers in Buildings and Infrastructure (DFBI2026), Barcelona, Spain, www.dfbi.org

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2027—Oct. 24-27, Caribe Royale Orlando, Orlando, FL

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ON FRONT COVER: 123-M11, p. 136, Fig. 13—Reaction mechanism of modified gypsum: (a) CG0; (b) CG2; and (c) ICMB.

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Title No. 123-M01

Cement-Based Mortar Performance with High Limestone Filler Additions

by Andrés A. Torres-Acosta, Brenda V. López-Arias, and Celene Arista-Perrusquía

The cement industry's strategy in many countries is to reduce its CO₂ emissions to diminish the greenhouse effect. This strategy aims to reduce these emissions by decreasing the clinker content in their new formulations, replacing it with supplementary cementitious materials (SCMs) or inert fillers. One of the most-used additions in Latin America's cement industry is inert limestone fillers, which is the most inexpensive one. In North America, there are restrictions on using this inert addition in portland cement, with a maximum allowable content of 15% as limestone content (LSC). Nevertheless, in Latin America and other countries, this limestone filler content restriction is not as strict, allowing contents of up to 35%. This investigation includes experimental results obtained from portland cement mortars where inert limestone fillers were used at a replacement level between 20 and 30% by clinker, and only 24-hour curing was considered. Results obtained include mechanical (compressive strength), physical (electrical resistivity, total void content, and capillary porosity), and chemical (carbonation after 1 year of natural exposure) performance of such mortars. The carbonation coefficients (k_{CO_2}) obtained after 1 year of exposure in a natural urban environment were 17.3, 22.9, and 24.5 mm/y^{1/2} for 23%, 27%, and 29% LSCs, respectively. These results were higher than typical k_{CO_2} values of approximately 4 mm/y^{1/2} obtained from ordinary portland cement (OPC)-based mortars with a 90 to 95% clinker content, and standard 28-day water curing.

Keywords: carbonation; compressive strength; durability; limestone fillers; mortar.

INTRODUCTION

Currently, the construction industry is transitioning toward sustainability; an example of this is the strategy of cement companies reducing the clinker in their portland cement formulations to decrease CO₂ emissions from their production processes. However, these solutions are specific to cement and do not consider the final product, which in many cases is mortar and concrete. This solution may seem sustainable at first, but it could have a negative impact on the durability and sustainability of cement-based materials. Concrete structures are typically considered to be durable structures with low maintenance costs during their service life. However, in the last two decades, an increase in cracking and delamination has been observed in concrete structures that are only 2 to 10 years in service.¹

In many cases, the increase in cracking and delamination is due to a limited understanding of the performance of hardened concrete fabricated with the new portland cement formulations used in the construction of concrete infrastructure, such as bridges, piers, industrial floors, hydraulic pavements, and so on. These new portland cement formulations

are named composite portland cements (referred to as CPC in many standards), which are increasingly being used in construction in Mexico and several Latin American countries.

CPCs are cement types that have displaced the use of ordinary portland cements (OPCs), which have a high clinker content (between 90 and 95%), as per ASTM C150/C150M-22.² In addition to clinker, other alternative products are added to CPCs, which, according to the Mexican standard NMX-C-414-ONNCCE-2017³ and other Latin American standards, can be granulated blast-furnace slag, natural pozzolans, fly ash, silica fume, limestone, and minor materials. All these products can be divided into two types of additions: supplementary cementitious materials (SCMs), such as granulated blast-furnace slag, natural pozzolans, fly ash, and silica fume, which react with water to generate a dense microstructure; and inert filler materials, such as limestone, which have low or no reaction and only serve as fillers in the production of these new portland cement formulations.

Depending on the percentage contents of these six components, six different types of cement are defined, which are listed in the NMX-C-414-ONNCCE standard³: OPC (abbreviated as CPO in Spanish), portland pozzolana cement (abbreviated as CPP in Spanish), granulated blast-furnace slag portland cement (abbreviated as CPEG in Spanish), composite portland cement (abbreviated as CPC in Spanish), silica fume portland cement (abbreviated as CPS in Spanish), and granulated blast-furnace slag cement (abbreviated as CEG in Spanish). When these new CPCs have one addition, besides clinker, they are called binary cements. When they have two additions besides clinker, they are called ternary cements. Currently, only one type is commercially available in the Mexican cement market (namely, CPC), and the other five cement types are made as special orders, including OPC.

RESEARCH SIGNIFICANCE

The cement industry in Latin America has been modifying the blends of its cement by reducing the clinker content in its new formulations as the main solution to mitigate greenhouse gas emissions and make the construction industry more sustainable. However, these modifications could affect the mechanical performance and durability of concrete structures, as many design methodologies assume that clinker is

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the only component of cement (that is, concrete mixture design following the ACI PRC-211.1 methodology). Therefore, it is necessary to determine the performance of cement-based materials when manufactured with binary and ternary cements from a more holistic view and to understand the real sustainability of the construction industry. This investigation evaluates the mechanical and durability performances of mortars made with binary cements made with portland cement and a high content of inert limestone addition: between approximately 20 and 30% by clinker replacement.

EXPERIMENTAL PROCEDURE

Material properties

Three different Mexican cement brands (CA, CT, and CM), all of the same type of cement as defined in the Mexican standard,³ CPC 30 R, were used to fabricate the mortar mixtures evaluated. Along with the type of cement, the name is defined by a number (30 in this case), which is the cement's strength at 28 days (in MPa) obtained from mortar cubes (30 MPa [4350 psi]); and the letter R, which means it is an early-strength cement, reaching 20 MPa (2900 psi) at 3 days of curing.

The cement compositions were not available because all three were purchased in a regular construction materials store in a 50 kg (111 lb) paper bag pack. Therefore, characterization of the three cement brands was obtained only from their densities and loss on ignition (LOI), following the ASTM C114-23 procedure.⁴

The sand used was from a local quarry. Because its grain-size distribution did not follow ASTM C33/C33M-23 minimum and maximum limits,⁵ it was necessary to sieve the sand to obtain a granulometry that complied with the standard.

Mortar mixtures were prepared following ASTM C1437-20⁶ and ASTM C109/C109M-21⁷ methods to estimate the amount of water for normal flow and cube fabrication, respectively. A total of 15 mortar cubes were fabricated for each cement brand (CA, CT, and CM). The mortar mixture proportions were the same for the three cement brands: a 0.68 water-cement ratio (w/c) and a 0.36 cement-sand ratio (c/s).

The mortar cubes were demolded after 24 hours of fabrication and maintained in a laboratory environment ($\sim 60\%$ relative humidity [RH] and $21 \pm 3^\circ\text{C}$ [$70 \pm 5.4^\circ\text{F}$]) for 28 days, following real-life curing times where structural elements are regularly demolded 24 hours after casting and no water curing is applied to the element.

Natural exposure conditions

After reaching 28 days of age, three mortar cubes from each mixture were selected to perform the physical/mechanical/chemical testing. The remaining mortar cubes (12 specimens) were placed outside the laboratory in an uncovered area close to the parking garage of the laboratory facilities for up to 365 days. The mortar cubes were exposed in this natural environment, and three of them were selected to perform the physical/mechanical/chemical testing at the defined dates of 90, 180, and 365 days.

Mortar physical testing

When the testing dates were reached, three cubes were selected to perform testing. Testing started by measuring the cube's mass and electrical resistivity (ρ). ρ was estimated following the ASTM C1876-19 procedure.⁸ Then, the cubes were placed in a high-humidity chamber ($\sim 95\%$ RH) at room temperature, and mass and ρ measurements were monitored once a day until constant mass was reached (mass difference between consecutive measurements should be $<0.5\%$). The final constant mass was defined as saturated mass (m_S). Once m_S was recorded, the mortar cubes were placed in a hydrostatic balance to get the saturated-submerged mass (m_{SS}).

After obtaining m_S and m_{SS} , the cubes were placed in an oven at a temperature of approximately 50°C (122°F) to dry them until constant mass was reached. This third mass was defined as dry mass (m_D). Once the three masses were obtained, the total void content was estimated from Eq. (1)

$$\%TV = 100 \cdot (m_S - m_D) \cdot (m_S - m_{SS})^{-1} \quad (1)$$

After obtaining m_D , the cubes were coated with paraffin on four of the six faces, and the initial dry mass with the paraffin-covered faces was measured (W_0). Each cube was placed on a wet sponge inside a flat-bottomed container, ensuring that the water level was only 5 mm (0.2 in.) above the lower edge of the cube, and the container was covered to avoid water evaporation. To maintain the water level of 5 mm (0.2 in.) above the lower edge of the cube, periodic additions of water were required. Changes in cube weight per exposed unit area ($[W_t - W_0] \cdot A_0^{-1}$) were recorded (A_0 is the cube's cross-sectional area) at time intervals of a few minutes during the first 8 hours, and then six times daily thereafter until reaching constant mass. The absorption coefficients were calculated using the following equations (average values of three cubes per mixture tested)

$$m = t_n \cdot z^{-2} \quad (2)$$

$$k = (W_t - W_0) \cdot A_0^{-1} \cdot t^{-1/2} \quad (3)$$

$$\varepsilon_{eff}(\%) = 0.001 \cdot k \cdot m^{-1/2} \quad (4)$$

where the coefficient k (capillary absorption) is evaluated as the slope of the linear region of the graph $(W_t - W_0) \cdot A_0^{-1}$ versus $t^{1/2}$; the coefficient m (water penetration resistance) is determined by calculating the time t_n required for water to reach the cube's upper surface (in the present case, when $z = 5$ cm [2 in.]); and the effective capillary porosity (ε_{eff}) is then estimated with the coefficients k and m . Using the coefficient m , capillary sorption (S) was calculated as

$$S = m^{-1/2} \quad (5)$$

Mortar mechanical testing

After water capillary absorption testing, the compressive strength test was performed with the same cubes, following the ASTM C109/C109M-21 procedure.⁷ A universal servo-hydraulic testing machine (1000 kN [225 kip] nominal maximum capacity) was used to perform testing.

Table 1—Results for CPC density and LOI for three brands evaluated

	CA	CM	CT
Density, g/cm ³	2.97 ± 0.08	3.00 ± 0.09	2.91 ± 0.05
LOI, %	10.17 ± 0.08	12.37 ± 0.06	11.62 ± 0.08
LSC, %	23.36 ± 0.12	28.92 ± 0.09	27.01 ± 0.12

Table 2—Results for CPC's percentage fluidity and w/c to obtain percentage fluidity for three brands evaluated

	CA	CM	CT
Fluidity, %	113	115	114

The loading rate was ~0.25 MPa/s (~36 psi/s), and the output (maximum load) was recorded automatically on a computer.

Mortar chemical testing

After obtaining each mortar cube's physical and mechanical performance, the final crushed cube was used to perform the carbonation front estimates by using a pH indicator called phenolphthalein, following the NMX-C-515-ON-NCCE-2016 standard procedure.⁹ Using a calibrated caliper, at least four measurements were taken from the cube's original border (surface exposed to the environment) until the mortar changed color from gray (regular mortar color) to pink (pH indicator reaction when it is in contact with alkaline mortar). The average depths obtained from all the measurements taken were recorded for each mortar mixture.

RESULTS AND DISCUSSION

Cement characterization

The three brands used to fabricate the evaluated mortars have the same cement type (CPC 30 R) as the Mexican standard.³ Nevertheless, no additional information is given to the user (that is, the clinker's chemical composition and content, type of addition(s), and content use to be defined as a CPC type). The cement industry argues that this additional information can be provided to the end user if they ask for it and if a confidentiality agreement has been signed previously. Therefore, regular users who buy a bagged cement are not able to know the clinker's chemical phases in a particular cement they acquire because a typical chemical analysis following ASTM C114-23⁴ will not be possible to help users, because other additions are masking the clinker phase determination using regular Bogue calculations.¹⁰

Without any further information on the selected cements (type CPC 30 R was the only information available), the estimation of the cement's density and LOI was selected to differentiate the three cement brands used. Results are listed in Table 1. As observed from Table 1, the densities of the three cements varied, on average, between 2.91 and 3.0 g/cm³ (0.105 and 0.108 lb/in.³), and the LOI varied between 10.17 and 12.37%. There was no apparent correlation between density and LOI for these three cement brands, possibly because there are no significant differences between them in both powder properties.

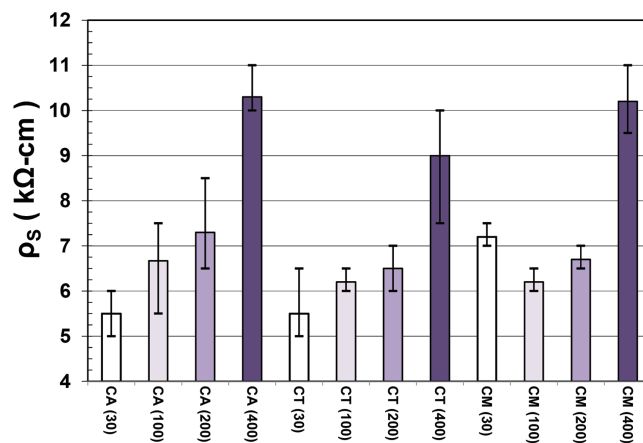


Fig. 1—Average saturated electrical resistivity (ρ_s) values for mortar mixtures evaluated at 30, 100, 200, and 400 days of age.

Based on the LOI experimental results, the limestone content (LSC) is estimated using Torres-Acosta et al.'s¹¹ approximation: $LSC (\%) = 1.864 \cdot (LOI \%)^{1.09}$ ($R^2 = 9.61$). Estimates are also included in Table 1. As observed from these LSC estimates, the three cement brands have between 23 and 29% LSC, which are values higher than the maximum accepted by the American Cement Association (ACA) of <15%.¹²

Mortar physical characterization

Mortar fluidity—Table 2 presents the percentage fluidity for the three cement brands evaluated. Mortar mixtures were prepared with w/c and c/s as constants (0.68 and 0.36, respectively); thus, mortars should perform similarly if their cements were also similar. Nevertheless, because of the mixture preparation, some percentage fluidity differences were observed, as shown in Table 2: the higher the cement's LOI, the greater the percentage fluidity. Still, the differences between the three cement brands were negligible: only 1% between them, and 2% between the minimum and maximum obtained values.

Saturated electrical resistivity (ρ_s)—Mortar ρ_s values for the three cement brands are presented in Fig. 1, obtained at each testing age (30, 100, 200, and 400 days). Results are presented as a bar histogram with the average value (averaged from three cubes) represented by the bar, and the minimum and maximum values represented by the limiting horizontal lines. Considering that the mortar cubes were cured only inside the molds for 24 hours and then were exposed to the laboratory environment ($60 \pm 5\%$ RH and $20 \pm 5^\circ\text{C}$ [$68 \pm 9^\circ\text{F}$] temperature) after the first 30 days, ρ_s values at 30 days did not show differences between the three cement brands.

Once the cubes were exposed to the unsheltered environment outside the laboratory, ρ_s values increased with exposure time for all cement brands. The increase in the average ρ_s also depends on the cement's LOI: a higher ρ_s difference for CA cement with the lowest LOI estimate was observed, as compared to CM cement's lower ρ_s with the highest LOI estimate. These differences are observed from the three different testing dates, with the highest ρ_s difference at 400-day values.

Interestingly, average ρ_S values at 100 and 200 days of exposure increased marginally, but the 400-day average value increased substantially compared to the first two dates. This performance difference was apparently due to the environment's humidity and rainfall increasing between the 200- and 400-day dates, as observed from the collected results of a meteorological test station located close to the exposure site. Data collected from the meteorological test station during the exposure time are presented in Fig. 2.¹³

The observed increase in ρ_S might be indicative that the three cement brands (CA, CT, and CM) continue their hydration, either due to the decrease in internal porosity or due to the mortar carbonation, which also may decrease the mortar's porosity, thus increasing ρ_S . This ρ_S performance will be corroborated with the other experimental data monitored (porosity decrease or carbonation increase) to determine which of the two phenomena affected ρ_S more.

Percentage of total void content (%TV)—The second physical property evaluated with the mortar specimens was

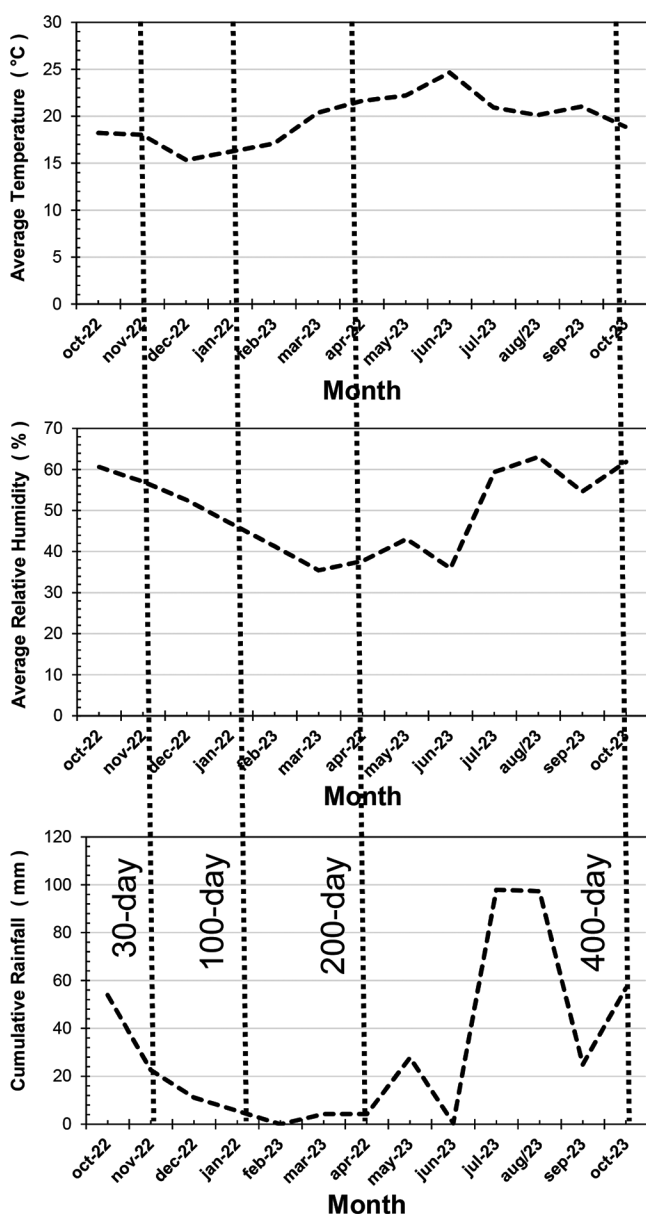


Fig. 2—Meteorological data for exposure site.¹³

the %TV, estimated with Eq. (1). The results of this test are presented in Fig. 3 using a similar format to ρ_S data: an average histogram of three specimens with minimum and maximum values. The performance observed is similar to that of ρ_S data: %TV remained constant between 30- and 200-day testing dates and decreased marginally at the 400-day testing date. This performance is again affected by either cement hydration or carbonation, decreasing the mortar's total porosity. There are also marginal differences between cement types: CM and CT mortars' average %TV values (22%) were marginally smaller (<5%) than those of CA (23%) at the 400-day testing date.

Capillary water absorption parameters—The third mortar physical property evaluated was the capillary porosity of the three cement brands, based on water absorption performance. After drying the mortar cube specimens to get m_D , they were used to determine Fagerlund's absorption parameters¹⁴ m , k , ε_{eff} , and S using Eq. (2) through (5), respectively. The mortar cubes were covered with paraffin on four of their six faces, leaving the bottom and top ones uncovered. Then, the cubes were placed in a 5 mm (0.2 in.) deep water tank, and absorption of the water started; thus, the mass change was monitored until a constant mass was reached for each mortar cube specimen.

Mass change monitoring was graphically represented using the square root of time ($t^{1/2}$) as the x-axis and $(W_t - W_0) \cdot A_0^{-1}$ as the y-axis to obtain k as the slope of the linear portion of the $(W_t - W_0) \cdot A_0^{-1}$ versus $t^{1/2}$ graph, following Eq. (3). This linear portion of the $(W_t - W_0) \cdot A_0^{-1}$ versus $t^{1/2}$ graph was extrapolated until intersecting the horizontal line extension of the constant mass values. The x-value of the intersection of these two lines was defined as $t_n^{1/2}$ following Fagerlund's procedure.¹⁴ The t_n value obtained was used in Eq. (2) to get the water penetration resistance (m) and, with m , the capillary sorption (S). Results obtained for Fagerlund's four parameters are shown in Fig. 4 through 7.

Figure 4 presents experimental results of one of the capillary absorption parameters, known as the water penetration strength (m) (in $\text{s} \cdot \text{m}^{-2}$ [$1 \text{ s} \cdot \text{m}^{-2} = 6.45 \times 10^{-4} \text{ s} \cdot \text{in.}^{-2}$]). As observed from this figure, m increased with exposure

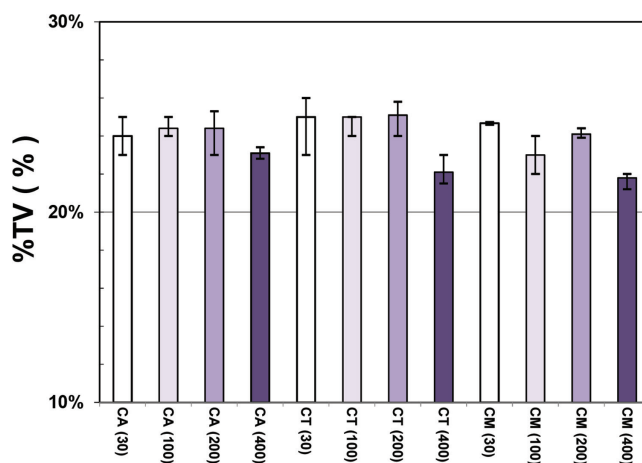


Fig. 3—Average percentage of total void content (%TV) values for mortar mixtures evaluated at 30, 100, 200, and 400 days of age.

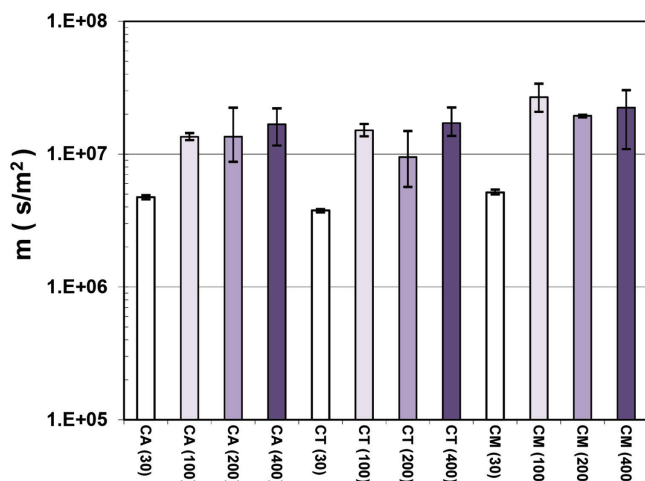


Fig. 4—Average water penetration resistance (m) values for mortar mixtures evaluated at 30, 100, 200, and 400 days of age.

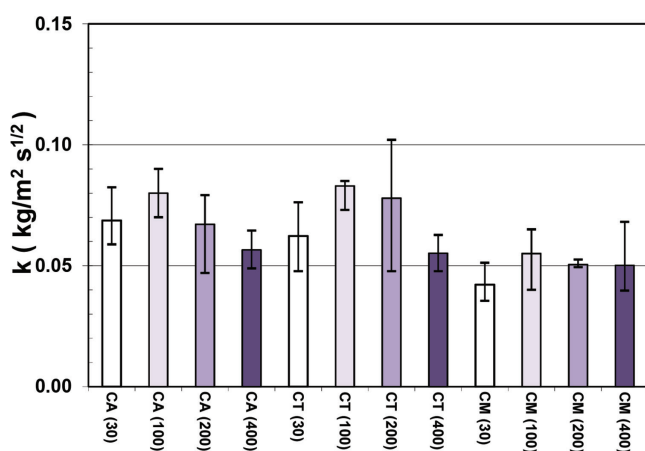


Fig. 5—Average capillary absorption coefficient (k) values for mortar mixtures evaluated at 30, 100, 200, and 400 days of age.

time for all mortar mixtures tested. A marginal m increase was observed for higher-LOI cements at 400-day exposure: $1.68 \times 10^7 \text{ s}\cdot\text{m}^{-2}$ for CA, $1.71 \times 10^7 \text{ s}\cdot\text{m}^{-2}$ for CT, and $2.2 \times 10^7 \text{ s}\cdot\text{m}^{-2}$ for CM.

The capillary absorption coefficient k (in $\text{kg}\cdot\text{m}^{-2}\cdot\text{s}^{-1/2}$ [$1 \text{ kg}\cdot\text{m}^{-2}\cdot\text{s}^{-1/2} = 1.423 \times 10^{-3} \text{ lb}\cdot\text{in}^{-2}\cdot\text{s}^{-1/2}$]) is presented in Fig. 5 for the evaluated mortar mixtures. A similar performance was obtained for the parameter k as for the parameter m : k decreased with exposure time, and a marginal decrease of k was observed for higher-LOI cements at 400-day exposure: $5.66 \times 10^{-2} \text{ kg}\cdot\text{m}^{-2}\cdot\text{s}^{-1/2}$ for CA, $5.52 \times 10^{-2} \text{ kg}\cdot\text{m}^{-2}\cdot\text{s}^{-1/2}$ for CT, and $5.02 \times 10^{-2} \text{ kg}\cdot\text{m}^{-2}\cdot\text{s}^{-1/2}$ for CM.

Experimental results obtained for the effective capillary porosity, ϵ_{eff} , are shown in Fig. 6. A similar performance was observed for this mortar's physical parameter: ϵ_{eff} decreased with exposure time, and a marginal decrease of ϵ_{eff} was observed for higher-LOI cements at 400-day exposure: 22.7% for CA, 22.4% for CT, and 22.0% for CM.

The final parameter estimates for mortar's sorptivity S are shown in Fig. 7. Again, S decreased with exposure time, and a marginal decrease of S was observed for higher-LOI

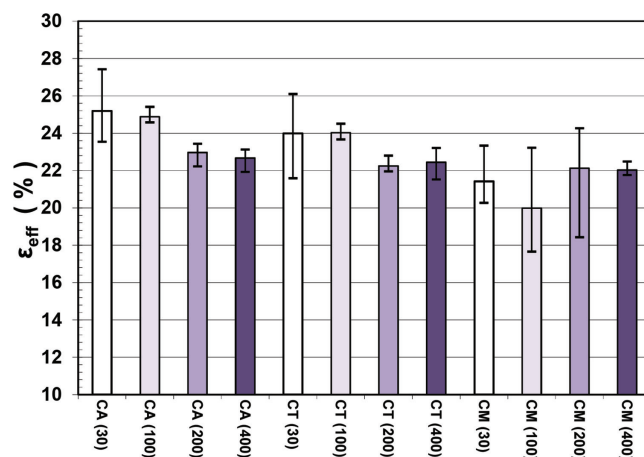


Fig. 6—Average capillary porosity (ϵ_{eff}) values for mortar mixtures evaluated at 30, 100, 200, and 400 days of age.

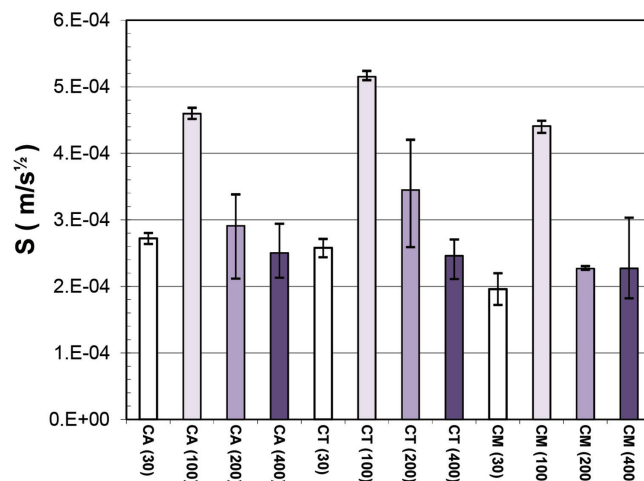


Fig. 7—Average sorptivity coefficient (S) values for mortar mixtures evaluated at 30, 100, 200, and 400 days of age.

cements at 400-day exposure: $2.5 \times 10^{-4} \text{ s}\cdot\text{m}^{-1/2}$ for CA, $2.46 \times 10^{-4} \text{ s}\cdot\text{m}^{-1/2}$ for CT, and $2.27 \times 10^{-4} \text{ s}\cdot\text{m}^{-1/2}$ for CM ($1 \text{ s}\cdot\text{m}^{-1/2} = 0.16 \text{ s}\cdot\text{in}^{-1/2}$).

Experimental results obtained for this physical parameter (capillary absorption) did not show important differences between the evaluated mortars. Nevertheless, the slight performance improvement observed could be due to, again, the continuous cement hydration due to the increase in the ambient humidity and rainfall between the 200- and 400-day time lapse.

Mortar mechanical characterization: compressive strength (f_c)

The next mortar property evaluated was the mechanical compressive strength f_c . Values of the average f_c (with minimum and maximum values) for all mortar mixtures evaluated are presented in Fig. 8. As observed from Fig. 8, no mortar mixture reached the nominal 28-day f_c (or f'_c) of 30 MPa (4350 psi) as defined in the cement type CPC 30 R for the three brands evaluated. Apparently, this was mainly because the mortar cubes had only 1 day of curing and not 28 days of curing, as defined in the standardized procedure

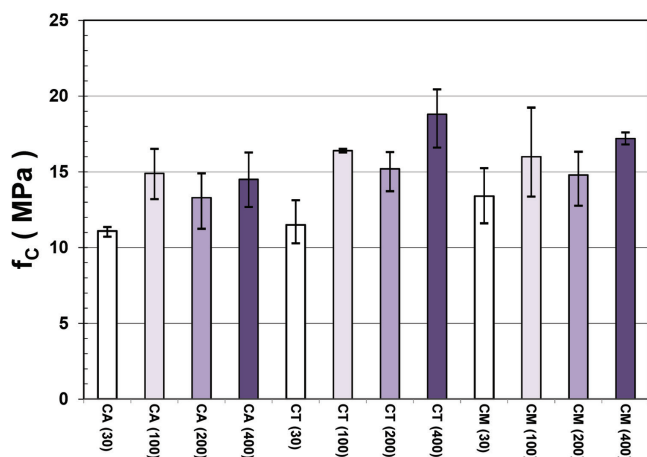


Fig. 8—Average compressive strength (f_c) values for mortar mixtures evaluated at 30, 100, 200, and 400 days of age.

to determine the cement's f_c' from NMX-C-414-ONNCCE³ and ASTM C109/C109M-21.⁷

As observed from Fig. 8, f_c values for the three cement brands were similar up to 200 days, where little differences were observed for CA and CT 30-day f_c , and CM reached a marginally higher value at the same 30-day age (only 20% more than the other two cement brands).

The 400-day f_c values increased between 15 and 19 MPa (2175 and 2755 psi) on average, which were 25 to 35% higher than the 30-day f_c obtained. As observed, there is no correlation between the cement's LOI and f_c , because CA (with the lowest LOI value) had the lowest 400-day f_c , and CT mortar, with the intermediate LOI values, had the highest 400-day f_c . CM's average 400-day f_c value, with the highest LOI value, was higher than CA but lower than CT at the same testing time. Apparently, there is another parameter that affects mortar's f_c : the carbonation of the mortar, which will be discussed in the next section.

Mortar chemical characterization: carbonation

The last property evaluated with the tested mortars was the pH (chemical) change of the material due to carbonation. At all testing times, after crushing the mortar in the compressive strength tests, cubes were sprayed with phenolphthalein, and the carbonation depths (x_{CO_2}) were recorded and averaged (average of at least seven measurements). The results are presented in Fig. 9 (with the same format as the previous test data).

As observed from Fig. 9, the average 30-day x_{CO_2} for all mortar mixtures evaluated was quite the same, but after placing the cubes outside the laboratory, the carbonation front increased with exposure time. The x_{CO_2} values obtained on average were directly proportional to cement's LOI, as observed from Fig. 9. As an example, 400-day exposure data obtained were as follows: the CT mortar mixture with 12.4% LOI had 17.5 mm (0.69 in.) average x_{CO_2} ; CM with 11.6% LOI had 16 mm (0.63 in.) x_{CO_2} ; and the CA mixture with 10.2% LOI had 11.5 mm (0.45 in.) x_{CO_2} . This corroborates test results from a previous investigation,¹⁵ which was performed with concrete specimens exposed to a natural urban environment in the city of Monterrey, Mexico, where

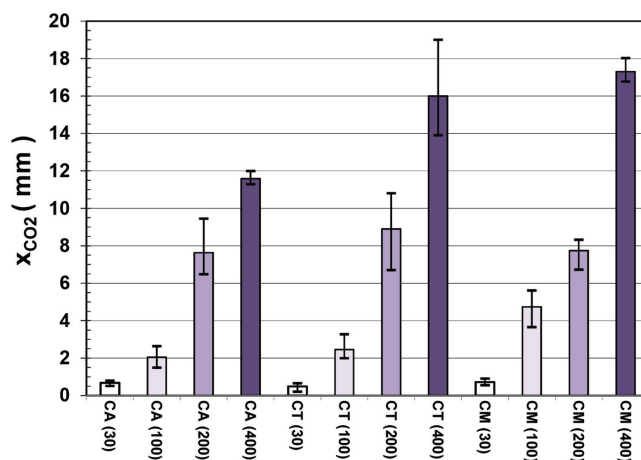


Fig. 9—Average carbonation depth (x_{CO_2}) values for mortar mixtures evaluated at 30, 100, 200, and 400 days of age.

two cement types were used with 2.55% and 8.2% LOI values: the higher the cement's LOI, the higher the x_{CO_2} value obtained due to such natural exposure.

Because all three mortars were fabricated with a similar w/c , this carbonation performance was not related to the amount of water used during the mortar mixture. Nevertheless, this performance was apparently due to the amount of limestone filler added to the portland cement by the cement manufacturer, because the higher the LOI, the more LSC.¹¹

As observed from x_{CO_2} and f_c experimental data obtained from the tested mortars, the increase in f_c was related not only to the mortar's change in porosity due to continuing hydration of the cement in the mortar (change in %TV and ε_{eff}), but also due to the mortar's carbonation. Therefore, a combined effect between cement's continuing hydration and carbonation was also observed with the ρ_s estimates, as observed in Fig. 1, where CA, which had the lowest LSC estimate, had the highest ρ_s . The following ρ_s value was obtained from CM mortar, which had the highest average x_{CO_2} of all three mortars tested. Finally, CT mortar achieved the lowest average ρ_s and the intermediate x_{CO_2} average value, indicating that both porosity and cement's alkalinity interacted to have a composite performance compared to CA and CM.

Using the x_{CO_2} data obtained, a composite plot of x_{CO_2} versus $t^{1/2}$ was prepared and presented in Fig. 10. The t values were estimated starting at 30 days of age, giving exposure times of 70, 170, and 370 days at the ages of 100, 200, and 400 days, respectively. The slopes of the three data sets were estimated and were defined as the carbonation coefficient k_{CO_2} , also known as the material's carbonation rate. As observed from Fig. 10, k_{CO_2} increased from CM > CT > CA, following the same pattern observed with x_{CO_2} : the higher the cement's LOI (or limestone addition), the higher the mortar's k_{CO_2} .

Based on the experimental results obtained, an empirical equation was obtained to estimate k_{CO_2} for cement-based mortars if composite cements between 10 and 12.5% LOI cement contents are used: $k_{CO_2} = 0.25 \cdot LOI^{1.84}$, with $R^2 = 0.981$. This empirical correlation needs to be corroborated

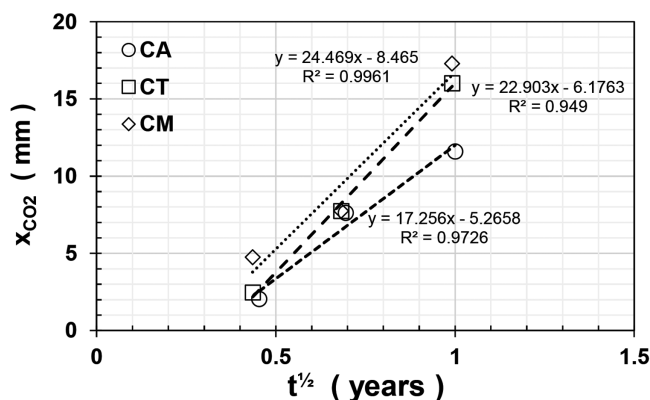


Fig. 10— x_{CO_2} versus $t^{1/2}$ correlation for three mortar mixtures. Includes tendency lines and their correlation coefficients.

with additional data from varied LOI values, and not only from the 20 to 30% range this investigation evaluated.

Previous investigations have shown experimental k_{CO_2} estimates between 4.1 and 4.4 mm/y^{1/2} for OPC-based mortars, without limestone additions, exposed to a similar natural urban environment for almost 14 years in Querétaro City.¹⁶ The mortar cubes used in this investigation also had a 28-day curing period before CO₂ natural exposure for almost 14 years. This important difference between the k_{CO_2} experimental results obtained previously and those from this investigation may warrant composite limestone cement users to reconsider its applicability in urban-like environments. Therefore, the need to improve mortar or concrete performance in such environments by warranting long periods of curing for such materials, to decrease the material porosity, is recommended.

General discussion

The idea of 1-day curing in the mold for the mortar evaluated was to simulate real-life applications, where little or no curing of the cement-based material (mortar or concrete) is considered. With this condition, the evaluation was performed to determine if a particular environment's humidity, temperature, and rainfall could help to improve the physical/mechanical/chemical performance of the mortars evaluated.

As observed from the physical properties of the mortars evaluated, ρ_s , %TV, and ε_{eff} improved with time, with some variations depending on the cement's LOI: the mortar's ρ_s improved with exposure time, with higher improvements if low-LOI cement was used in the mortar; %TV and ε_{eff} were almost the same regardless of the cement's LOI in the mortar.

From the mortar's mechanical performance, the improvement observed in Fig. 8 might be due to the cement's hydration and mortar carbonation, which both helped to decrease porosity of the material and thus increase its f_c .

Based on the mortar's chemical performance, there is an evident detriment to the durability of cement-based mortars if clinker content is reduced and curing of the material is not considered before it is exposed to the environment: k_{CO_2} values were between 17.3 and 24.5 mm/y^{1/2} (0.68 and 0.97 in./y^{1/2}). If, for example, a reinforcing bar is embedded

in a mortar like the ones evaluated, with k_{CO_2} values between 17.3 and 24.5 mm/y^{1/2} (0.68 and 0.97 in./y^{1/2}), and with a typical mortar cover depth of 2.5 cm (1 in.), the time for full mortar carbonation will be between 1.0 and 2.1 years only. This means that the corrosion initiation, and thus the service life of the hypothetical reinforced mortar element, will be between 1 and 2 years. More experimental data are needed to evaluate the impact of LSC on mortar durability in a CO₂ environment, which will be presented soon.

In the case of marine environment exposure, the chloride diffusivity of the evaluated mortars can be estimated from empirical correlations obtained in previous investigations (based on mortar¹⁷ and concrete¹⁸ experimental data) between ρ_s and a durability index called the rapid chloride permeability (RCP), obtained following the ASTM C1202-22 procedure.¹⁹ An analytical correlation between RCP and ρ_s was determined in Torres-Acosta et al.¹⁸ for concrete, which was Q (coulombs) = 20,400 · ρ_s^{-1} (kΩ·cm). In Visairo-Méndez et al.,¹⁷ an empirical equation between RCP and ρ_s was also determined for mortar specimens, which was Q (coulombs) = 23,320 · $\rho_s^{-1.06}$ (kΩ·cm).

As observed, both correlations are almost identical; thus, it is possible to use either one with very little difference in the estimated results. Using the correlation obtained from mortar specimens of Visairo-Méndez et al.,¹⁷ ρ_s values for the evaluated mortars in this investigation (between 9 and 10 kΩ·cm) may reach Q values between 2270 and 2030 coulombs. According to Visairo-Méndez et al.'s¹⁷ evaluation criteria, mortars with $Q > 2001$ coulombs are considered highly permeable for chlorides, and $2000 < Q < 1000$ coulombs are considered as mortars with intermediate permeability for chlorides. Therefore, mortars fabricated with this cement (CPC 30 R) with LOI between 10 and 12% may not be suitable if they are placed in a chloride-laden environment.

CONCLUSIONS

Based on the experimental results obtained in the present investigation using mortar cubes with 1 day of curing and high-percentage loss on ignition (LOI) (which, in turn, means that they have high limestone additions from clinker replacement), the following conclusions are drawn:

Three different cement brands were used with the same cement type (CPC 30 R), in accordance with the Mexican cement standard NMX-C-414-ONNCCE, and were named CA, CT, and CM. The chemical composition of the three cements was not given by the manufacturers, as they argue that it is a registered trademark, and the information is only given if the user asks for it. Therefore, the cements were characterized only based on their density and percentage LOI. Average density values were between 2.91 and 3.0 g/cm³ (0.105 and 0.108 lb/in.³), and average LOI values were between 10.2 and 12.4%, which means that the three cements have high inert limestone filler contents (the estimates were 23% for CA, 29% for CT, and 27% for CM).

After mortar fabrication, cubes remained in their standardized molds for 24 hours, and after demolding them, they were placed in a laboratory environment for 28 days before ambient exposure. This treatment of the mortar cubes was considered to follow typical practices in the construction

industry, where little or no curing period is considered in real-life mortar or concrete elements.

Physical characterization of the evaluated mortars included saturated electrical resistivity (ρ_s), percentage of total void content (%TV), and water capillary absorption or effective capillary porosity (ε_{eff}). All physical properties of the three mortars evaluated improved with exposure time: ρ_s increased, and %TV and ε_{eff} decreased with exposure time. This physical improvement was more noticeable when ambient relative humidity (RH) and rainfall increased between the 170- and 370-day exposure periods. The 370-day average values were: $7.5 \text{ k}\Omega\cdot\text{cm} < \rho_s < 11 \text{ k}\Omega\cdot\text{cm}$; $22\% < \%TV < 23\%$; and $22\% < \varepsilon_{eff} < 22.4\%$. Marginal differences were observed among the three mortars evaluated.

Mechanical characterization was also evaluated for the three mortar types by obtaining the compressive strength (f_c) at four different ages: 30, 100, 200, and 400 days. The f_c values were between 10 and 20 MPa (1450 and 2900 psi), improving all three mortar mixtures after 370 days of ambient exposure. Similar to the physical properties, f_c might be improved due to continuous cement hydration with exposure to higher humidity and rainfall. However, this improvement was also due to mortar carbonation, which reduced the mortar's porosity.

Chemical characterization from carbonation testing showed high carbonation rates (k_{CO_2}) for the three mortars, with CM mortar having the highest rate. A good correlation was observed between k_{CO_2} and the cement's LOI: the higher the cement's LOI, the higher the mortar's k_{CO_2} . Estimates of k_{CO_2} were $17.3 \text{ mm/y}^{1/2}$ for CA (LOI = 10.2%), $22.9 \text{ mm/y}^{1/2}$ for CT (LOI = 11.6%), and $24.5 \text{ mm/y}^{1/2}$ for CM (LOI = 12.4%). This performance corroborates the hypothesis that clinker replacement with limestone in portland cements decreases the durability of cement-based materials such as mortar and concrete in a CO_2 -laden environment.

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Factors Affecting Bridge Deck Cracking: Crack-Reducing Technologies, Paste Content, and Construction Practices

by Muzai Feng, David Darwin, and Rouzbeh Khajehdehi

Crack densities obtained from on-site surveys of 74 bridge deck placements containing concrete mixtures with paste contents between 22.8 and 29.4% are evaluated. Twenty of the placements were constructed with a crack-reducing technology (shrinkage-reducing admixtures, internal curing, or fiber reinforcement) and 54 without; three of the decks with fiber reinforcement and nine of the decks without crack-reducing technologies involved poor construction practices. The results indicate that using a concrete mixture with a low paste content is the most effective way to reduce bridge deck cracking. Bridge decks with paste contents exceeding 27.3% had a significantly higher crack density than decks with lower paste contents. Crack-reducing technologies can play a role in reducing cracking in bridge decks, but they must be used in conjunction with a low-paste-content concrete and good construction practices to achieve minimal cracking in a deck. Failure to follow proper procedures to consolidate, finish, or cure concrete will result in bridge decks that exhibit increased cracking, even when low paste contents are used.

Keywords: bridge deck; consolidation; construction practices; crack-reducing technologies; cracking; fiber-reinforced concrete; internal curing (IC); lightweight aggregate; shrinkage-reducing admixtures (SRAs).

INTRODUCTION

The cracking of reinforced concrete in bridge decks accelerates corrosion of reinforcing steel and freezing-and-thawing damage of concrete, reducing the service life and increasing the maintenance costs (Mindess et al. 2003). According to a national survey led by the Federal Highway Administration, bridge deck cracking, corrosion of reinforcing steel, and freezing-and-thawing damage of concrete were among the top forms of distress in bridge decks recognized by state departments of transportation (DOTs) (Triandafilou 2005).

Methods to reduce bridge deck cracking include lowering the paste content of the concrete (the volume percentage of cementitious material and water) and enforcing good construction practices, such as minimal finishing, thorough consolidation, and prompt application of curing (Schmitt and Darwin 1995, 1999; Darwin et al. 2004; TRB Committee AFN10 2006; Deshpande et al. 2007; Radlińska and Weiss 2012; Khajehdehi and Darwin 2018). These methods, however, are not always used (McLeod et al. 2009; Khajehdehi and Darwin 2018; Lafikes et al. 2018). Additionally, innovative technologies that promise to reduce cracking in bridge decks, including shrinkage-reducing admixtures (SRAs), fiber reinforcement, and internal curing (IC), have become increasingly popular.

Theoretically, SRAs and IC reduce bridge deck cracking by reducing concrete shrinkage (Berke et al. 2003; Browning et al. 2011), while fiber reinforcement does so by increasing the toughness of concrete (Gopalaratnam et al. 1991). The effectiveness of these crack-reducing technologies is supported by several laboratory studies (Voigt et al. 2004; Naaman et al. 2005; Brown et al. 2007; Bentz and Weiss 2011; Delatte and Crawl 2012; Pendergrass and Darwin 2014; Al-Qassag et al. 2015, 2022; Khajehdehi et al. 2018). The evaluation of crack-reducing technologies based on long-term observations of in-service bridge decks, on the other hand, is limited and sometimes contradicts laboratory findings. Based on a survey involving 116 bridge decks, Delatte and Crawl (2012) reported that synthetic fibers do not prevent cracking in bridge decks; the cracking performance of individual bridge decks, however, was not reported. Streeter et al. (2012) evaluated the cracking performance of three decks with IC provided by pre-wetted fine lightweight aggregate and two decks without IC and found that, with or without IC, all five decks performed similarly and exhibited none or minimal cracking (one crack per deck) when inspected at an age of 1 year or less. Polley et al. (2015) compared the cracking performance of six bridge decks with SRAs and found that, at ages of 10 to 19 months, decks with SRAs exhibited similar or higher cracking than decks without SRAs.

The significance of construction practices used by contractors, especially the methods used to consolidate, finish, and cure concrete, on the cracking performance of bridge decks is largely underappreciated. The need for proper construction practices has been highlighted by McLeod et al. (2009), Darwin et al. (2016), and Khajehdehi and Darwin (2018), who illustrated quantitatively that when contractors fail to properly consolidate, finish, or cure the concrete, the resulting bridge decks will exhibit high crack densities.

To further examine the long-term effects of crack-reducing technologies, paste content, and construction practices on bridge deck cracking, cracking performance was evaluated quantitatively following a consistent on-site survey method for 20 bridge deck placements incorporating an SRA, IC, or fiber reinforcement and 54 deck placements without these technologies. All but two of the deck placements were

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surveyed at least twice. Because the crack density of bridge decks increases with age (Darwin et al. 2004; Lindquist et al. 2005; Yuan et al. 2011; Pendergrass and Darwin 2014), crack densities at 36 months of age are compared for all the decks to provide a consistent measure of cracking.

RESEARCH SIGNIFICANCE

This paper illustrates the influence of three crack-reducing technologies—IC, SRAs, and fiber reinforcement—on the crack performance of in-service bridge decks. The impact of poor construction practices on the cracking performance of bridge decks is also examined. The method to quantify bridge deck cracking used in this paper can also be used to quantify the effects of other crack-reducing technologies and factors that may contribute to the cracking performance of bridge decks.

METHODOLOGY

Quantification of cracking in bridge decks (crack survey)

The initial crack survey on a bridge deck is typically performed at a deck age of 12 months and is followed by surveys at approximately 1-year intervals thereafter. Because some personnel changes are made each year, a standard procedure was developed to consistently quantify the extent of the cracking of in-service bridge decks. The survey procedures outlined in this section have been successfully applied since 1993 (Schmitt and Darwin 1995, 1999).

Prior to a survey, the weather forecast is checked to ensure that the air temperature during the survey will be 60°F (15.5°C) or higher to help ensure that the survey personnel are physically comfortable. The day should be at least mostly sunny, and the deck must be completely dry. A survey is deemed invalid and must be repeated unless these conditions are satisfied.

A map of the bridge deck in plain view with a scale of 1:120 (1 in. = 10 ft [25 mm = 3 m]) is printed along with a second copy that includes grid lines spaced at 5 x 5 ft (1.52 x 1.52 m). During the survey, the map with grid lines is

aligned with and placed under the map without grid lines so that the surveyor can use the former as a reference to locate any point of the bridge deck when marking cracks. A north arrow is also shown on the map to help the surveyor orient the map while on the deck.

Traffic control during surveys is provided by the state DOT to ensure the safety of the surveyors. At least one lane is closed to traffic at a time. Grid points spaced at 5 x 5 ft (1.52 x 1.52 m), matching the grid lines on the maps, are drawn on the deck surface using sidewalk chalk.

To find cracks on the deck surface, surveyors bend from the waist to lower their eyes to view the deck and walk in a zigzag pattern to ensure the entire deck is checked. Each section of the bridge deck is checked by at least two surveyors. Cracks visible at waist level (width $\approx$ 0.003 in. [0.08 mm] and wider) are marked using sidewalk chalk on the deck. When marking a crack, the surveyor may bend closer or crouch down to mark the crack to its end, including the portions of the crack with a width that is too narrow to be seen at waist height. When crouching down to mark a crack, the surveyor can sometimes see other cracks. If such cracks are not connected to the crack currently being marked, they are not to be marked unless they can be seen when the surveyor is bending at waist height. A surveyor then draws the marked cracks on the scaled bridge map, using the grid lines on both the bridge deck and the map to keep track of the location and length of cracks.

To calculate crack density, the maps are scanned using computer-aided drafting software, where the individual and total crack lengths are calculated for the entire deck, each span, and in the cases where the deck was constructed in multiple placements, each placement. The total length of the cracks is then divided by the area of the deck, span, or placement to obtain the crack density, which is reported in m/m^2 ($1 \text{ m}/\text{m}^2 = 0.025 \text{ in.}/\text{in.}^2$).

Bridge decks included for analysis

The bridge decks covered in this paper are listed in Tables 1 through 4. Table 1 summarizes the paste contents

Table 1—Paste content and construction issues of fiber and SRA decks*

Bridge deck placement	Paste content	Construction issue	Bridge deck placement	Paste content	Construction issue
KS-Fiber-1 p1 [†]	23.8%	No issue	KS-Control-5	24.7%	No issue
KS-Fiber-1 p2 [†]	23.8%	No issue	KS-Fiber-6	24.6%	No issue
KS-Fiber-2 p1 [†]	23.8%	No issue	KS-Control-6	24.6%	No issue
KS-Fiber-2 p2 [†]	23.8%	No issue	KS-Fiber-7	24.6%	No issue
KS-Fiber-3	22.2%	Loss of consolidation [‡]	KS-Control-7	24.6%	No issue
KS-Control-3	22.2%	Loss of consolidation [‡]	VA-SRA-1	28.2%	Not observed
KS-Fiber-2 p2 [†]	22.2%	Loss of consolidation	VA-SRA-2	27.1%	Not observed
KS-Fiber-4 p2 [†]	22.2%	Loss of consolidation	VA-SRA-3	27.0%	Not observed
KS-Control-4 p1 [†]	22.2%	Loss of consolidation [‡]	VA-SRA-4	27.3%	Not observed
KS-Control-4 p2 [†]	22.2%	Loss of consolidation [‡]	VA-Control	29.4%	Not observed
KS-Fiber-5	24.7%	No issue	—	—	—

*Construction observed unless noted; KS is bridge constructed in Kansas; VA is bridge constructed in Virginia.

[†]p is placement.

[‡]Deduced based on construction practice followed by contractor on KS-Fiber-4.

and construction issues for the decks with fiber reinforcement or an SRA cast in Kansas or Virginia and paired control decks constructed without the use of fibers or an SRA. For bridge decks constructed in multiple placements, each placement is analyzed separately. Ten bridge deck placements constructed in Kansas with fiber reinforcement (KS-Fiber-1 p1 through KS-Fiber-7) are compared with six deck placements constructed with similar construction procedures and materials, but without fiber reinforcement (KS-Control-3 through KS-Control-7). These decks had paste contents ranging from 22.2 to 24.7%. In cases where the fiber and control decks have the same number, the deck design and the contractors were the same on the two decks. Four bridge deck placements constructed in Virginia with SRAs (VA-SRA-1 through VA-SRA-4), with paste contents ranging from 27.0 to 28.2%, and one placement constructed using a similar method and materials but without an SRA (VA-Control), with a paste content of 29.4%, are also included, although there is not the same direct tie between the VA-SRA and VA-Control decks as between the KS-Fiber decks and corresponding KS-Control decks. The crack survey results for the bridge decks with fiber reinforcement or an SRA and the associated control decks are reported by Feng and Darwin (2020). When

possible, concrete placement operations were observed by the researchers. Decks whose construction was not observed are identified as “Not observed” in the “Construction issue” column of Table 1. During the construction of KS-Fiber-4, workers walked through the previously consolidated concrete before screeding, causing a loss of consolidation and increasing the likelihood of settlement cracking. Given that the same contractor constructed KS-Fiber-3, KS-Control-3, KS-Fiber-4, and KS-Control-4 within a short period (between March 13 and August 26, 2014), and these decks exhibited similar cracking patterns, it is deduced that the same construction procedures were followed for KS-Fiber-3, KS-Control-3, KS-Fiber-4, and KS-Control-4, and that all of these decks experienced a loss of consolidation.

Six bridge deck placements constructed in Indiana with internally cured concrete (IN-IC-1 through IN-IC-5) and one without IC (IN-Control) are listed in Table 2. IN-Control and IN-IC-1 had a paste content of 27.6%, while the paste contents of the other five placements with IC ranged from 24.6 to 25.9%. The crack survey results for the internally cured decks are reported by Lafikes et al. (2018). IN-IC-1 and IN-Control are supported by prestressed box girders; the other IC decks are supported by steel girders. IN-IC-5 was constructed in two placements; Placement 1 is the northbound lane and Placement 2 is the southbound lane (Fig. 1). A coal mine operated just south of the deck, and trucks carrying coal traveled frequently on Placement 1. As a result, the first placement of IN-IC-5 exhibited unusually high cracking (Fig. 1) and is excluded from the analysis.

Twenty-seven bridge deck placements surveyed by Schmitt and Darwin (1995, 1999), Miller and Darwin (2000), and Lindquist et al. (2005) are included to represent decks constructed without a crack-reducing technology and are listed in Table 3. Referred to as conventional decks, they were constructed between 1985 and 1995 following the then-current Kansas Department of Transportation (KDOT)

Table 2—Paste content of decks with or without IC*

Bridge deck placement	Paste content	Bridge deck placement	Paste content
IN-Control	27.6%	IN-IC-3	25.3%
IN-IC-1	27.6%	IN-IC-4	25.9%
IN-IC-2 p1†	24.6%	IN-IC-5 p2†	25.7%
IN-IC-2 p2†	25.2%	—	—

*Construction not observed; IN is bridge constructed in Indiana; IN-IC-5 p1 excluded from analysis.

†p is placement.

Table 3—Paste content of conventional decks*

Bridge deck placement	Paste content	Bridge deck placement	Paste content
KS-3-046 east deck	26.4%	KS-70-095 deck	27.2%
KS-3-046 west deck	26.4%	KS-70-103 right	27.2%
KS-3-046 center deck	25.7%	KS-70-103 left	27.2%
KS-75-044 deck	27.9%	KS-70-104 deck	27.2%
KS-75-045 deck	27.9%	KS-70-107 deck	27.2%
KS-89-204 deck	28.8%	KS-99-076 p4†	28.7%
KS-3-045 west deck	26.4%	KS-99-076 p5†	28.7%
KS-3-045 east deck	26.4%	KS-99-076 north (west lane)	28.7%
KS-3-045 west center deck	26.4%	KS-99-076 north (east lane)	28.7%
KS-3-045 center deck	26.4%	KS-99-076 p2†	27.9%
KS-3-045 east center deck	26.4%	KS-99-076 p3†	27.9%
KS-56-142 positive moment	26.5%	KS-89-208 deck	27.1%
KS-56-142 negative moment	26.5%	KS-56-49 deck	25.7%
KS-56-148 deck	27.2%	—	—

*Construction not observed; KS is bridge constructed in Kansas.

†p is placement.

Table 4—Paste content and construction issues of LC-HPC decks*

Bridge deck placement	Paste content	Construction issue	Bridge deck placement	Paste content	Construction issue
LC-HPC-1 p1 [†]	24.6%	No issue	LC-HPC-12 p1 [†]	24.3%	Loss of consolidation
LC-HPC-1 p2 [†]	24.6%	No issue	LC-HPC-12 p2 [†]	24.3%	Loss of consolidation
LC-HPC-2	24.6%	No issue	LC-HPC-13	24.1%	Loss of consolidation
LC-HPC-3	24.4%	No issue	LC-HPC-14 p1 [†]	24.4%	Insufficient consolidation, excessive finishing
LC-HPC-4 p2 [†]	23.4%	No issue	LC-HPC-14 p2 [†]	24.4%	Insufficient consolidation, excessive finishing
LC-HPC-5	23.9%	No issue	LC-HPC-14 p3 [†]	24.4%	Insufficient consolidation, excessive finishing
LC-HPC-6	24.4%	No issue	LC-HPC-15	22.8%	No issue
LC-HPC-7	24.6%	No issue	LC-HPC-16	22.8%	No issue
LC-HPC-9	24.2%	No issue	LC-HPC-17	24.6%	No issue
LC-HPC-11 [‡]	23.4%	No issue	—	—	—

*Construction observed; decks constructed in Kansas.

[†]p is placement.

[‡]Right eastbound driving lane of LC-HPC-11 excluded from analysis.

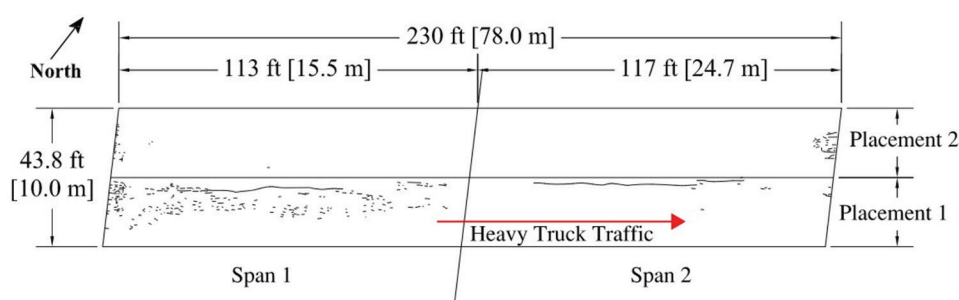


Fig. 1—Direction of truck traffic and uneven cracking on IN-IC-5.

specifications. The paste contents of the conventional decks ranged from 25.7 to 28.8%. This wide range allows the comparison of cracking performance among decks with different paste contents. All the conventional decks are supported by steel girders.

Nineteen bridge deck placements constructed in Kansas following low-cracking high-performance concrete (LC-HPC) specifications are included (Table 4). The LC-HPC decks did not use any crack-reducing technology other than specifications calling for low paste contents and quality construction procedures. The LC-HPC specifications and crack survey results are reported by Lindquist et al. (2008), McLeod et al. (2009), Yuan et al. (2011), Pendergrass and Darwin (2014), Bohaty et al. (2013), Alhmoode et al. (2015), and Darwin et al. (2016). These LC-HPC decks are supported by steel girders and have paste contents between 22.8 and 24.6%, as shown in Table 4. The first placement of LC-HPC-4 is not included in the analysis because the mixture proportions were changed during placement, and the actual paste content is not known (Lindquist et al. 2008). LC-HPC-8 and -10 are excluded because these decks are supported by prestressed girders. The right eastbound driving lane of LC-HPC-11 is excluded from the analysis because, like Placement 1 of IN-IC-5, heavy truck traffic in this lane resulted in unusually high cracking (Darwin et al. 2016).

Researchers from The University of Kansas observed the construction of all LC-HPC decks; the influence of construction practices can be evaluated by comparing the crack densities of the LC-HPC decks with and without issues. Similar to Fiber-4, during the construction of LC-HPC-12 and -13, workers walked through consolidated concrete, as shown in Fig. 2, leaving holes (footprints) in the surface. The concrete was not reconsolidated prior to finishing. This loss of consolidation increased the likelihood of settlement cracking directly over the reinforcing bars, as shown in Fig. 3 for the survey of Placements 1 and 2 of LC-HPC-12 at ages of 49.5 and 38.1 months, respectively. The same contractor constructed KS-Fiber-3, KS-Control-3, KS-Fiber-4, and KS-Control-4, listed in Table 1.

Especially problematic construction practices were observed during the construction of LC-HPC-14. The contractor used spring-loaded gang vibrators for consolidation; after consolidating the concrete, the vibrators were abruptly extracted from the concrete, leaving a series of holes in the deck surface (Fig. 4). These holes were later closed by the finishing machine rather than by reconsolidation, resulting in insufficient consolidation and thus increasing the likelihood of settlement cracking. Furthermore, the contractor devoted extra effort to bull floating and hand-finishing the deck surface and used a finishing aid; these steps resulted in bringing extra cement paste to

the surface and delayed the application of curing, both of which increased the likelihood of plastic shrinkage cracking. The issues observed during the construction of LC-HPC-12, -13, and -14 are described in greater detail by McLeod et al. (2009), Pendergrass and Darwin (2014), and Khajehdehi and Darwin (2018).

Crack densities at 36 months

The crack density of bridge decks increases with age. To eliminate the influence of deck age for the comparisons that follow, crack densities at a deck age of 36 months are used for the analyses.

To establish the 36-month crack density of the decks using the survey results, for decks surveyed both before and after 36 months of deck age, the crack density at 36 months is linearly interpolated using the two consecutive survey results. For decks whose latest survey was before 36 but no earlier than 30 months of age, the last survey result is treated as the 36-month crack density. Similarly, for decks whose earliest survey was after 36 but no later than 42 months of deck age, the first survey result is used to approximate the 36-month crack density. For decks whose first survey was after 42 months of age and those whose most recent survey was before 30 months of age, their 36-month crack densities

are linearly extrapolated based on the two available survey results closest to 36 months.

Exceptions were made for the following decks. KS-Fiber-1 and KS-Fiber-2 showed reduced cracking in later surveys (Survey 4, with deck ages between 43.2 and 45.5 months) due to scaling of the decks; the crack densities obtained in Survey 3, at ages of 31.7 to 34.0 months, respectively, are treated as the crack densities at 36 months. Decks VA-SRA-1, VA-SRA-2, and KS-75-045 were surveyed twice but showed lower crack densities in the second survey due to scaling of the concrete surface; the crack densities found in the first survey are used as the 36-month crack densities.

The 36-month crack densities of the bridge decks are listed in Table 5 for the decks containing fiber reinforcement; the decks containing SRAs, and their associated control decks; the decks with and without IC; and the LC-HPC decks. The survey results used to calculate 36-month crack densities are shown in Appendix A.

When analyzing the influence of crack-reducing technologies, paste contents, and construction practices, bridge decks with similar combinations of these variables are grouped, and the average crack densities of the groups are used for comparison. When comparing the average crack densities of two groups of bridge decks (X_1 and X_2), Student's t -test is used to verify whether the difference between X_1 and X_2 is due to the difference between the means of the two underlying populations from which the samples are drawn (μ_1 and μ_2) or merely due to the variance among samples in the same population. Student's t -test is a statistical hypothesis test to determine if the difference between two population means is statistically significant. The result of the t -test is expressed as the p -value, which is the probability that the difference between X_1 and X_2 is caused by chance and that there is, in fact, no difference between μ_1 and μ_2 . The value of p depends on the sample size, the difference between the population means, and the variance of the samples. In this paper, $p = 0.05$ is used as the threshold. Values of p less than or equal to 0.05 are taken as meaning that the difference between two means is statistically significant.



Fig. 2—Construction workers walking in previously vibrated concrete and causing loss in consolidation during construction of LC-HPC-12 Placement 1 (direction of placement is from left to right in picture).

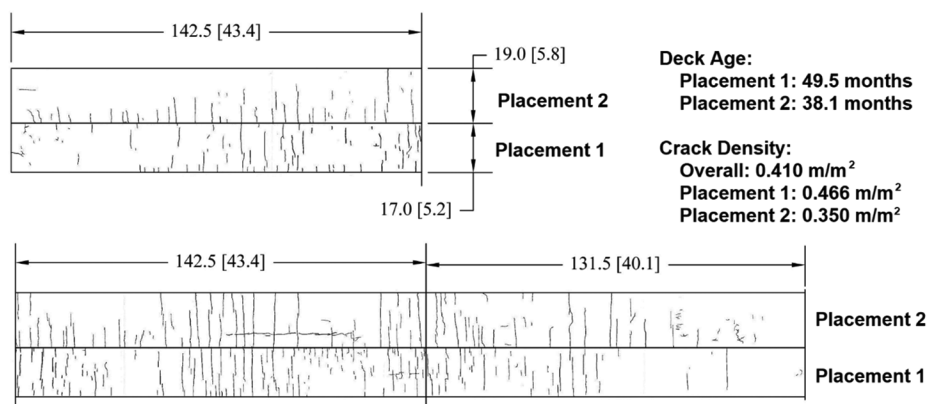


Fig. 3—Crack survey results of LC-HPC-12 at 49.5 and 38.1 months of age for Placement 1 and 2, respectively (Bohaty et al. 2013).



Fig. 4—Holes left by vibrators on LC-HPC-14 Placement 1 (McLeod et al. 2009).

DISCUSSION OF RESULTS

Influence of paste content

Khajehdehi et al. (2021) analyzed the crack densities of 40 bridge deck placements at 96 months of age and found that paste content has a dominant effect on cracking. Bridge decks with paste contents above 27.2% are associated with high cracking, while decks with paste contents of 26.4% or less consistently exhibit low cracking. For bridge decks with similar paste contents, crack density is affected only slightly, if at all, by other material properties, such as concrete slump, strength, and air content.

At 36 months of age, bridge decks with low paste contents are also significantly more likely to have low crack densities. Figure 5 shows the 36-month crack density of the bridge decks in this study without construction issues as a function of the paste content. As shown in the figure, decks with paste contents of 27.2% and less, in general, exhibited low crack densities at 36 months of age. For example, 30 of the 34 (88%) decks with a paste content of 27.2% or less and no crack-reducing technologies (in this case, meaning without IC, SRAs, or fibers) had a crack density below 0.3 m/m^2 . The probability of low cracking is further increased for those with paste contents of 26.5% or less: 25 out of the 27 (93%) of these decks had crack densities below 0.3 m/m^2 . Decks without crack-reducing technologies with paste contents greater than 27.3%, on the other hand, had a much higher probability of exhibiting high crack densities at 36 months of age. For example, eight out of the 11 (73%) decks without crack-reducing technologies with paste contents higher than 27.3% had crack densities above 0.4 m/m^2 . The paste content of the

concrete mixture used in bridge decks clearly appears to be a major, if not the major, governing factor on bridge deck cracking. A paste content of 26.5% or less greatly increases the likelihood that the deck will have a low crack density.

Influence of crack-reducing technologies

Compared to paste content, the use of the crack-reducing technologies of IC, SRAs, and fibers is a secondary factor in bridge deck cracking. As shown in Fig. 5, in general, the crack densities of bridge decks with IC, SRAs, or fiber reinforcement are generally, but not totally, comparable to those of decks without these technologies with similar paste contents, indicating that these technologies have less effect on the cracking performance of bridge decks. The influence of each crack-reducing technology on bridge deck cracking is analyzed separately in this section.

Shrinkage-reducing admixtures—Figure 6 compares the average 36-month crack densities of bridge decks in Virginia with an SRA and decks in Virginia and Kansas without an SRA. The bridges are grouped together based on the paste content in their concrete mixtures. Decks with paste contents higher than 27.3% (VA-SRA-1, VA-Control, and KS-W/O SRA [High Paste]) are categorized as High Paste, and decks with an SRA and without an SRA with paste contents equal to or less than 27.3% (VA-SRA [Low Paste] and KS-W/O SRA [Low Paste], respectively) are categorized as Low Paste. Error bars indicate the ranges of the crack densities for each bridge deck type.

The bridge decks with paste contents higher than 27.3%, including those with SRAs, showed noticeably higher crack densities at 36 months of age than those with 27.3% or lower paste contents. The average 36-month crack densities of the decks with 27.3% or lower paste contents, VA-SRA (Low Paste) and KS-W/O SRA (Low Paste), are 0.16 and 0.15 m/m^2 , respectively, significantly lower than those of the decks with paste contents higher than 27.3%, VA-Control, VA-SRA-1, and KS-W/O SRA (High Paste), which were 0.26 , 0.46 , and 0.73 m/m^2 , respectively. Thus, using concrete mixtures with low paste contents proves to be a more effective way to reduce bridge deck cracking than using an SRA for concrete with a higher paste content.

Among bridge decks with similar paste contents, there is not a clear correlation between crack density and the use of SRAs. As shown in Fig. 6, of the bridge decks with high paste contents, the VA-SRA-1 deck has a higher crack density than the VA-Control deck without an SRA (0.46 versus 0.26 m/m^2); its crack density, however, is lower than the average of the decks with paste contents higher than 27.3% and without an SRA in Kansas (KS-W/O SRA [High Paste], 0.73 m/m^2). On the other hand, for the decks with low paste contents, the three decks with an SRA in Virginia and the 34 decks without an SRA in Kansas have comparable average 36-month crack densities (0.16 m/m^2 and 0.15 m/m^2 , respectively), and the difference is not statistically significant ($p = 0.82$).

It has been reported that SRAs can reduce drying shrinkage of concrete by over 50% and delay the initiation of cracking (Shah et al. 1992; Folliard and Berke 1997; Nmai et al. 1998; Lindquist et al. 2008). The survey results presented

Table 5—Crack density of bridge decks at 36 months of age

Placement	36-month crack density, m/m ²	Placement	36-month crack density, m/m ²
KS-Fiber-1 p1*	0.112	KS-Fiber-1 p2*	0.220
KS-Fiber-2 p1*	0.127	KS-Fiber-2 p2*	0.456
KS-Fiber-3	0.285	KS-Control-3	0.233
KS-Fiber-4 p1*	0.709	KS-Fiber-4 p2*	0.431
KS-Control-4 p1*	0.766	KS-Control-4 p2*	0.393
KS-Fiber-5	0.061	KS-Control-5	0.052
KS-Fiber-6	0.011	KS-Control-6	0.011
KS-Fiber-7	0.004	KS-Control-7	0.033
VA-SRA-1	0.455	VA-SRA-2	0.344
VA-SRA-3	0.083	VA-SRA-4	0.056
VA-Control	0.232	IN-Control	0.236
IN-IC-1	0.181	IN-IC-2 p1*	0.000
IN-IC-2 p2*	0.020	IN-IC-3	0.017
IN-IC-4	0.057	IN-IC-5 p2*	0.032
KS-3-046 west deck	0.254	KS-3-046 center deck	0.042
KS-3-046 east deck	0.392	75-044 deck	0.165
75-045 deck	0.468	89-204 deck	0.736
KS-3-045 west deck	0.074	KS-3-045 east deck	0.078
KS-3-045 west center deck	0.178	KS-3-045 center deck	0.174
KS-3-045 east center deck	0.043	KS-56-142 positive moment	0.071
KS-56-142 negative moment	0.064	KS-56-148 deck	0.259
KS-70-095 deck	0.025	KS-70-103 right	0.253
KS-70-103 left	0.396	KS-70-104 deck	0.069
KS-70-107 deck	0.326	KS-99-076 p4*	0.872
KS-99-076 p5*	0.861	KS-99-076 north (west)	0.801
KS-99-076 north (east)	0.412	KS-99-076 p2*	1.536
KS-99-076 p3*	0.739	KS-89-208 deck	0.009
KS-56-49 deck	0.246	LC-HPC-1 p1*	0.049
LC-HPC-1 p2*	0.024	LC-HPC-2	0.048
LC-HPC-3	0.121	LC-HPC-4 p2*	0.090
LC-HPC-5	0.154	LC-HPC-6	0.271
LC-HPC-7	0.012	LC-HPC-9	0.325
LC-HPC-11	0.163	LC-HPC-12 p1*	0.301
LC-HPC-12 p2*	0.332	LC-HPC-13	0.344
LC-HPC-14 p1*	0.543	LC-HPC-14 p2*	1.223
LC-HPC-14 p3*	0.695	LC-HPC-15	0.228
LC-HPC-16	0.250	LC-HPC-17	0.283

*p is placement.

in this study, however, show that the effectiveness of SRAs in reducing the cracking of bridge decks is much subtler than what has been shown in laboratory tests, and the influence of SRAs on the cracking performance of bridge decks is minor, if any, compared to that of paste content. These results indicate that the use of an SRA for decks with high paste contents will not ensure low cracking.

Internal curing—The crack densities of bridge decks with or without IC are compared in Fig. 7. The decks with IC are divided into two groups: five with 25.9% or lower paste contents (IN-IC [Low Paste]) and one with a higher paste content (27.6%, IN-IC-1). Three groups of decks without IC are included: one deck with a high paste content (27.6%, IN-Control), nine decks in Kansas with high paste contents (27.9% to 28.8%, labeled “KS-W/O IC [High Paste]”),

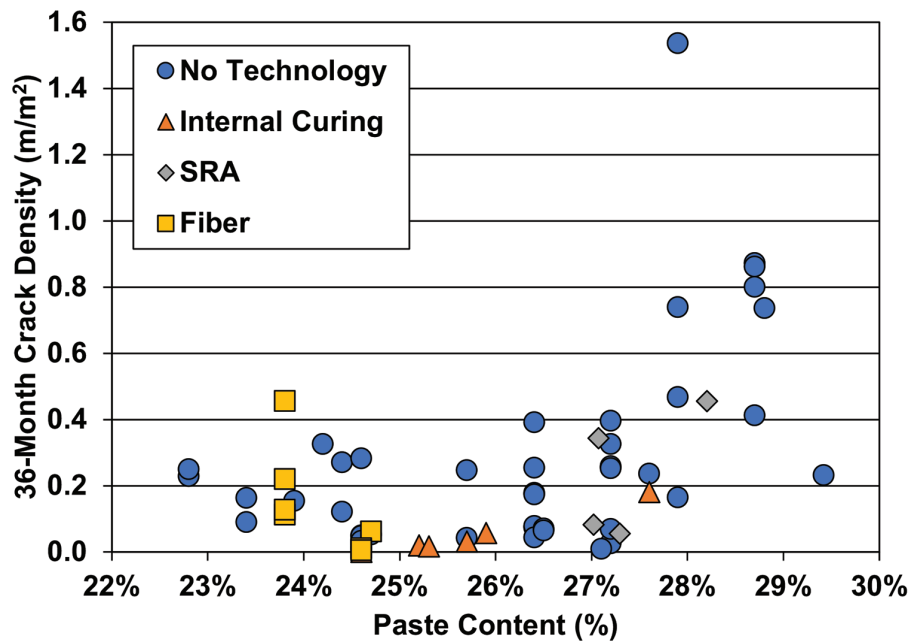


Fig. 5—Thirty-six-month crack density versus paste content for bridge deck placements. In this case, “No Technology” means without IC, SRA, and fibers.

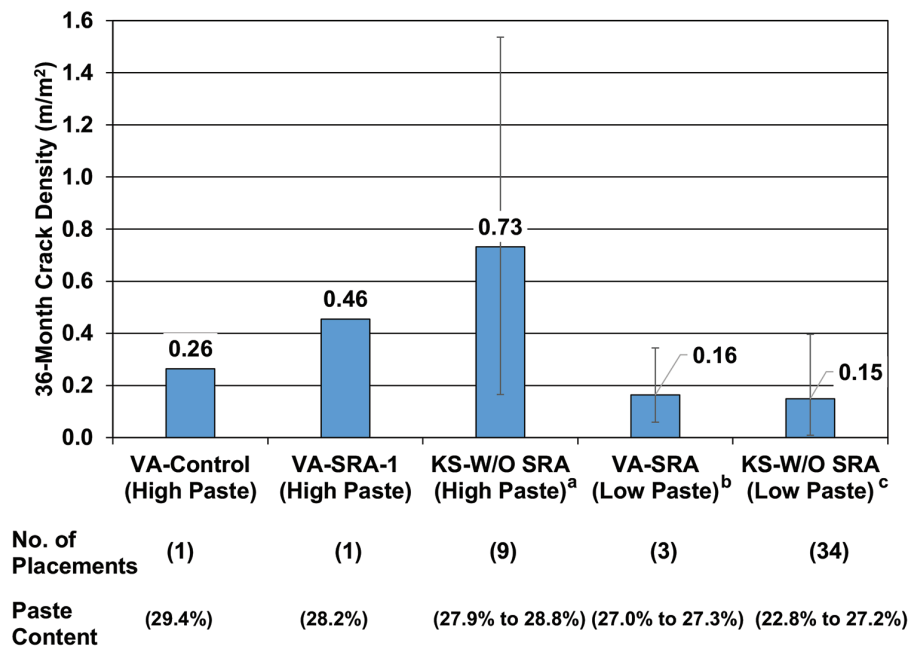


Fig. 6—Average 36-month crack densities of bridge decks with and without SRAs: ^aincludes nine conventional decks with paste contents of 27.9% or higher in Table 3; ^bincludes three deck placements with SRAs and paste contents of 27.3% or lower in Table 1; and ^cincludes 18 conventional deck placements with paste contents of 27.2% or lower in Table 3, 13 LC-HPC deck placements with no construction issues in Table 4, and three control decks paired with fiber decks with no construction issues in Table 1.

and 34 decks in Kansas with a low paste content (22.8% to 27.2%, labeled “KS-W/O IC [Low Paste]”).

For the comparison of decks with or without SRAs, paste content is the most significant factor affecting the crack density. The two IN decks with a high paste content (27.6%), one without and one with IC (IN-Control and IN-IC-1), have crack densities (0.24 and 0.18 m/m²) that are higher than the average crack densities of the decks with low paste contents (IN-IC [Low Paste], 0.03 m/m², and KS-W/O IC

[Low Paste], 0.15 m/m²). The two decks with high paste contents (IN-Control and IN-IC-1), however, have lower crack densities than the decks with high paste contents in Kansas (KS-W/O IC), likely due to their lower paste contents compared with the average paste content of the Kansas decks (27.6% versus 27.9% to 28.8%). In addition, between the two, the deck with IC had the lower crack density, and they both had 36-month densities below 0.30 m/m².

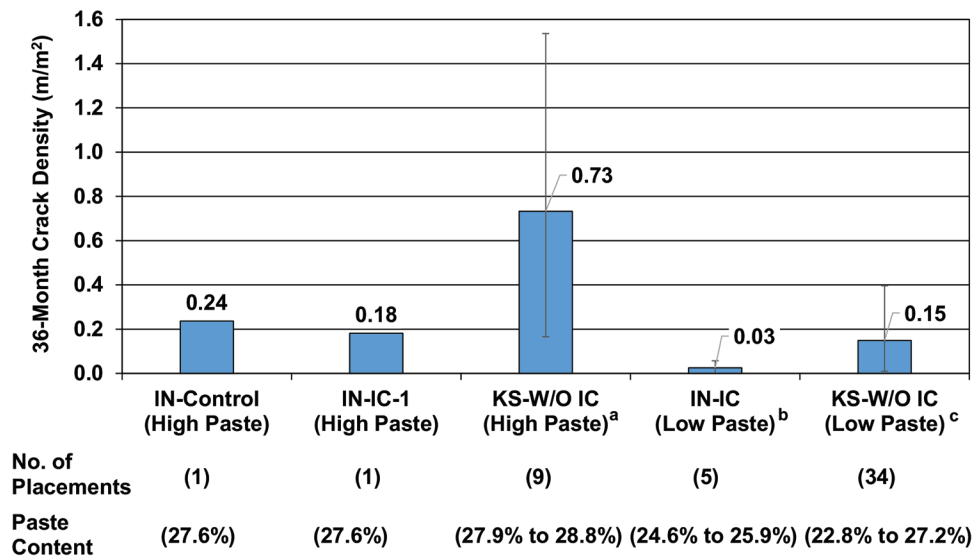


Fig. 7—Average 36-month crack densities of bridge decks with or without IC: ^aincludes nine conventional decks with paste contents of 27.9% or higher in Table 3; ^bincludes five deck placements with IC and paste contents of 25.9% or lower in Table 2; and ^cincludes 18 conventional deck placements with paste contents of 27.2% or lower in Table 3, 13 LC-HPC deck placements with no construction issues in Table 4, and three control decks paired with fiber decks with no construction issues in Table 1.

When comparing bridge decks with similar paste contents, the decks with IC consistently had lower 36-month crack densities. Among the three groups of decks with high paste contents, the one with IC (IN-IC-1) had a crack density of 0.18 m/m², lower than that of the deck in Indiana without IC (IN-Control, 0.24 m/m²) and the average of the decks without IC and with high paste contents in Kansas (KS-W/O IC [High Paste], 0.73 m/m²). The same observation can be made in reference to the two groups with low paste contents: decks with IC and low paste contents (IN-IC [Low Paste]) had an average 36-month crack density of 0.03 m/m², clearly lower than that of the decks without IC (KS-W/O IC [Low Paste], 0.15 m/m²), a difference that is statistically significant ($p = 0.03$). Overall, however, it appears that for IC to pay off, it must be paired with low paste content.

Influence of fiber reinforcement and construction practices

Figure 8 compares the average 36-month crack densities of bridge decks in Kansas (Tables 1 and 4) where construction was observed. The bridge decks are divided into four groups depending on whether fiber reinforcement was used and whether poor construction procedures were observed. The decks labeled “No issue” in Tables 1 and 4 were built without significant construction issues; the decks labeled “poor construction” were constructed using problematic procedures (labelled “Loss of consolidation,” “Insufficient consolidation,” or “Excessive finishing” in Tables 1 and 4). The two groups of bridge decks labeled “Fibers” collectively include the 10 decks with fiber-reinforced concrete, while the two groups labeled “W/O Fibers” include the control decks associated with the fiber decks (Table 1) and the LC-HPC decks (Table 4), all without fiber reinforcement. All the decks have low paste contents (22.2 to 24.7%), and if not for the use of poor construction practices, should exhibit low cracking.

As shown in Fig. 8, poor construction practices are associated with substantially increased cracking (likely caused by settlement of concrete, high paste content at the surface, or delayed curing). This is true for bridge decks constructed both with and without fibers in the concrete. For bridge decks with fiber reinforcement, those with bad construction practices exhibited a much higher average crack density than the decks with fiber reinforcement but without construction issues (0.48 versus 0.14 m/m²). A similar observation can be made for the decks without fiber reinforcement: poor construction practices resulted in a substantially increased average crack density (0.54 versus 0.13 m/m²).

The results further show that the negative impact of poor construction practices cannot be alleviated by either a low paste content or fiber reinforcement. This is illustrated by the similar crack densities of the decks with poor construction practices, with or without fiber reinforcement (0.48 m/m² for the decks constructed with fiber-reinforced concrete and 0.54 m/m² for the decks constructed without fiber-reinforced concrete). Although the decks constructed with fiber reinforcement have a lower average crack density, the difference between the two values is not statistically significant ($p = 0.76$). Laboratory tests (which usually involve proper consolidation) have shown that fiber reinforcement reduces the settlement of fresh concrete and cracking due to settlement (Qi 2003; Al-Qassag et al. 2015, 2022); the survey results reported herein, however, show that, when used in bridge decks, fiber reinforcement cannot overcome the increased likelihood of settlement cracking due to poor construction practices. To reduce bridge deck cracking, it is, therefore, imperative that state DOT strengthen the requirements for construction practices to ensure adequate consolidation, protect concrete after consolidation, avoid excessive finishing, and promptly apply curing.

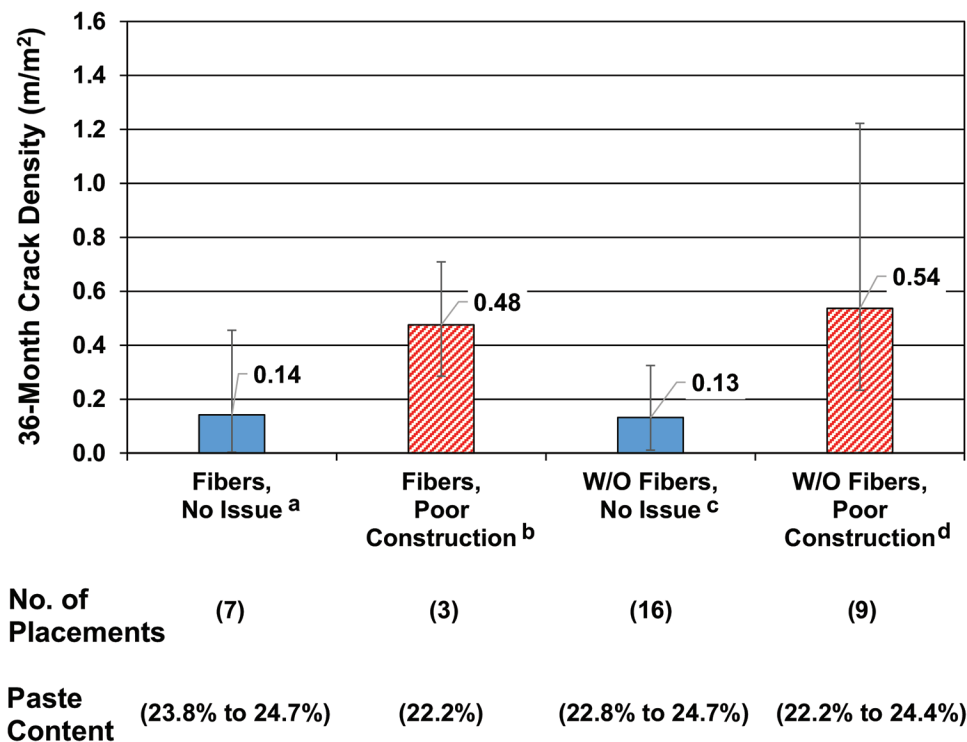


Fig. 8—Average 36-month crack densities of bridge decks with and without fibers and with no construction issues and with verified poor construction practices: ^aincludes seven decks with fiber reinforcement and no construction issues in Table 1; ^bincludes three decks with fiber reinforcement and construction issues in Table 1; ^cincludes three decks without fiber reinforcement and without construction issues in Table 1 and the 13 LC-HPC decks without construction issues in Table 4; and ^dincludes three decks without fiber reinforcement and with construction issues in Table 1 and six LC-HPC decks with construction issues in Table 4.

SUMMARY AND CONCLUSIONS

Crack densities at 36 months obtained from on-site surveys were studied for 74 bridge deck placements containing concrete mixtures with paste contents between 22.8 and 29.4%. Twenty of the placements were constructed with a crack-reducing technology (shrinkage-reducing admixtures [SRAs], internal curing [IC], or fiber reinforcement) and 54 without; three of the decks with fiber reinforcement and nine of the decks without crack-reducing technologies involved poor construction practices.

Based on the analysis of the crack survey results, the following conclusions can be made:

1. The paste content of the concrete mixture is the paramount material factor affecting cracking in bridge decks. A paste content of 26.5% or less significantly increases the probability that a bridge deck will have good cracking performance. When the paste content is higher than 27.3%, the bridge deck is more likely to exhibit a high crack density.
2. Crack-reducing technologies can play a role in reducing cracking in bridge decks, but they must be used in conjunction with a low paste content and good construction practices to achieve minimal cracking in a deck.
3. When contractors fail to follow proper procedures to consolidate, finish, or cure the concrete, bridge decks will exhibit substantially higher crack densities, even when a low paste content is used. Despite the use of concrete with low paste contents and crack-reducing technologies, departments of transportation should strictly regulate the proce-

dures that contractors follow during construction to achieve low cracking in bridge decks.

4. While laboratory tests can provide some insight into the effectiveness of crack-reducing technologies, those tests do not fully reflect the cracking behavior of in-service bridge decks and cannot account for the impact of poor construction practices.

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APPENDIX A: CRACK DENSITIES AT TIME OF SURVEY AND CRACK DENSITIES USED FOR ANALYSIS

Table A1—Crack densities at time of survey and crack densities used for analysis of fiber, control, and SRA decks

Placement	Survey A		Survey B		Crack density used for analysis, m/m ²
	Deck age, months	Crack density, m/m ²	Deck age, months	Crack density, m/m ²	
KS-Fiber-1 p1	33.7	0.112	—	—	0.112
KS-Fiber-1 p2	31.7	0.220	—	—	0.220
KS-Fiber-1 entire deck	32.7	0.166	—	—	0.166
KS-Fiber-2 p1	34.0	0.127	—	—	0.127
KS-Fiber-2 p2	32.4	0.456	—	—	0.456
KS-Fiber-2 entire deck	33.2	0.291	—	—	0.291
KS-Fiber-3	26.8	0.272	37.8	0.287	0.285
KS-Control-3	36.0	0.233	—	—	0.233
KS-Fiber-4 p1	33.6	0.709	—	—	0.709
KS-Fiber-4 p2	33.4	0.431	—	—	0.431
KS-Fiber-4 entire deck	33.5	0.594	—	—	0.594
KS-Control-4 p1	35.8	0.766	—	—	0.766
KS-Control-4 p2	35.6	0.393	—	—	0.393
KS-Control-4 entire deck	35.7	0.615	—	—	0.615
KS-Fiber-5	31.1	0.044	44.7	0.091	0.061
KS-Control-5	31.2	0.038	44.8	0.077	0.052
KS-Fiber-6	25.0	0.005	38.6	0.013	0.011
KS-Control-6	25.3	0.002	38.9	0.013	0.011
KS-Fiber-7	24.6	0.000	38.0	0.005	0.004
KS-Control-7	25.8	0.014	38.3	0.037	0.033
VA-SRA-1	19.1	0.455	43.0	0.333	0.369
VA-SRA-2	18.6	0.344	42.5	0.217	0.252
VA-SRA-3	33.9	0.083	—	—	0.083
VA-SRA-4	34.0	0.056	—	—	0.056
VA-Control	31.0	0.222	54.1	0.266	0.232

Note: Survey A is last crack survey performed before bridge deck reached 36 months of age; Survey B is first survey performed after bridge deck reached 36 months of age.

Table A2—Crack densities at time of survey and crack densities used for analysis for IC and control decks

Placement	Survey A		Survey B		Crack density used for analysis, m/m ²
	Deck age, months	Crack density, m/m ²	Deck age, months	Crack density, m/m ²	
IN-Control	71.6	0.507	93	0.67	0.236
IN-IC-1	71.6	0.347	93	0.447	0.181
IN-IC-2 p1	34.7	0	—	—	0.000
IN-IC-2 p2	37.2	0.02	—	—	0.020
IN-IC-3	34.8	0.016	56.8	0.033	0.017
IN-IC-4	21.6	0.003	43.8	0.086	0.057
IN-IC-5 p2	32.8	0.032	—	—	0.032

Note: Survey A is last crack survey performed before bridge deck reached 36 months of age; Survey B is first survey performed after bridge deck reached 36 months of age.

Table A3—Crack densities at time of survey and crack densities used for analysis for conventional decks in Kansas

Placement	Survey A		Survey B		Crack density used for analysis, m/m ²
	Deck age, months	Crack density, m/m ²	Deck age, months	Crack density, m/m ²	
KS-3-046 east deck	102	0.402	210	0.418	0.392
KS-3-046 west deck	102	0.362	210	0.539	0.254
KS-3-046 center deck	102	0.153	210	0.334	0.042
KS-75-044 deck	48	0.179	155	0.304	0.165
KS-75-045 deck	47	0.468	—	—	0.468
KS-89-204 deck	34	0.732	82	0.825	0.736
KS-3-045 west deck	112	0.122	223	0.192	0.074
KS-3-045 east deck	112	0.196	223	0.368	0.078
KS-3-045 west center deck	112	0.188	223	0.203	0.178
KS-3-045 center deck	112	0.215	220	0.273	0.174
KS-3-045 east center deck	112	0.163	220	0.333	0.043
KS-56-142 positive moment	80	0.108	189	0.200	0.071
KS-56-142 negative moment	80	0.093	188	0.163	0.064
KS-56-148 deck	36	0.259	—	—	0.259
KS-70-095 deck	106	0.069	212	0.136	0.025
KS-70-103 right	102	0.395	219	0.647	0.253
KS-70-103 left	102	0.557	219	0.842	0.396
KS-70-104 deck	106	0.083	212	0.104	0.069
KS-70-107 deck	34	0.322	82	0.417	0.326
KS-99-076 p4	42	0.872	—	—	0.872
KS-99-076 p5	42	0.861	—	—	0.861
KS-99-076 north (west)	42	0.801	—	—	0.801
KS-99-076 north (east)	42	0.412	—	—	0.412
KS-99-076 p2	42	1.536	—	—	1.536
KS-99-076 p3	42	0.739	—	—	0.739
KS-89-208 deck	36	0.009	—	—	0.009
KS-56-49 deck	25.8	0.230	47.5	0.265	0.246

Note: Survey A is last crack survey performed before bridge deck reached 36 months of age; Survey B is first survey performed after bridge deck reached 36 months of age.

Table A4—Crack densities at time of survey and crack densities used for analysis for LC-HPC decks

Placement	Survey A		Survey B		Crack density used for analysis, m/m ²
	Deck age, months	Crack density, m/m ²	Deck age, months	Crack density, m/m ²	
LC-HPC-1 p1	32.1	0.044	44.1	0.060	0.049
LC-HPC-1 p2	31.5	0.024	—	—	0.024
LC-HPC-2	21.2	0.028	44.5	0.059	0.048
LC-HPC-3	19.2	0.110	54.0	0.173	0.140
LC-HPC-4 p2	32.7	0.094	44.9	0.080	0.090
LC-HPC-5	31.1	0.128	43.0	0.190	0.154
LC-HPC-6	31.4	0.231	43.4	0.336	0.271
LC-HPC-7	34.8	0.012	—	—	0.012
LC-HPC-9	26.5	0.237	38.3	0.362	0.338
LC-HPC-11	23.4	0.060	36.2	0.265	0.262
LC-HPC-15	30.8	0.161	43.0	0.316	0.227
LC-HPC-16	31.2	0.211	43.5	0.311	0.250
LC-HPC-17	32.5	0.274	45.5	0.308	0.283

Note: Survey A is last crack survey performed before bridge deck reached 36 months of age; Survey B is first survey performed after bridge deck reached 36 months of age.

Table A5—Crack densities at time of survey and crack densities used for analysis for conventional decks (Yuan et al. 2011)

Bridge number	Placement	Survey A		Survey B		Crack density used for analysis, m/m ²
		Deck age, months	Crack density, m/m ²	Deck age, months	Crack density, m/m ²	
KS-3-046	East deck	210	0.418	102	0.402	0.392
	West deck	210	0.539	102	0.362	0.254
	Center deck	210	0.334	102	0.153	0.042
KS-75-044	Deck	155	0.304	48	0.179	0.165
KS-75-045	Deck	154	0.433	47	0.468	0.468
KS-89-204	Deck	82	0.825	34	0.732	0.736
KS-3-045	West deck	223	0.192	112	0.122	0.074
	East deck	223	0.368	112	0.196	0.078
	West center deck	223	0.203	112	0.188	0.178
	Center deck	220	0.273	112	0.215	0.174
	East center deck	220	0.333	112	0.163	0.043
KS-56-142	Positive moment	189	0.2	80	0.108	0.071
	Negative moment	188	0.163	80	0.093	0.064
KS-56-148	Deck	36	0.259	—	—	0.259
KS-70-095	Deck	212	0.136	106	0.069	0.025
KS-70-103	Right	219	0.647	102	0.395	0.253
	Left	219	0.842	102	0.557	0.396
KS-70-104	Deck	212	0.104	106	0.083	0.069
KS-70-107	Deck	34	0.322	82	0.417	0.326
KS-99-076	Placement 4	42	0.872	—	—	0.872
	Placement 5	42	0.861	—	—	0.861
	North (west lane)	42	0.801	—	—	0.801
	North (east lane)	42	0.412	—	—	0.412
	Placement 2	42	1.536	—	—	1.536
	Placement 3	42	0.739	—	—	0.739
KS-89-208	Deck	36	0.009	—	—	0.009
KS-56-49	Deck	25.8	0.230	47.5	0.265	0.246

Note: Survey A is last crack survey performed before bridge deck reached 36 months of age; Survey B is first survey performed after bridge deck reached 36 months of age.

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Effect of Flow Ratio and Angle of Friction on Dynamic Behavior of High-Strength Concrete

by Dipte Khan, Sumaiya Hassan, and Abu Sufian Md. Zia Hasan

The effect of the parameters of a model, namely flow ratio (K) and angle of friction (β), is analyzed numerically on high-strength concrete (HSC) under a time-dependent pressure load. HSC, with a compressive strength of 120 MPa under static loading, when simulated under a rate of loading of 1100 GPa/s, shows an ultimate compressive strength ranging from 133 to 160 MPa. The dynamic stress and strain of HSC is proportional to the flow ratio and angle of friction. Multiple regression equations have been established to define the relationship between model parameters and dynamic strength, which are applicable for flow ratios between 0.80 and 1.00 and an angle of friction between 50 and 65 degrees.

Keywords: angle of friction; Comité Euro-International du Béton (CEB) model; dynamic behavior; flow ratio; high-strength concrete (HSC).

INTRODUCTION

According to ACI 363R-92, high-strength concrete (HSC) is generally characterized by compressive strengths on the order of 60 MPa at 28 days.¹ ACI 363.2R-98 discusses high-strength concrete in the strength range of approximately 55 MPa and above. Special concrete “made with exotic materials or processes” are not taken into consideration by the Committee.² Extensive research has been conducted on HSC with a compressive strength ranging from 60 to 130 MPa.^{3–5}

HSC is essential for engineering applications requiring a high compressive strength such as high-rise buildings, where it is used in foundations, shear walls, and columns, particularly on lower floors with higher stresses. It enables longer spans in prestressed or reinforced concrete girders. Thus, the use of HSC is ideal for bridge superstructures and extending bridge deck longevity and durability. Recently, numerous studies have been carried out in relation to the experimental and numerical finite element (FE) modeling of these structures by using FE model software, namely Abaqus.^{6–12} Ensuring the protection of these HSC structures from dynamic loading is a crucial design factor for enhancing their safety and blast resistance. These dynamic loads can result from ballistic or tornado-driven debris impacts, air blast or wind gust impulses, and seismic activity, which are very unpredictable and destructive. The behavior of concrete under dynamic loads shows a critical difference in comparison with static loads due to the effects of the strain rate.¹³ The compressive strength in the case of dynamic loading is reported to be larger than that of static loading due to inelastic deformation delaying effects in concrete.¹⁴ Therefore, it is particularly important to find a suitable concrete

model for simulating the performances of concrete structures under dynamic loads.

The choice of material models of concrete is possible in Abaqus for the smeared cracking model, the brittle cracking mode, and the damaged plasticity model.¹⁵ Each model is designed for a particular type of use. The smeared cracking model can handle only monotonic loading. The brittle cracking model assumes linear elastic material behavior in compression. As a result, the model can be reliably used only in cases where concrete behavior is dominated by tensile failure. The damaged plasticity model is the most complex concrete model incorporated into Abaqus that can be used in any loading regime. In this study, the behaviors of the constituents are characterized by an extended Drucker-Prager (DP) model, and the numerical modeling is performed by using the Abaqus/Explicit finite element package.¹⁵

Though the behavior of composite materials is complex, under essentially monotonic loading conditions, simple constitutive models provide useful design information. These constitutive models are essentially pressure-dependent plasticity models that have historically been popular in the geotechnical engineering field. However, more recently, they have also been found to be useful for the modeling of some composite materials that exhibit significantly different yield behavior in tension and compression.

The linear DP model is a simplified case of the extended DP model, which offers more flexibility by incorporating nonlinear behaviors, anisotropy, and volume change effects. Moreover, it provides a possibly noncircular yield surface in the deviatoric plane to match different yield values in triaxial tension and compression, associated inelastic flow in the deviatoric plane, and separate dilation and friction angles. For these reasons, the linear DP model is used to determine the behavior of HSC under dynamic loading conditions.

The three parameters required to define the DP model of concrete are the angle of friction (β), flow ratio (K), and dilation angle (ψ). The angle of friction is a critical parameter for understanding the material resistance to shear under loading. It defines the slope of the failure envelope in shear stress versus normal stress space and is a crucial parameter for simulating the behavior of materials such as concrete, soil, and rock under shear loading. The friction angles are varied

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from 32.4 to 65.1 degrees¹⁶ under standard test locations of the recycled concrete aggregates. K is the ratio between the yield triaxial tension stress and the yield triaxial compression stress. It can be defined as the ratio of the second stress invariant on the tensile meridian to that on the compressive meridian.¹⁷ The dilation angle is a parameter within plasticity-based constitutive models that quantifies the volumetric expansion (dilatancy) of concrete under shear stress. It represents the angle of inclination of the plastic potential surface in stress space, governing the ratio of plastic volumetric strain to plastic shear strain during inelastic deformation. The material behavior is ductile when the value of the dilation angle is very close to the angle of internal friction.¹⁸ In this model, the parameters associated with cohesion and the friction angle control the yielding and hardening criteria, while a plastic dilatation-related parameter controls the flow rule.¹⁹ When the flow ratio rises, the stress also rises with the gradual decrease in strain. Massive deformation and comparatively lower strength occur when the flow ratio is low.²⁰

This paper investigates the effect of the friction angle and flow ratio on the dynamic stress and strain behavior under dynamic loading. The HSC specimen simulations are performed by using the commercial software Abaqus.¹⁵ Abaqus enables simulation under blast or dynamic loading.¹⁰ Therefore, this commercial software is used to determine the effect of the flow ratio and angle of friction of HSC to develop a numerical FE model. A significant number of simulations are performed to establish a relation of dynamic stress-strain behavior along with the angle of friction and flow ratio. In addition, several regression models are predicted and finally, two working diagrams are proposed for the practical application of HSC under dynamic loading.

RESEARCH SIGNIFICANCE

The suggested model offers a simple means of calibrating the HSC modeling parameters—that is, the angle of friction and flow ratio—saving both resources and time by avoiding the need for time-consuming and expensive experimental work under dynamic loading. On the basis of the simulation, the proposed multiple regression equations are established. The authors believe that the predicted model equations can enable the illustration of the relationship of the two independent variables such as the flow ratio and angle of friction of HSC with the dynamic strength. The obtained results are useful for practical applications to determine the failure behavior of HSC accurately.

LINEAR DRUCKER-PRAGER MODEL

The yield criteria for the material model are based on the shape of the yield surface in the meridional plane. The yield surface can have a linear form, a hyperbolic form, or a general exponent form. The choice of model to be used depends largely on the analysis type, the kind of material, the experimental data available for calibration of the model parameters, and the range of pressure stress values that the material is likely to experience. In the case of the linear DP model, the yield surface on the meridional plane is shown in Fig. 1, which is explained in the yield criterion section.

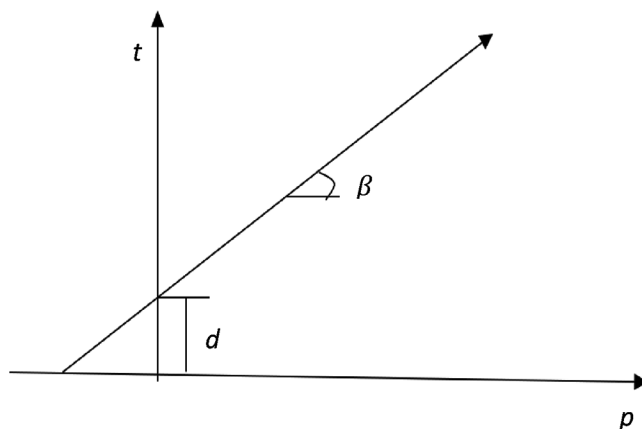


Fig. 1—Typical flow surface of linear DP model in meridional plane.

The yield criterion—that is, the flow rule described by Abaqus¹⁵—is described as follows.

Yield criterion

The yield stress surface makes use of two invariants, defined as the equivalent pressure stress

$$p = -\frac{1}{3}\text{trace}(\sigma) \quad (1)$$

and the von Mises equivalent stress

$$q = \sqrt{\frac{3}{2}(S:S)} \quad (2)$$

where $S = \sigma + pI$ is the stress deviator. In addition, the linear model also uses the third invariant of deviatoric stress

$$r = \left[\left(\frac{9}{2} \right) S:S:S \right]^{1/3} \quad (3)$$

In the case of the linear DP model, the deviatoric stress measure is defined as

$$t = \frac{q}{2} \left[1 + \frac{1}{K} - \left(1 - \frac{1}{K} \right) \left(\frac{r}{q} \right)^3 \right] \quad (4)$$

where β is the slope of the linear yield surface in the p - t stress plane and is commonly referred to as the angle of friction of the material; and K is the flow ratio. To ensure the convexity of the yield surface, $0.778 \leq K \leq 1.00$. This measure of deviatoric stress is used because it allows the matching of different stress values in tension and compression in the deviatoric plane, thereby providing flexibility in fitting the experimental results when the material exhibits different yield values in triaxial tension and in compression tests. The function is sketched in Fig. 2.

Because $(r/q)^3 = 1$ in uniaxial tension, $t = (q/K)$ in this case. And because $(r/q)^3 = -1$ under uniaxial compression, $t = q$ in that case. When $K = 1$, the dependence on the third deviatoric stress invariant is removed; the von Mises circle is recovered in the deviatoric plane: $t = q$. With this expression for the deviatoric stress measure, the yield surface is defined as

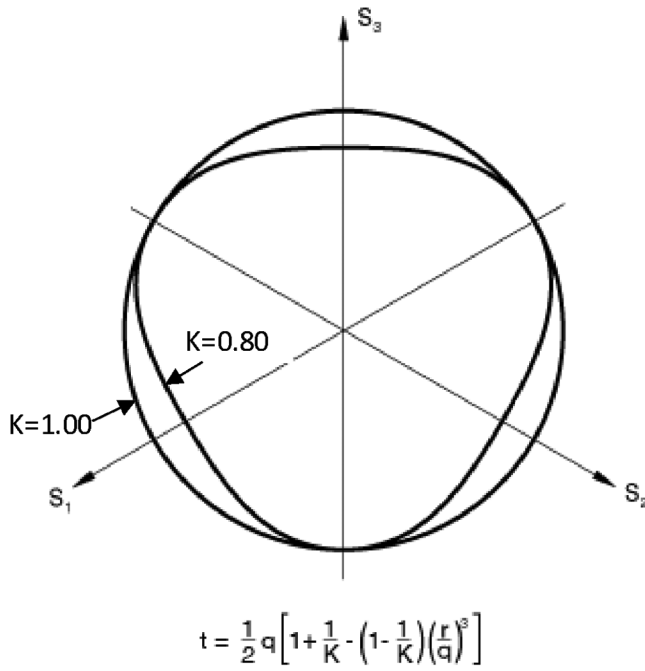


Fig. 2—Typical flow surfaces of linear DP model in deviatoric plane.

$$F = t - p \tan \beta - d = 0 \quad (5)$$

where d is the cohesion of the material. When hardening is defined by the uniaxial compression yield stress, σ_c , cohesion can be written as

$$d = \left(1 - \frac{1}{3 \tan \beta}\right) \sigma_c \quad (6)$$

The cohesion of material (d) is a hardening parameter which represents isotropic hardening, as illustrated in Fig. 3.

Plastic flow

Potential flow in the linear model is assumed so that

$$d\epsilon_{ij}^p = \frac{d\bar{\epsilon}^p}{c} \frac{\delta G}{\delta \sigma_{ij}} \quad (7)$$

where

$$c = 1 - \frac{1}{3} \tan \psi \quad (8)$$

$d\epsilon_{ij}^p$ is the plastic part of the rate of deformation; and $d\bar{\epsilon}^p$ is the equivalent plastic strain rate satisfying the plastic consistency condition such that $q d\bar{\epsilon}^p = \sigma_{ij} / d\epsilon_{ij}^p$. The flow potential is defined as

$$G = t - \tan \psi \quad (9)$$

where ψ is the dilation angle in the p - t plane. A geometrical interpretation of ψ is shown in the p - t diagram of Fig. 3. A comparison of Eq. (5) and Eq. (9) shows that the flow is associated in the deviatoric plane because the yield surface and the flow potential both have the same functional dependence on t . However, the dilation angle ψ and the material

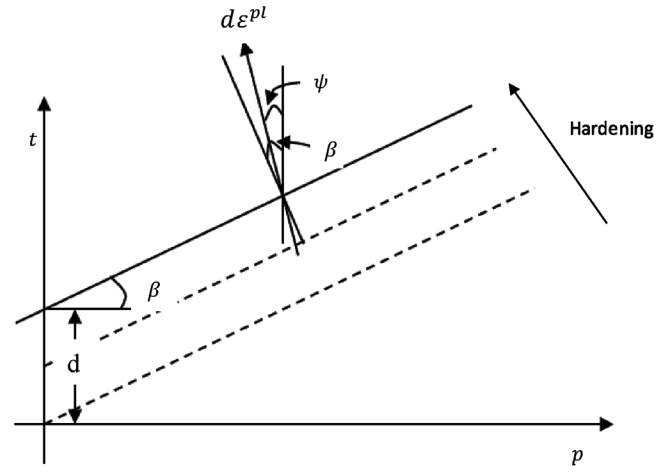


Fig. 3—Schematic of hardening and flow direction for linear DP model in p - t plane.

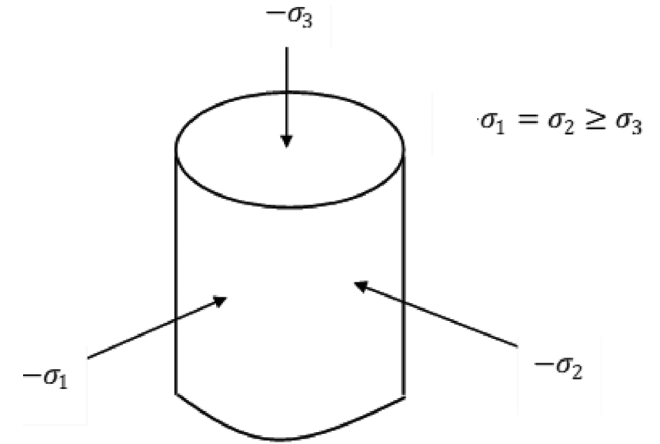


Fig. 4—Triaxial compression for linear DP model.

angle of friction β may be different, so the model may not be associated in the p - t plane. Non-associated flow is generally assumed when the model is used for polymeric materials. If $\psi = 0$, the inelastic deformation is incompressible; if $\psi \geq 0$, the material dilates. An associated flow results from setting $\psi = \beta$. The original DP model is available by setting $\psi = \beta$ and $K = 1$. Hence, ψ is referred to as the dilation angle.

Matching experimental triaxial test data

HSC parameters are determined from triaxial testing data which has been described in Abaqus.¹⁵ In a triaxial compression test, the specimen is confined by a pressure stress that is held constant during the test with an additional compression stress which is superposed in one direction. Thus, the principal stresses are all negative, with $0 \geq \sigma_1 = \sigma_2 \geq \sigma_3$, where σ_1 is the maximum principal stress; σ_2 is the intermediate principal stress; and σ_3 is the minimum principal stress. The schematic diagram of the triaxial compression test is shown in Fig 4. The stress invariants are now

$$p = -\frac{1}{3}(2\sigma_1 + \sigma_3) \quad (10)$$

$$q = \sigma_1 - \sigma_3 \quad (11)$$

Table 1—Static properties and dimension of HSC prism and load-bearing steel block

Materials	Concrete prism	Load-bearing steel block
Dimension, mm	100 x 100 x 350	100 x 100 x 250
Density, kg/m ³	2400	7800
Modulus of elasticity, MPa	50,310	210,000
Poisson's ratio	0.19	0.3
Compressive strength, MPa	120	—

and

$$r^3 = (\sigma_1 - \sigma_3)^3 \quad (12)$$

So that

$$t = \frac{q}{K} = \frac{\sigma_1 - \sigma_3}{K} \quad (13)$$

From the triaxial compression test, the value of the angle of friction β varies from 30 to 40 degrees in the case of HSC with a compressive strength of 55 to 85 MPa.²¹ However, it can be seen that material with a higher strength has a higher angle of friction.²² The value of the angle of friction for mortar has been found to be 40 to 60 degrees.²³ The effect of the dilation angle on the load deflection is very limited.²⁴ For this reason, in this study, the effect of dilation angle has been neglected. The value has been assumed as per the associated flow rule—that is, $\beta = \psi$.

FINITE ELEMENT MODELING (FEM)

The FE method is good at modeling non-uniform concrete because it can capture the stress variations in concrete, which are complex.²⁵ For FE and analytical concrete modeling, numerous constitutive models such as models of plasticity are applied.²⁶ The description of the numerical FE model is as follows.

Geometric model and their material properties

In this research, the model has two parts—namely, concrete prism and load-bearing steel block. A concrete prism is defined as solid, homogenous, and deformable, and the bearing block is defined as discrete rigid. The static properties of a concrete prism and load-bearing block are given in Table 1. The modulus of elasticity obtained through regression analysis is as follows²⁷

$$E = 10,200(f'_c)^{1/3} \quad (14)$$

where E and f'_c are expressed in MPa.

There are very few experimental data on the Poisson's ratio values for HSC. A clear trend for Poisson's ratio may not be evident, but an increase in concrete strength is associated with a decrease in its average Poisson's ratio.²⁸ For a concrete mixture with a compressive strength of 120 MPa, the observed Poisson's ratio is approximately 0.19.²⁹ Additionally, as the ratio of water to cement increases, Poisson's ratio

Table 2—Modified stress-strain relationship of HSC under compression

Yield stress, MPa	Plastic strain
90	0.00000
100	0.00200
110	0.00220
115	0.00230
120	0.00260
115	0.00262
110	0.00270
100	0.00275
90	0.00285
80	0.00300
70	0.00313
60	0.00340
50	0.00360
40	0.00410
30	0.00460
20	0.00600

tends to decrease.³⁰ Therefore, a Poisson's ratio of approximately 0.19 is used in the simulation of the HSC model.

The linear DP model is used to describe the potentiality of plastic flow. This model obeys nonlinear kinematic and linear isotropic hardening. This type of plasticity model is useful to estimate the behavior of concrete structural members. In this study, the concept of yielding criteria and flow rule of the linear DP model is used to simulate the characteristics of HSC under dynamic loading. The yield stress and plastic strain relationship is shown in Table 2.²⁷

Two parts are assembled in such a way that the load-bearing block is just above the concrete prism and the bottom surface of the block is in contact with the top surface of the concrete prism. The simulation setup, load, and boundary conditions are shown in Fig. 5.

Boundary condition and loading

A loading rate of 1100 GPa/s is applied on the upper surface of the steel block while the bottom surface of the concrete prism is kept fixed—that is, $U1 = U2 = U3 = UR1 = UR2 = UR3 = 0$. In this model, a strain rate of 21.86 s⁻¹ is used, which is equivalent to 1100 GPa/s. The strain rate is calculated by using the formula $\dot{\epsilon} = (\dot{\sigma}/E)$, where E is the modulus of elasticity of HSC; and $\dot{\sigma}$ is the loading rate.

Generation of mesh

To make sure that the outcomes of the simulation are precise and trustworthy, a mesh convergence study is carried out, performing a significant number of simulations to optimize the element size. The results obtained are summarized in Fig. 6. This figure shows that constant results are obtained along with the 3600 elements. Achieving the mesh refinement level is the aim, to guarantee accurate results and cut down on the simulation time. Though a number of models

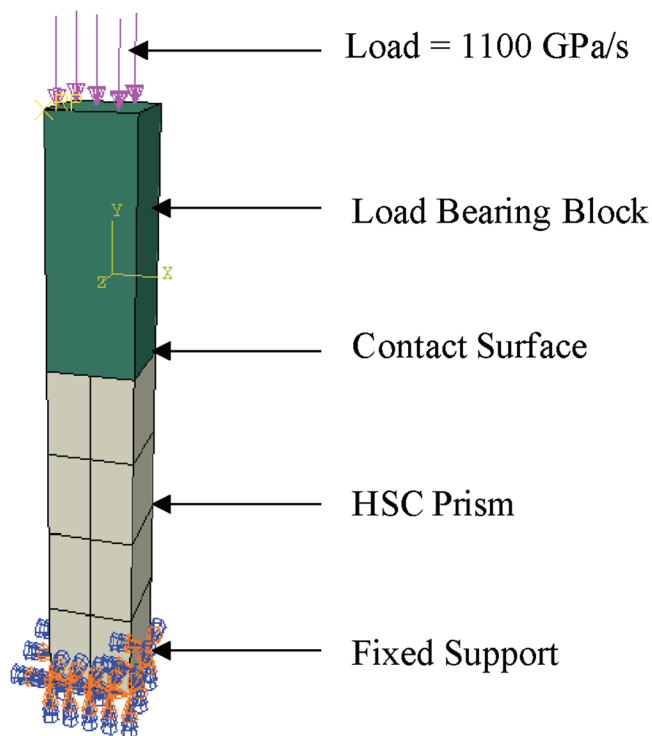


Fig. 5—Arrangement of concrete prism and load-bearing block with loading setup and support condition.

use coarser meshes, dense FE meshes are used to simulate concrete. In this model, the Lagrangian brick element (hex shape) is used to obtain a precise, reliable, and speedy solution for the HSC prism and load-bearing block. All the elements in the model are purposely assigned the same mesh size to ensure that each two different materials share the same node. A concrete prism has 4477 nodes and 3600 elements, which are hexahedra of the C3D8R type. Herein, C3D8R represents a general-purpose eight-node brick element with three degrees of freedom per node (displacements in the x-, y-, and z-directions), hourglass control, and reduced integration, which is shown in Fig. 7. In Abaqus, quadrilateral and hexahedral elements can use a reduced-integration scheme, employing fewer integration points than fully integrated elements. Reduced-integration linear elements integrate at a single centroid point. Reduced integration has been used in the dynamic analyses of concrete structures.³¹ To mitigate the occurrence of hourglass modes, Abaqus introduces a small amount of artificial hourglass stiffness in first-order reduced-integration elements. This stiffness becomes more effective with mesh refinement, which ensures acceptable accuracy for most applications. As a result, these elements are generally preferred for stress/displacement analyses.

In this study, the calibration of the concrete material is performed for standard three-dimensional (3-D) stress linear and quadratic hexahedral elements with reduced integration (C3D8R). The analysis time is captured to be less for C3D8R elements with relatively accurate results. Furthermore, C3D8R elements follow the constitutive law of integration accurately, and usually, it is the most suitable element type for the nonlinear analysis of structures.³²⁻³⁴

On the other hand, a steel bearing block has 1202 nodes and 1200 elements, which are linear quadrilaterals of the

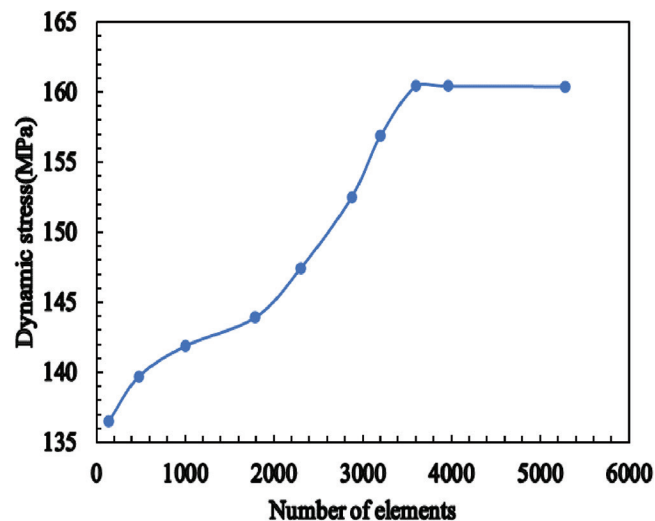


Fig. 6—Dynamic stress variation of HSC with respect to number of elements of concrete prism.

R3D4 type. Herein, R3D4 represents 3-D quadrilateral rigid body elements with four nodes, which is shown in Fig. 7.

Contact properties

A surface-to-surface contact property is chosen where the bearing block's bottom surface is defined as the master surface and the concrete prism's top surface is defined as the slave surface. For proper interaction between two surfaces, a tie constraint is used. To transfer normal and tangential forces accurately, frictionless hard contact is used. The mechanical constraint formulation is performed using the penalty contact method. Dynamic-Explicit is selected for the step, and the period of the step is kept at 0.0001. The number of intervals for history output and field output are selected to be 200 to provide more accurate results.

RESULTS AND DISCUSSION

The simulation of the model is accomplished to find out the dependency of the dynamic strength of HSC on the DP model parameters. The obtained results are presented first, with the effect of the flow ratio and angle of friction on the dynamic strength with a constant loading rate presented second. A few multiple regression models are predicted. Third, the results of the proposed model are compared with those of the Comité Euro-International du Béton (CEB) (the organization is now known as the International Federation for Structural Concrete [*fib*]) model, and finally, a working manual of HSC is produced with the relationship between the dynamic stress-strain and the model parameters such as the angle of friction and flow ratio under dynamic loading presented.

Effect of flow ratio and angle of friction

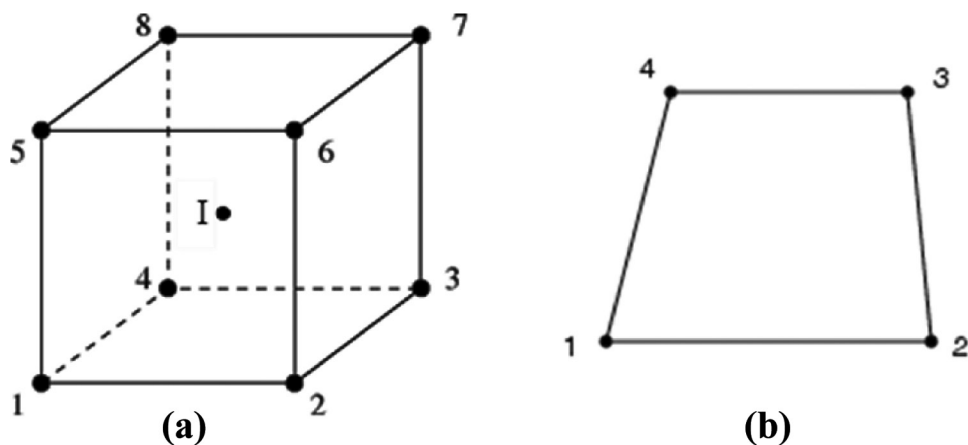
The effect of the flow ratio and angle of friction angle on the dependency of dynamic stress-strain is presented in Tables 3 and 4. From these tables, it can be seen that dynamic stress and strain increase with the increase in the angle of friction under a constant flow ratio. On the other hand, dynamic stress increases but strain decreases with

Table 3—Variation in dynamic stress and strain with respect to angle of friction

Flow ratio K	Angle of friction β , degrees	Dynamic stress σ_d , MPa	Dynamic strain ϵ_d , MPa	Remarks
1.00	50	132.46	0.00037	Dynamic stress and strain increase with the increase in angle of friction
	55	141.46	0.00039	
	60	151.32	0.00041	
	65	160.44	0.00043	

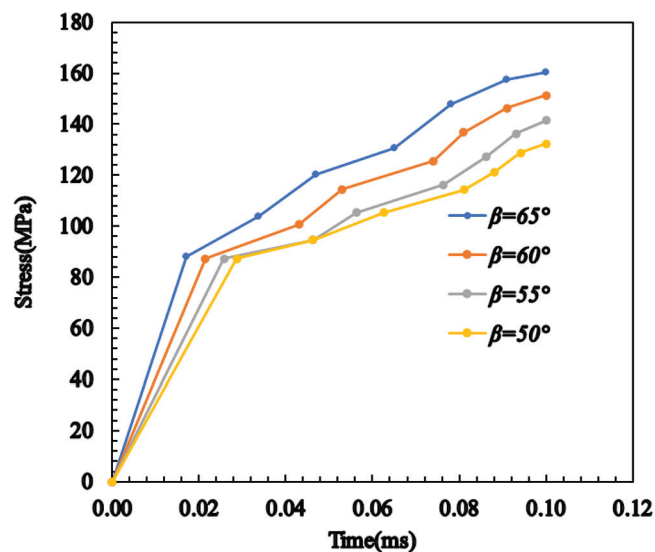
Table 4—Variation of dynamic stress and strain with respect to flow ratio

Angle of friction β , degrees	Flow ratio K	Dynamic stress σ_d , MPa	Dynamic strain ϵ_d , MPa	Remarks
60	0.80	131.41	0.00053	Dynamic stress increases but strain decreases with the increase in flow ratio
	0.85	135.70	0.00050	
	0.90	139.85	0.00047	
	0.95	144.45	0.00044	
	1.00	151.32	0.00041	

*Fig. 7—(a) Node numbering and integration point (I) of C3D8R; and (b) node numbering of R3D4.*

the increase in the flow ratio under a constant angle of friction of HSC. Figure 8 shows that the variation in dynamic stress of HSC with respect to time in milliseconds (ms) for an angle of friction which varied from 50 to 65 degrees along with a constant value of flow ratio $K = 1.00$. It can be seen that the dynamic compressive stress of HSC increases with the increase in the angle of friction and time. The dynamic compressive stress of HSC is obtained as 132.46 and 160.44 MPa corresponding to friction angles of 50 and 65 degrees, respectively. The dynamic stress increases by 21.21% for HSC when the applied loading is approximately 1100 GPa/s.

The variation in the dynamic strain of HSC with respect to time at different angles of friction is shown in Fig. 9. Initially, the dynamic strain of HSC is approximately constant with time—for example, 0.030 and 0.065 ms—while the angle of friction changes from 50 to 65 degrees. A small magnitude of dynamic strain changes is observed with a variation in the angle of friction of HSC when the applied loading is approximately 1100 GPa/s. The sensibility of the angle of friction of HSC is less important in the case of dynamic strain under applied dynamic loading. This type of behavior causes the brittle type of failure for HSC under applied dynamic

*Fig. 8—Stress versus time diagram at different friction angles.*

loading. After a certain period of time, such as 0.065 to 0.10 ms, the stress increases with the increase in time. This means that the failure of material has already happened.

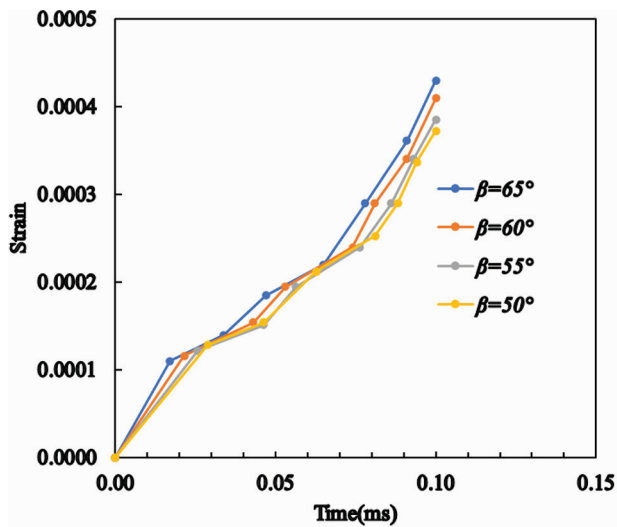


Fig. 9—Strain versus time diagram at different friction angles.

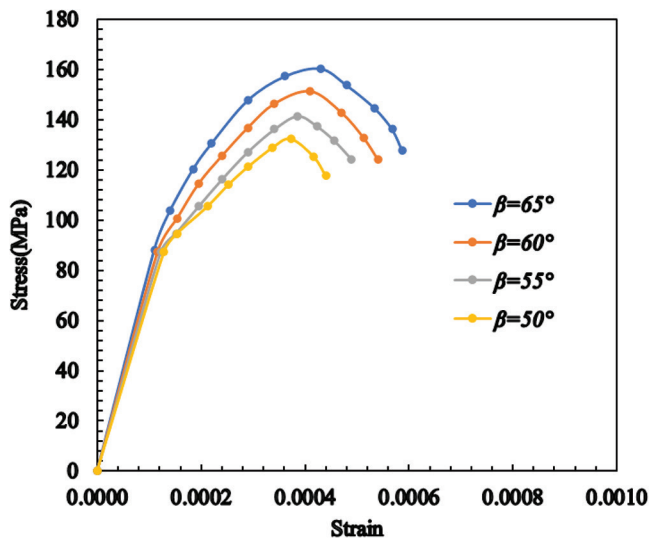


Fig. 10—Stress versus strain diagram at different friction angles.

Figure 10 illustrates the dynamic stress-strain relationship with different angles of friction ranging from 50 to 65 degrees. This figure reveals that both the dynamic stress and strain of HSC increase over the range of 133 to 160 MPa and 0.000373 to 0.000430. From Fig. 9 and 10, it can be concluded that the dynamic stress and strain are also proportional to the angle of friction when the flow ratio is constant. Although the dynamic strain is almost constant, just the load applied at 1100 GPa/s within a few milliseconds for HSC is described in Fig. 9. It is evident from this that the material properties of HSC, notably the higher modulus of elasticity and lower Poisson's ratio, make the effect of the angle of friction on strain negligible. Thus, taking into account the constant flow ratio, it can be concluded that dynamic stress increases with an increase in the angle of friction, and strain is almost independent with the variation in the angle of friction.

Figure 11 shows the stress versus time relationship at various flow ratios when the angle of friction is 60 degrees. It reveals

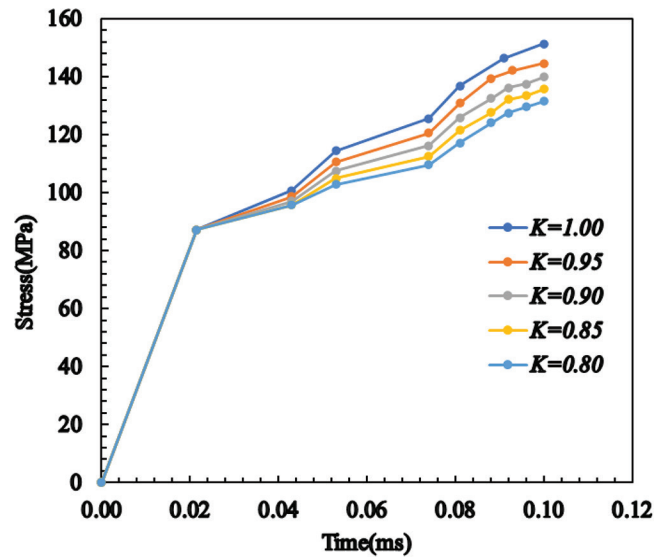


Fig. 11—Stress versus time diagram at different flow ratios.

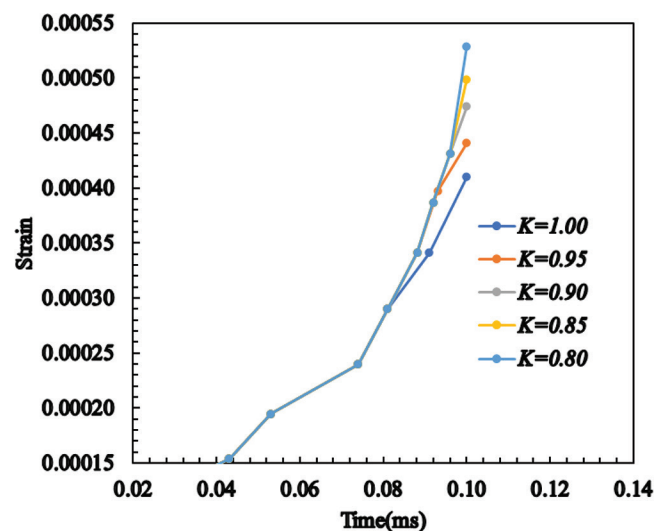


Fig. 12—Strain versus time diagram at different flow ratios.

that dynamic stress increases from 131.41 to 151.32 MPa with an increase in the flow ratio ranging from 0.80 to 1.00. This observation concludes that stress increases 15.15% under a dynamic loading rate of 1100 GPa/s. Again, Fig. 12 shows the stress versus time relationship at different flow ratios when the angle of friction of HSC is 60 degrees. It shows that strain decreases, ranging from 0.000529 to 0.00041, with an increase in flow ratio under the dynamic loading condition. It undergoes only a 0.012% overall change. So, strain varies very little with respect to the flow ratio.

Figure 13 illustrates that a lower flow ratio at a constant friction angle results in a higher strain value. As a result, the dynamic stress increases as the flow ratio increases, while strain eventually decreases for HSC under the application of a loading rate of 1100 GPa/s. The lower magnitude of flow ratio is directed a large deformation; thus, the occurred failure shows a comparatively lower strength. The angle of friction of concrete shows sensitivity to the loading rate and adds complexity to the stress behavior analysis. In most cases, it is difficult to determine how concrete will behave

dynamically. By establishing a more reasonable correlation between the parameters of the DP model and dynamic strength, this challenge is avoided in the current work. This correlation provides an easy way to determine the model's flow ratio and angle of friction values for more precise numerical results.

Multiple regression analysis of the flow ratio and angle of friction

A mathematical equation of multiple regressions can be used to express the interaction between dynamic strength and the flow ratio and angle of friction, which are the DP model parameters. The flow ratio and angle of friction are considered independent variables in the equation, while dynamic strength is designated as the dependent variable. The following is a possible term for the regression equation

$$\sigma_d = l + m\beta + nK \quad (15)$$

where σ_d is dynamic strength; β is angle of friction; K is flow ratio; and l , m , and n are regression constants. From the statistical analysis, the values of regression constants are as follows: $l = -40.0765$; $m = 1.7616$; and $n = 85.0250$. Hence, the mathematical regression equation of dynamic strength of HSC can be formulated as

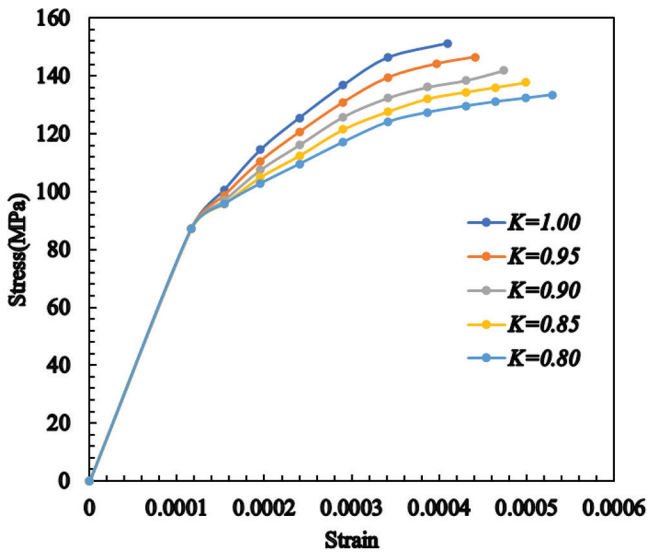


Fig. 13—Stress versus strain diagram at different flow ratios.

Table 5—Regression analysis summary of Eq. (16)

Regression statistics		ANOVA						
Multiple R	0.9992		df	SS	MS	F	Significance F	
R^2	0.9983	Regression	2	2662.4467	1331.2233	5228.8577	1.9391×10^{-24}	
Adjusted R^2	0.9982	Residual	17	4.3281	0.2546			
Standard error	0.5046	Total	19	2662.7747				
Observations	20		Coefficient	Standard error	t-statistic	p-value	Lower 95%	Upper 95%
		Intercept	-40.0765	1.8498	-21.6655	8.0660×10^{-14}	-43.9792	-36.1738
		K	85.0250	1.5956	53.2874	2.3209×10^{-20}	81.6586	88.3914
		β	1.7616	0.0202	87.2821	5.4403×10^{-24}	1.7190	1.8042

$$\sigma_d = -40.0765 + 1.7616\beta + 85.0250K \quad (16)$$

Table 5 shows the summary output of the linear regression Eq. (16), which has three parts. The first part, regression statistics, describes how effectively the calculated linear regression equation adjusts the data. The percentage of the dependent variable's variance that the independent variables in the model have been able to explain is shown in this section by R^2 , also known as the coefficient of determination. It indicates how effectively unseen samples are likely to be predicted by the suggested model. R^2 and adjusted R^2 can be calculated by using the following formulas³⁵

$$R^2 = \sum(\hat{y} - \bar{y})^2 / \sum(y - \bar{y})^2 \quad (17)$$

$$\text{Adjusted } R^2 = 1 - [(1 - R^2)(N - 1)/(N - M - 1)] \quad (18)$$

where y is actual value; $\bar{y}$ is mean of actual values; $\hat{y}$ is estimated value; M is number of independent variables; and N is total number of observations. A larger R^2 value suggests that the independent variables accurately explain the regression model. The R^2 value in the present study is 99.83%, which is extremely satisfactory. The standard error can be calculated through the following equation

$$\text{Standard error} = \sum(\hat{y} - y)^2 / (N - 2) \quad (19)$$

The smaller value of the standard error is also satisfactory. The second part, analysis of variance (ANOVA), analyzes the variance where df is degrees of freedom; SS is sum of squares around mean; and MS is mean square. The regression equation's overall statistical significance is tested in this case using the F test. These can be calculated by the following equations³⁶

$$\text{Degree of freedom of regression} = k - 1 \quad (20)$$

$$\text{Degree of freedom of error} = N - k \quad (21)$$

$$\text{Sum of squared regression (SSR)} = \sum(\hat{y} - \bar{y})^2 \quad (22)$$

$$\text{Sum of squared error (SSE)} = \sum(\hat{y} - y)^2 \quad (23)$$

$$\text{Mean squared regression (MSR)} = \text{SSR} / (k - 1) \quad (24)$$

Table 6—Regression analysis summary of Eq. (27)

Regression statistics		ANOVA						
Multiple R	0.9989		df	SS	MS	F	Significance F	
R^2	0.9978	Regression	2	4.4672×10^{-8}	2.2336×10^{-8}	3795.5810	2.9372×10^{-23}	
Adjusted R^2	0.9975	Residual	17	1.0004×10^{-10}	5.8847×10^{-12}			
Standard error	2.4258×10^{-6}	Total	19	4.4772×10^{-8}	—			
Observations	20		Coefficient	Standard error	t -statistic	p -value	Lower 95%	Upper 95%
		Intercept	0.00078218	2.374×10^{-5}	100.61705	8.389×10^{-31}	0.00234	0.00244
		K	−0.000598	2.404×10^{-5}	−33.16309	2.801×10^{-20}	−0.00085	−0.00075
		β	3.7760×10^{-6}	2.404×10^{-7}	52.36739	1.369×10^{-24}	1.209×10^{-5}	1.309×10^{-5}

Table 7—Regression analysis summary of Eq. (28)

Regression statistics		ANOVA						
Multiple R	0.9992		df	SS	MS	F	Significance F	
R^2	0.9984	Regression	3	2662.5848	887.5283	3389.1680	1.2389×10^{-22}	
Adjusted R^2	0.9981	Residual	16	4.1900	0.2619			
Standard error	0.5117	Total	19	2662.5848	—			
Observations	20		Coefficient	Standard error	t -statistic	p -value	Lower 95%	Upper 95%
		Intercept	−11.0145	40.0628	−0.2749	0.7869	−95.9438	73.9148
		K	62.8062	30.6384	2.0499	0.0571	−2.1442	127.7566
		β	1.9019	0.1943	9.7898	3.6886×10^{-8}	1.4901	2.3137
		ε_d	−37,155.14	51,163.198	−0.7262	0.4782	−145,616.27	71,305.99

$$\text{Mean squared error (MSE)} = \text{SSR}/(N - k) \quad (25)$$

$$F = \text{MSR}/\text{MSE} \quad (26)$$

where k is the number of parameters being estimated. Herein, significance F gives an idea of how reliable (statistically significant) the result is. The critical value of significance F is 5%. So, a smaller value leads to higher accuracy. In the third part, the coefficient of independent variables and the intercept are analyzed.

Again, considering dynamic strain as a dependent variable and friction angle and flow ratio as independent variables, a regression equation can be formulated. From statistical analysis

$$l = 0.00078218$$

$$m = 0.000003776$$

$$n = -0.000598$$

Hence, the equation becomes

$$\varepsilon_d = 0.00078218 + 0.000003776\beta - 0.000598K \quad (27)$$

Table 6 reveals the regression analysis of the linear regression Eq. (27). From the first part, regression statistics, the R^2 value is 99.78% and the adjusted R^2 is 99.75%, which are highly satisfactory. In the second part, ANOVA, the value of significance F is 2.9372×10^{-23} , which is very small compared to the critical value.

A mathematical regression equation can be formulated where dynamic strength is the dependent variable and dynamic strain, friction angle, and flow ratio are independent variables which can be expressed as follows

$$\sigma_d = 9.601082671 + 1.91816965\beta + 74.7934499K - 16,138.9958\varepsilon_d \quad (28)$$

Table 7 reveals the regression analysis of the linear regression Eq. (28). From the regression statistics part, the R^2 value is 99.92% and the adjusted R^2 is 99.84%, which are highly satisfactory. In the part of ANOVA, the value of significance F is 1.2389×10^{-22} , which is very small as compared to the critical value. Equations (16), (27), and (28) are applicable when the flow ratio is between 0.80 and 1.00 and the angle of friction is between 50 and 65 degrees.

VALIDITY OF MODEL

According to *fib*,³⁷ the CEB model is regarded as a practical and simple method for determining the load-bearing capacity of reinforced concrete. The most effective model for improving the strain rate of concrete under compression as well as in tension is shown by this model code. The model's outputs include the largest displacement of the slab, the punching force, and the global free oscillation after impact.³⁸ According to *fib*³⁷

$$\sigma_d = [(\dot{\varepsilon}_d/\dot{\varepsilon}_s)^{1.025\alpha}] \sigma_s \text{ for } \dot{\varepsilon}_d \leq 30 \quad (29)$$

$$\sigma_d = [\gamma_s(\dot{\varepsilon}_d/\dot{\varepsilon}_s)^{1/3}] \sigma_s \text{ for } \dot{\varepsilon}_d > 30 \quad (30)$$

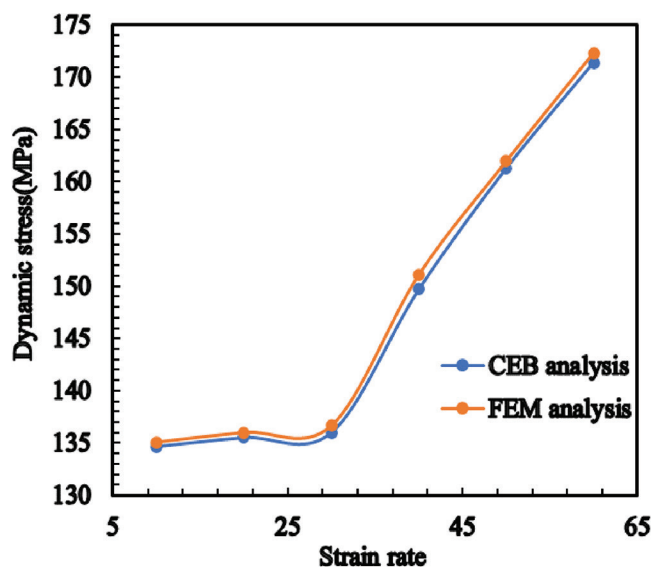


Fig. 14—Dynamic stress corresponding to strain rate.

where $\dot{\epsilon}_d$ is dynamic strain rate; $\dot{\epsilon}_s = 30 \times 10^{-6} \text{ s}^{-1}$; σ_d is uniaxial dynamic compressive strength of HSC; σ_s is uniaxial static compressive strength of HSC; $\gamma_s = 10^{(6.15\alpha-2)}$; $\alpha = 1/[5 + 9(\sigma_s/\sigma_0)]$; and σ_0 is equivalent strength, equal to 10 MPa.

Figure 14 shows the dynamic stress corresponding to the strain rate both for the results obtained from the present study (FEM) and the CEB model analysis of HSC. The predicted results agree with those of the CEB model along with the high strain rate of HSC. Hence, this figure describes the accuracy of the present study, though the methods of analysis are different. Figures 15 and 16 illustrate the graphic representation of the interaction between dynamic strength and dynamic strain with DP parameters—that is, flow ratio and angle of friction. These serve as working manuals, which are helpful in practical applications to precisely ascertain how HSC fails.

In this model, HSC with a static strength of 120 MPa and dynamic strain rate of 21.86 s^{-1} is used. According to Eq. (29), the dynamic strength is found to be 136 MPa. The values of the angle of friction and flow ratio (β°, K), according to this dynamic strength, are found to be (52 degrees, 1.00), (54 degrees, 0.95), (57 degrees, 0.90), (59 degrees, 0.85), and (61 degrees, 0.80) from Fig. 15.

Now substituting these values for the angle of friction and flow ratio in Eq. (16) and (28), the dynamic strengths are found to be 136.55, 135.82, 136.86, 136.13, and 135.40 MPa, respectively. As indicated in Table 8, these findings are very close to the value of the CEB model in Eq. (29).

In the same way, when the static compressive strength is 100 MPa and dynamic loading rate is 65 s^{-1} , according to the CEB model in Eq. (30), HSC has a dynamic strength of 150 MPa. The values for the angle of friction and flow ratio, according to this dynamic strength, are found to be (60 degrees, 1.00), (62 degrees, 0.95), and (64 degrees, 0.90) from Fig. 15.

Now, substituting these values for the flow ratio and angle of friction into Eq. (16) and (28), the dynamic strengths are found to be 150.64, 149.92, and 149.19 MPa, respectively,

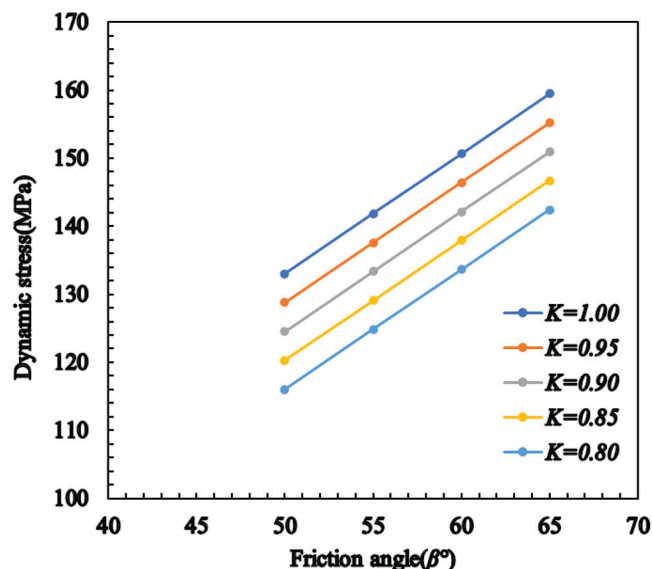


Fig. 15—Dynamic stress versus angle of friction corresponding to flow ratio.

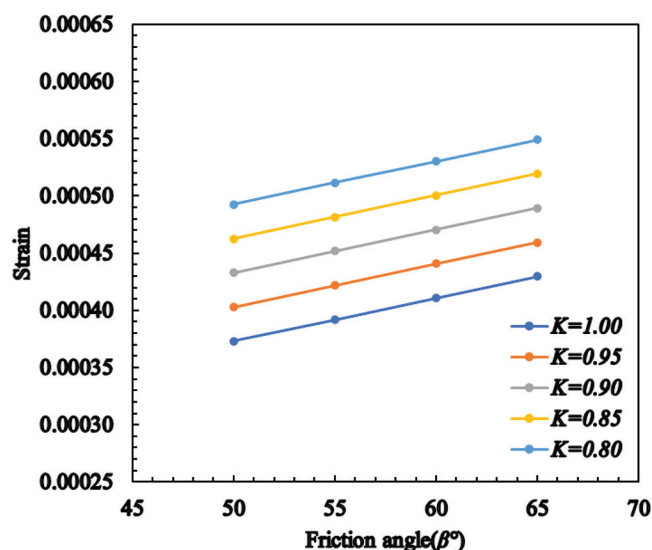


Fig. 16—Dynamic strain versus angle of friction corresponding to flow ratio.

which are very close to the value of dynamic strength obtained from CEB model in Eq. (30). The model equation that was suggested thus demonstrates that the current numerical studies are precise only in the concrete's dynamic compressive strength range, which is between 133 and 160 MPa.

CONCLUSIONS

In this study, under dynamic loading conditions, the behavior of stress and strain of high-strength concrete (HSC) is analyzed with respect to different friction angles and flow ratios. The suggested model equations, which illustrate the effect of the flow ratio and angle of friction, are established solely on the basis of analysis and model simulation results. From the numerical analysis, the following conclusions can be made:

Table 8—Comparative study of dynamic strength between present study and CEB model

Static strength σ_s , MPa	Strain rate $\dot{\epsilon}_s$, s ⁻¹	Dynamic strength based on CEB model σ_d , MPa	Model parameters from Fig. 9		Dynamic strength based on proposed model σ_d , MPa	Average deviation, %	Remarks
			Friction angle β , degrees	Flow ratio K			
120	21.86	136	52	1.00	136.55	-0.40	Highly satisfactory
			54	0.95	135.82	+0.13	
			57	0.90	136.86	-0.63	
			59	0.85	136.13	-0.10	
			61	0.80	135.40	+0.44	
100	65.00	150	60	1.00	150.64	-0.43	Highly satisfactory
			62	0.95	149.92	+0.05	
			64	0.90	149.19	+0.54	

1. The flow ratio and angle of friction both rise with dynamic stress, up to 15.15% and 21.12%, respectively, under dynamic loading conditions of HSC.

2. Dynamic stress increases but strain decreases with the increase in the flow ratio of HSC.

3. A significant number of multiple regression models are predicted with the correlation of dynamic stress and strain regarding both the flow ratio and angle of friction of HSC. The R^2 values reveal the accuracy of the predicted model in all the cases.

4. The proposed Fig. 15 and 16 can be used as a working manual for the practical application of HSC under dynamic loading.

5. The proposed model equations and Fig. 15 and 16 are valid only for HSC with a dynamic strength ranging from 133 to 160 MPa. In addition, the model equations are applicable for friction angles ranging from 50 to 65 degrees and flow ratios ranging from 0.80 to 1.00.

6. The comparison of results obtained from the proposed model equations and CEB model validates the legitimacy and capability of the proposed model.

The following further research can be carried out to enrich the knowledge:

1. The proposed multiple regression models are developed based on simulation results only. The experimental work can be conducted, and then a comparison of the obtained results can be made with those of the simulation and thus the validity of the model can be checked.

2. Finite element (FE) modeling of HSC can be performed on the basis of thermal effect under blast loading.

3. FE modeling of HSC can be performed with different specimen shapes and sizes under dynamic loading.

4. FE modeling of ultra-high-performance concrete can be performed under dynamic loading.

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Experimental Study on Interface Shear Resistance through Slant Shear Tests

by B. Li, A. Campos Sanchez, H.-C. Wang, Y. Yu, A. C. Ferche, and O. Bayrak

This paper summarizes the results of an experimental study investigating interface shear resistance through slant shear tests. A total of 54 tests were conducted on 6 x 12 in. (150 x 300 mm) cylinders with inclined cold joints. Five key test parameters were investigated: cold joint inclination, interface roughness, variation in concrete compressive strength between layers, casting age difference, and aggregate size. The test results demonstrated that the most influential parameters affecting interface shear resistance were interface roughness, casting age difference between layers, and aggregate size. Conversely, cold joint inclination and variations in compressive strength between layers were identified as comparatively less influential factors. A comprehensive analytical study was conducted including the comparison of five current design codes. A modified approach is proposed for calculating the interface shear resistance, compatible with current design codes' expressions. The proposed approach was validated using the experimental results generated from this study and a database of 124 slant shear test results from different research programs. Overall, the proposed approach resulted in more accurate estimations for the interface shear resistance, particularly for specimens with intermediary levels of amplitude at the interface.

Keywords: cold joint; concrete-to-concrete interface; interface roughness; interface shear resistance; slant shear.

INTRODUCTION

Cold joints are formed at the interface between layers of concrete cast at different times. Cold joints occur intentionally in new construction projects, such as staged construction, spliced girder bridges, and accelerated bridge construction techniques, as well as in retrofit and expansion projects where new structural elements are attached to existing structures. Cold joints may also occur unintentionally due to significant delay in placing consecutive layers of concrete. These cold joints, whether intentional or unintentional, represent a discontinuity between the concrete layers, potentially creating a weak link within the concrete member. Consequently, it becomes imperative to evaluate the capacity of cold joints to ensure structural integrity and to realistically assess the overall performance of the structure.

The primary factors contributing to the interface shear resistance of cold joints include cohesion of concrete, interface friction, and reinforcement crossing the interface. Cohesion of concrete refers to the ability of the concrete particles to stick together and resist separation or sliding when subjected to shear forces. Frictional forces develop along the interface when slip occurs. The magnitude of interface friction is influenced by factors such as interface roughness, aggregate interlock, and the presence of coexisting normal forces. The normal forces can stem from externally applied

loads and the clamping effect of the reinforcement crossing the cold joint. Well-anchored reinforcement crossing the interface plays a significant role in interface shear resistance. The widely accepted shear-friction model¹ evaluates the normal force to the interface as a clamping force when the reinforcement reaches its design yield strength. In addition, although not commonly accounted for in design expressions, the reinforcement crossing the interface contributes to the interface shear-transfer mechanism through dowel action.²

At the location of cold joints, current design specifications³⁻⁷ require the evaluation of the interface shear resistance. Generally, Code design expressions for interface shear resistance consider the contribution of cohesion, interface friction, and clamping forces. The coefficients of cohesion and friction are expressed as functions of the interface roughness. However, it has been shown that there are additional factors contributing to the interface shear resistance, including normal stresses acting on the interface, variation in concrete compressive strength between layers, casting age difference, and maximum aggregate size.⁸⁻¹⁸ These variables are deemed influential to the strength of cold joint interfaces; however, they are not incorporated in the Code expressions.

The influence of normal stresses on the interface is paramount in determining the interface shear resistance. Compressive stresses notably augment the friction along the interface, consequently leading to an elevation in interface shear resistance. Conversely, tensile stresses exhibit a contrasting effect. In the context of a slant shear test, both the normal force and shear force, which equilibrate the applied load, are subject to alteration by the inclination of the cold joint.^{8,9} As the inclination angle measured from the vertical increases, the contribution of the applied load to the normal force acting on the interface becomes more pronounced. This augmentation in normal force precipitates additional frictional forces, thereby culminating in heightened interface shear resistance.

Aggregate size was shown to significantly impact the shear resistance of concrete-to-concrete interfaces.^{10,11} The interlocking of aggregate is more effective with a larger aggregate size. The increase of aggregate interlock effect provides additional frictional resistance to the interface, thereby increasing the interface shear resistance.

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Table 1—Slant shear test matrix

Series	Groups	Angle, degrees	Interface roughness	Concrete strength	Casting age	Aggregate size
I-Angle	I-40	40	R1	N-N	3 days	A1-A1
	I-45	45	R1	N-N	3 days	A1-A1
	I-50	50	R1	N-N	3 days	A1-A1
	I-60	60	R1	N-N	3 days	A1-A1
II-Interface roughness	II-M	—	M	N-N	3 days	A1-A1
	II-NR	40	NR	N-N	3 days	A1-A1
	II-R1	40	R1	N-N	3 days	A1-A1
	II-R2	40	R2	N-N	3 days	A1-A1
III-Concrete strength	III-H1	40	R1	N-H1	3 days	A1-A1
	III-H2	40	R1	N-H2	3 days	A1-A1
IV-Casting age	IV-1/6	40	R1	N-N	4 hours	A1-A1
	IV-28	40	R1	N-N	28 days	A1-A1
	IV-56	40	R1	N-N	56 days	A1-A1
V-Aggregate size	V-A2	40	R1	N-N	3 days	A1-A2
	V-A3	40	R1	N-N	3 days	A1-A3

Note: Angle is inclination measured from vertical, shown as Fig. 5; M is placed monolithically; NR is not intentionally roughened; R1 is intentionally roughened with stripes; R2 is intentionally roughened with gridlines; N is normal-strength concrete ($w/c = 0.45$); H1 is higher-strength concrete ($w/c = 0.37$); H2 is higher-strength concrete ($w/c = 0.42$); A1 is 5/8 in. (16 mm) river gravel used in mixture; A2 is only fine aggregate used in mixture; A3 is 3/8 in. (9.5 mm) crushed concrete used in mixture.

Previous research¹¹⁻¹⁵ demonstrated that the shear resistance of a cold joint increases when the interface is intentionally roughened. Notably, these studies underscore that the increase of shear friction becomes progressively less significant beyond a certain threshold of interface roughness. These thresholds were implemented by current design specifications to determine the corresponding cohesion and friction factors when calculating the shear resistance of cold joints.

The variation in concrete compressive strength between layers is highly relevant regarding interface shear resistance. Although past studies suggest that the difference in compressive strength between layers of a cold joint does not significantly impact shear capacity,^{15,16} experimental data with environmental consistency and adequate boundary conditions is needed to support this conclusion.

Among the aforementioned key variables discussed, only the effect of interface roughness is explicitly considered in current design provisions for calculating the interface shear resistance of cold joints. Accordingly, cold joints are commonly roughened per design provisions to enhance the interface shear resistance in practice. However, the effect of other key variables discussed previously were often investigated on untreated smooth cold joints with no clear evidence supporting that the influence of these variables is negligible on the intentionally roughened interface. As a result, current specifications may overlook the negative effects of these variables, potentially leading to an overestimation of the shear resistance of intentionally roughened cold joints.

Therefore, the objective of this research was to investigate the implications of each influencing factor summarized previously on the interface shear resistance through an experimental program. Several experimental approaches have been employed by researchers to investigate the interface shear resistance of cold joints. Soltani and Ross¹⁷

conducted a thorough review of commonly employed test methods used to evaluate the characteristics of interface shear resistance based on the loading and boundary conditions. These methods include splitting tests, corbel with moment tests, pulloff tests, pushoff tests,^{10,16} slant shear tests,^{8,11-14} and composite beam tests.^{15,18} Among these various experimental methods, the slant shear test stands out for its simplicity in elucidating stress-transfer mechanisms and provides unobscured boundary conditions conducive to the evaluation of Code-prescribed expressions. A test matrix including 45 specimens categorized into five series was developed (Table 1). The test results made it possible to assess and introduce modifications to relevant design code provisions. This paper further examines whether the effect of each variable is influential when determining the interface shear resistance, especially with the application of interface roughening.

RESEARCH SIGNIFICANCE

- Experimental study: Conducted 54 slant shear tests on 6 x 12 in. (150 x 300 mm) cylinders with inclined cold joints to investigate interface shear resistance.
- Key parameters: Identified interface roughness, casting age difference, and aggregate size as the most influential factors on shear resistance.
- Analytical comparison: Compared five current design codes and proposed a modified approach for calculating interface shear resistance.
- Validation: Validated the proposed approach using experimental results and a database of 124 slant shear test results from various studies.
- Improved estimations: The proposed method provided more accurate shear resistance estimates, particularly for specimens with intermediary levels of interface amplitude.

EXPERIMENTAL PROGRAM

Specimen design and fabrication

The experimental program was structured into five distinct series, each focusing on one test variable. Within each series, the slant shear cylinders were further divided into groups with design alternatives corresponding to the variable under investigation. Each group contained three specimens of identical design. An overview of the test matrix is presented in Table 1.

Additionally, for each different cast, three 6 x 12 in. (150 x 300 mm) cylinders and three dog-bone specimens with a 4 x 4 in. (100 x 100 mm) cross-sectional area and a total length of 20 in. (500 mm) were fabricated to acquire the material properties of both the substrate and overlay. Standard compression tests were performed on cylinders following ASTM C39/C39M-21.¹⁹ Dog-bone specimens were used for direct tensile tests following CRD C164-92²⁰ procedures. The experimentally obtained material properties are summarized in Table 2.

A thorough procedure was employed to fabricate the 6 x 12 in. (150 x 300 mm) cylinders with inclined cold joints for

the slant shear tests. As shown in Fig. 1(a), wooden stands with the desired inclination were constructed to establish the inclined cold-joint interface. The substrate concrete was placed in this inclined position, with careful rodding and tamping performed while the cylindrical molds were attached to the wooden stand. The level of the substrate relative to the top of the cylindrical mold was measured to verify the accuracy of the cast, as shown in Fig. 1(b). This measurement ensures equal volumes of substrate and overlay, aligning the center of the cold joints to the midheight of the specimens. Subsequently, for certain specimens, intentional interface roughening was applied to the substrate, as shown in Fig. 1(c). The surface of substrate was cleaned with compressed air and pre-wetted with water prior to the cast of the overlay. The cylinder molds were detached from the wooden stand, and the overlay was cast on a horizontal surface after reaching the designated casting age difference. Specimens within the same series, sharing identical properties, had the substrate or overlay cast using the same batch of concrete to ensure material consistency.

All specimens in the experimental program were cured using water-saturated burlaps. After the cast of the substrates of the specimens, a piece of burlap was placed in each cylinder mold to maintain the curing condition before the cast of the overlay. This burlap was removed 7 days after the cast of the substrates for specimens with casting intervals longer than 7 days. For the rest of the specimens, the burlap was removed immediately prior to the cast of the overlay. After casting the overlay, the slant shear specimens were removed from the cylinder molds and cured by wrapping water-saturated burlap around the specimens for 7 days. Similarly, the material test specimens, including the compression cylinders and the dog-bone specimens, cast along with each batch of concrete, were also cured for 7 days after the cast by placing water-saturated burlap around each sample.

The fabrication procedure was consistent for the entirety of the specimens; however, specific methods were implemented depending on the test variable of each series. For instance, Series I focused on the impact of the cold joint inclination by investigating four different angles: 40, 45, 50, and 60 degrees measured from the vertical. The wooden stands were adjusted to achieve the corresponding inclination angles of the cold joints.

Series II examined the effect of the interface condition at the cold joint through different interface roughness levels. One

Table 2—Material properties

Series	Groups	$f'_{c\ sub}$, ksi	$f'_{c\ ovr}$, ksi	$f'_{t\ sub}$, psi	$f'_{t\ ovr}$, psi
I-Angle	I-40	2.48	2.55	334	332
	I-45				
	I-50				
	I-60				
II-Interface roughness	II-M	2.58	2.56	325	323
	II-NR				
	II-R1				
	II-R2				
III-Concrete strength	III-H1	2.50	3.75	325	335
	III-H2		2.95		290
IV-Casting age	IV-1/6	3.10	3.42	332	267
	IV-28	3.17	4.04		323
	IV-56	2.98	5.08		277
V-Aggregate size	V-A2	2.24	2.95	277	235
	V-A3	4.36	3.05		—

Note: $f'_{c\ sub}$ is concrete compressive strength of substrate; $f'_{c\ ovr}$ is concrete compressive strength of overlay; $f'_{t\ sub}$ is concrete tensile strength of substrate; $f'_{t\ ovr}$ is concrete tensile strength of overlay; 1 ksi = 6.8948 MPa; 1 psi = 0.0068948 MPa.

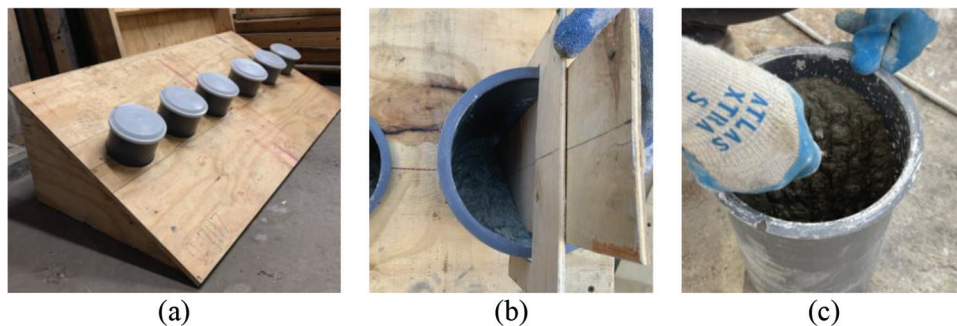


Fig. 1—Fabrication procedure: (a) cylinder mold and wood formwork for casting of substrate; (b) gauge for volume control; and (c) intentional roughening.

Table 3—Mixture design

Mixture	Cement, lb/yd ³	Water, lb/yd ³	Coarse aggregate, lb/yd ³	Fine aggregate, lb/yd ³	w/c
N	611	275	1720	1196	0.45
H1	635	235	1720	1196	0.37
H2	620	260	1720	1196	0.42

Note: 1 lb/yd³ = 0.593 kg/m³.

group of specimens had no intentional roughening applied to the substrate (NR), while two other groups were designed with different interface conditions to achieve varying roughness levels. The first treatment, denoted as R1, involved the application of unidirectional stripes spaced at 0.5 in. (12.7 mm) with a depth of 0.125 in. (3.2 mm) onto the substrate. A more intensive interface roughening, referred to as R2, was accomplished by applying orthogonal grid-lines to the substrate with the same spacing and depth as R1. Because intentional roughening of cold joints is encouraged in practice to enhance the interface shear resistance, the R1 treatment was applied to all slant shear specimens in Series I, III, IV, and V to investigate the effect of each test variable on the response of slant shear specimens with intentionally roughened interfaces.

The variation in concrete compressive strength between layers was introduced in Series III. The control mixture (N) used in this research maintained a water-cement ratio (w/c) of 0.45, as shown in Table 3. The average compressive strength of the control mixture is 2.7 ksi (18.6 MPa). For the overlay of each group in Series III, the w/c were reduced to 0.42 and 0.37, increasing the average compressive strength of the overlays to 3.0 and 3.8 ksi (20.7 and 26.2 MPa), respectively.

Unlike Series I, II, III, and V, for which the overlay was cast 3 days after the placement of the substrate, Series IV consisted of specimens with distinct casting age differences. For specimens in groups IV-1/6, IV-28, and IV-56, the overlay was cast 4 hours, 28 days, and 56 days after the substrate, respectively, to investigate the influence of casting age difference. With 4 hours of casting age difference, IV-1/6 simulated an unintentional delay during the construction phase. This short time difference between the casting of the substrate and overlay also allowed for the investigation of the minimum time difference to form a cold joint. The 28 days and 56 days casting age differences were chosen to simulate scenarios representative for various sequences of construction, retrofit, repair, or expansion.

In Series V, two different maximum aggregate sizes were adopted for the concrete mixtures of the overlay (A2 and A3). A2 substituted all coarse aggregate with fine aggregate, while A3 used crushed concrete with a maximum diameter of 3/8 in. (9.5 mm).

Experimental setup

The slant shear tests were performed by applying a uniaxial compression load under a constant displacement rate. The 6 x 12 in. (152 x 305 mm) slant shear cylinders were tested using the 440 kip load frame, following the guidelines of ASTM C882/C882M.²¹ The boundary conditions were



Fig. 2—Test setup of slant shear test.

idealized by using a tilt saddle at the top and a roller at the bottom, thereby allowing the specimen to develop rotation and lateral displacement. The experimental setup is illustrated in Fig. 2. To monitor the post-peak behavior of the cold joints, the tests were conducted at a constant displacement rate of 0.05 in./min (1.27 mm/min) until the applied load decreased to 50% of the maximum load.

To ensure a uniform loading condition, sulfur caps were applied to both the top and bottom of the cylinders following ASTM C617/C617M.²² The sulfur mortar compound was melted and poured into the specially designed vertical cylinder capper. The cylindrical specimen was then vertically placed on the capper, allowing the sulfur to solidify and form a contact surface. With a nominal strength of 9.0 ksi (62 MPa), the sulfur caps performed adequately during testing without fracture.

The load cell of the load frame was used to monitor the applied load. The slip of the cold joint was monitored during the slant shear test using a motion-capture system. The motion-capture system featured a pair of cameras that captured the spatial coordinates of each target. To quantify interface slip, five pairs of targets were strategically positioned along the cold joint—each pair spaced one inch apart—as illustrated in Fig. 3. One additional target was installed on each side of the cold joint to provide additional data for determining the cold joint orientation. The orientation of coordinates was carefully calibrated to align the axis with the loading direction. Locational data of the targets were then recorded using the data acquisition system throughout the test to calculate the slip between the overlay and the substrate.

Experimental data processing methodology

To validate the inclination of the cold joint interface, considering the potential variation in angle caused by

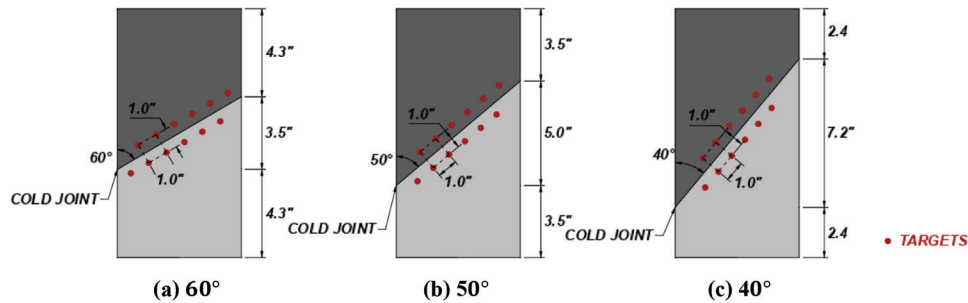


Fig. 3—Arrangement of targets based on different inclinations.

Table 4—Experimental results of slant shear test

Series	Groups	P_{avg} , kip	θ_{cj} , degrees	n_{ss}	n_{cc}	n_{cb}	V_{avg} , kip	δ_{s_peak} (in. $\times 10^{-3}$)
I-Angle	I-40	78	44	2	0	1	56	1.34
	I-45	82	48	2	1	0	53	3.55
	I-50	76	48	0	3	0	—	—
	I-60	84	67	0	2	1	—	—
II-Interface roughness	II-M	73	—	0	3	0	—	—
	II-NR	51	40	3	0	0	39	3.27
	II-R1	71	43	3	0	0	52	4.33
	II-R2	72	43	2	0	1	53	1.71
III-Concrete strength	III-H1	86	44	0	2	1	—	—
	III-H2	83	43	1	2	0	59	0.17
IV-Casting age	IV-1/6	110	39	0	3	0	—	—
	IV-28	92	40	3	0	0	70	1.09
	IV-56	129	39	2	1	0	105	3.20
V-Aggregate size	V-A2	44	39	3	0	0	32	3.93
	V-A3	71	43	2	0	1	55	1.75

Note: P_{avg} is average maximum applied load of three slant shear specimens in same group; θ_{cj} is as-built inclination of cold joint from regression of targets; n_{ss} is number of specimens that experienced slant shear failure out of three identical tests; n_{cc} is number of specimens that experienced crushing of concrete out of three identical tests; n_{cb} is number of specimens that experienced combined failure out of three identical tests; V_{avg} is average shear resistance of specimens governed by slant shear failure; δ_{s_peak} is average slip of cold joints corresponding to peak strength for specimens governed by slant shear failure; 1 kip = 4.4482 kN; 1 ksi = 6.8948 MPa; 1 in. = 25.4 mm.

inherent variability during construction, linear regression using the least-squares method was used for the coordinates of all 12 targets attached along the cold joint. Two regression lines determined based on the six targets attached on the overlay and the other six targets attached on the substrate were independently used to calculate two inclination angles. The average of the two inclination angles was taken as the as-built inclination, θ_{cj} , of the cold joint. The average inclination angles of three duplicate specimens in each group is presented in Table 4. This approach allowed for an accurate assessment of the inclination angle, ensuring the robustness of the validation process.

To obtain the slip along the cold joint, δ_s , a transformation of coordinates to the spatial data was necessary, as illustrated in Fig. 4. The locational data of the targets recorded during the tests were based on the coordinate with the x-axis parallel to the direction of loading, denoted as the global axis. Given the orientation of the cold joint, a transformation matrix was applied to convert the coordinates of the targets along the cold joint from the global axis to the coordinate with the x'-axis parallel to the cold joint, denoted as the local axis. In particular, the u -value of the target coordinate was projected

onto the local x'- and y'-axes. The same procedure was done for the v -value. In this way, the locational data of targets were transformed from global to local coordinates, u' and v' , by combining the projection results from both the u - and v -values. The transformation matrix in Eq. (1) transforms a target coordinate to its localized location

$$\begin{bmatrix} u' \\ v' \end{bmatrix} = \begin{bmatrix} \cos \theta_{cj} & -\sin \theta_{cj} \\ \sin \theta_{cj} & \cos \theta_{cj} \end{bmatrix} \begin{bmatrix} u \\ v \end{bmatrix} \quad (1)$$

where the as-built inclination angle, θ_{cj} , is measured from the global axis to the local axis, as shown in Fig. 4; and u' and v' are the local target coordinate values.

The targets were arranged into five pairs. This organization concentrates on the center of the interface. Each pair consisted of one target on the substrate layer and the other directly across from it on the overlay. Because the local x'-axis was parallel to the cold joint interface, the slip of the cold joint was represented by the local displacement along the x'-direction. In this manner, the slip was computed using the difference between the location of the target relative to its pair at each time step.

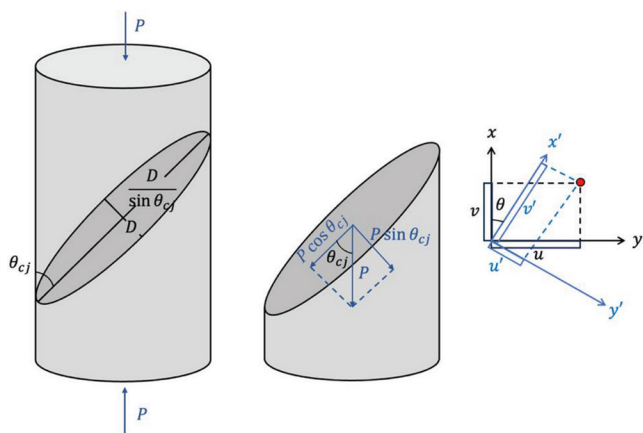


Fig. 4—Relationship between local and global coordinates.

The shear force acting on the interface, V , was calculated at each time step from the applied load, P , and cold joint inclination, θ_{cj} . The shear force-slip diagram for specimens governed by slant shear failure is presented in Appendix B.* The shear force acting along the interface was calculated as follows

$$V = P \cos \theta_{cj} \quad (2)$$

The shear stress, τ , at each time step was computed by dividing the shear force, V , by the area of the elliptical interface, as shown in Fig. 4, given by

$$\tau = \frac{4V \sin \theta_{cj}}{\pi D^2} \quad (3)$$

where D is the diameter of the specimen.

The methodology introduced in this section was used to determine the interface shear resistance and the cold joint behavior. The interface shear and the slip at peak load are listed in Table 4 and the results are discussed in the following section. Furthermore, interface normal and shear stress were evaluated from the experimental data and compared with the calculated values according to design specifications.

TEST RESULTS AND DISCUSSION

This section presents a discussion of the test variables, and the behavior of cold joints based on the experimental results. The slant shear experimental results are summarized in Table 4. Photographs of all specimens at failure are presented in Appendix A.

The interface shear resistance and the governing failure mode were the primary factors used in quantifying the effect of each test variable. The classification of failure modes for slant shear specimens was based on the observed crack pattern and the characteristics of fracture, resulting in three distinct modes: slant shear failure, as shown in Fig. 5(a); crushing of concrete, as shown in Fig. 5(b); and the combination of slant shear failure and crushing of concrete, as

shown in Fig. 5(c). For all specimens governed by slant shear failure, the specimens were separated into two pieces along the cold joint after the experiments, exposing the roughening patterns applied to the substrate.

The specimens in Series I reached similar maximum applied loads, P_{avg} . For all specimens governed by slant shear failure in Series I, the compressive stress in the direction of the compression load exceeded the concrete compressive strength of both the substrate and the overlay. These results suggest that the cold joints provided sufficient shear resistance for the compressive strength to develop until reaching a state of failure. Specimens with inclinations greater than 48 degrees predominantly exhibited a failure mode of concrete crushing.

The application of interface roughening played a crucial role in enhancing the shear resistance of cold joints. In the absence of intentional interface roughening, the shear resistance of II-NR was found to be 28% lower compared to intentionally roughened cold joints in II-R1. Interestingly, despite the additional grid treatment applied to II-R2, the maximum applied load remained unchanged compared to II-R1. Because both R1 and R2 treatments contained identical amplitude, the results also indicate that the influence of interface amplitude is more significant than interface texture.

The variation in concrete compressive strength between layers was found to have a negligible influence on the response of the slant shear specimens. Despite the 10 and 40% increase of compressive strength of the substrate, III-H1 and III-H2 had similar ultimate capacities. The failure mode observed in Series III specimens was a combination of slant shear failure and crushing of concrete. This outcome resulted from the sufficient shear resistance provided by cold joints with appropriate interface roughening, causing the weaker layer to experience crushing while the initiation of slant shear failure occurred at the cold joint.

The test results show that the casting age difference influences both the maximum applied load and the failure mode of the specimens. IV-56 exhibited a higher maximum applied load than IV-28, consistent with results from previous research.¹² The behavior of specimens with a casting age difference of four hours closely resembles the behavior of monolithically cast cylinders rather than slant shear specimens because all specimens in IV-1/6 experienced crushing of concrete. These results highlight the minimum casting age difference necessary to classify the interface between layers as a cold joint.

The test results from Series V demonstrate a significant effect of aggregate size on shear resistance. As the maximum aggregate size was reduced, the shear resistance decreased accordingly. Even with intentional interface roughening, specimens without coarse aggregate in V-A2 exhibited a 44% decrease in maximum applied load compared to specimens in I-40, while the maximum applied load of specimens with smaller maximum aggregate size in V-A3 remained similar.

Based on the test results, interface roughness of a cold joint and aggregate size appear to be the most effective factors for interface shear resistance. Decrease of interface shear resistance was observed when reducing the interface roughness or using only fine aggregate.

*The Appendix is available at www.concrete.org/publications in PDF format, appended to the online version of the published paper. It is also available in hard copy from ACI headquarters for a fee equal to the cost of reproduction plus handling at the time of the request.

Table 5—Shear-friction provisions of design codes

Design code	Surface roughness	c , ksi	μ	Design expression
AASHTO LRFD ³	Not roughened	0.075	0.6	$\tau_n = c + \mu(\rho f_y + \sigma_n)$
	Roughened	0.24	1.0	
ACI 318-19 ⁴	Not roughened	N/A	0.6λ	$\tau_n = \mu(\rho f_y \sin \alpha + \sigma_n) + \rho f_y \cos \alpha$
	Roughened	N/A	1.0λ	
CSA A23.3:19 ⁵	Not roughened	0.036	0.6	$\tau_n = \lambda[c + \mu](\rho f_y \sin \alpha + \sigma_n) + \rho f_y \cos \alpha$
	Roughened	0.073	1.0	
Design code	Surface roughness	c_a	μ	Design expression
<i>fib</i> MC 2010 ⁷	Smooth	0.35	0.6	$\tau_n = c_a f_{ctd} + \mu(\rho f_y \sin \alpha + \sigma_n) + \rho f_y \cos \alpha$
	Rough	0.45	0.7	
	Very rough	0.5	0.9	
Eurocode 2 ⁶	Smooth	0.2	0.6	$\tau_n = c_a f_{ctd} + \mu(\rho f_y \sin \alpha + \sigma_n) + \rho f_y \cos \alpha$
	Rough	0.4	0.7	

Note: τ_n is nominal interface shear resistance; c is cohesion factor of concrete-to-concrete interface; μ is coefficient of friction; c_a is unitless cohesion factor of concrete-to-concrete interface; ρ is reinforcement ratio of reinforcing steel crossing interface; f_y is yielding strength of reinforcing steel; σ_n is normal stress acting on interface; α is inclination of reinforcement crossing interface; λ is concrete density modification factor; f_{ctd} is design axial tensile strength of concrete of weaker layer; 1 ksi = 6.8948 MPa.

CODE EVALUATION AND COMPARISON

The shear resistance of cold joints was evaluated per design code provisions through the determination of interface shear resistance. The following evaluation and discussion only include the shear resistance of cold joints computed from specimens that experienced slant shear failure.

This section compares the test results with interface shear resistance calculated from five design code provisions. The design expressions for interface shear resistance from different design codes are presented in Table 5. AASHTO LRFD,³ CSA A23.3:19,⁵ Eurocode 2,⁶ and *fib* MC 2010⁷ consider the contribution of the cohesion of concrete, frictional resistance, and the reinforcement crossing the interface. Conversely, ACI 318-19⁴ considers the same components except for the cohesion of concrete. Notably, Eurocode 2⁶ and *fib* MC 2010⁷ present a different approach to consider the effect of concrete cohesion. Eurocode 2⁶ and *fib* MC 2010⁷ determine the cohesion of concrete as functions of tensile strength of the weaker concrete layer, whereas AASHTO LRFD³ and CSA A23.3:19⁵ dictate the cohesion of concrete only from the interface roughness and the roughening approach. Moreover, the frictional resistance is determined as a function of normal stress acting on the interface for all five design codes. Because the contribution of reinforcement crossing the interface is not the focus of this research, the difference between each design expression on the influence of reinforcement is not discussed in this paper.

The coefficients of cohesion and friction were determined by the interface roughness in all five expressions listed in Table 5. To classify the roughness of a concrete-to-concrete interface, the code specifications use the amplitude of roughening applied to the substrate. An interface is considered intentionally roughened when the amplitude exceeds 0.25 in. (6.4 mm) in AASHTO LRFD³ and ACI 318-19,⁴ whereas CSA A23.3:19⁵ defines an intentionally roughened interface as having an amplitude larger than 0.20 in. (5.1 mm). The spacing between dents is accounted for in Eurocode 2⁶ and *fib* MC 2010⁷ as a factor

to determine interface roughness. Eurocode 2⁶ considers a surface with an amplitude of 0.12 in. (3 mm) and a spacing of 1.60 in. (40.6 mm) to be rough. Both the average peak-to-valley height and the roughening approaches applied to the interface are inspected in *fib* MC 2010⁷ to evaluate roughness level. Interfaces blasted by sand or high-pressure water with a mean peak-to-valley height larger than 0.06 in. (1.5 mm) are considered rough surfaces. Meanwhile, indented surfaces or surfaces treated with high-pressure water jetting are considered in *fib* MC 2010⁷ as very rough when the mean peak-to-valley height exceeds 0.12 in. (3 mm).

Current design expressions

The peak shear stress obtained from the experiments, τ_{test} , and the interface shear resistance calculated from five design expressions³⁻⁷ are presented in Table 6. Peak shear stress of each specimen and the calculated shear resistance were plotted as data point for each specimen in Fig. 6. According to AASHTO LRFD,³ ACI 318-19,⁴ and CSA A23.3:19,⁵ specimens intentionally roughened with a depth of 0.125 in. (3.2 mm) (R1 and R2) are classified as not intentionally roughened interface due to insufficient amplitude. The root mean squared error (RMSE) and coefficient of determination (R^2) listed in Table 6 were used as statistical parameters to determine the accuracy of the calculated interface shear resistance, computed as shown in Eq. (4) and (5). The R^2 value of interface shear resistance calculated from AASHTO LRFD,³ ACI 318-19,⁴ and CSA A23.3:19⁵ is 0.84, 0.80, and 0.82, respectively. Consequently, the shear resistance of the cold-joint interface calculated from these codes shows approximately 40% conservatism, as observed in Fig. 6(a), (b), and (c). Eurocode 2⁶ considers the R1 and R2 cold joints to be both rough interfaces. *fib* MC 2010⁷ classifies the R1 cold joints as rough interfaces, while the R2 cold joints as very rough interfaces, based on the roughness amplitude that was achieved. Consequently, the experimental results have a better alignment with the estimations from Eurocode 2⁶ and *fib* MC 2010,⁷ specifically with an R^2 value of 0.90 and 0.91, respectively.

Table 6—Experimental results and predictions from design provisions

Specimen	τ_{test}	Current Code expression					RI			LIF		
		τ_{AASHTO}	τ_{ACI}	τ_{CSA}	τ_{Euro}	τ_{fib}	τ_{AASHTO}^{RI}	τ_{ACI}^{RI}	τ_{CSA}^{RI}	τ_{AASHTO}^{LIF}	τ_{ACI}^{LIF}	τ_{CSA}^{LIF}
I-40-1	1.27	1.38	1.14	1.21	0.88	0.87	0.76	0.68	0.72	1.07	0.91	0.96
I-40-3	1.43	1.66	1.42	1.50	1.08	1.07	0.93	0.85	0.89	1.30	1.14	1.19
I-45-1	1.43	2.03	1.79	1.87	1.34	1.33	1.15	1.08	1.11	1.59	1.43	1.49
I-45-2	1.43	1.78	1.54	1.61	1.16	1.15	1.00	0.92	0.96	1.39	1.23	1.29
II-R1-1	1.23	1.33	1.09	1.16	0.85	0.84	0.73	0.65	0.69	1.03	0.87	0.92
II-R1-2	1.22	1.38	1.14	1.21	0.89	0.88	0.76	0.68	0.72	1.07	0.91	0.97
II-R1-3	1.31	1.46	1.22	1.29	0.94	0.93	0.81	0.73	0.77	1.13	0.97	1.03
II-R2-1	1.29	1.26	1.02	1.09	1.01	0.79	0.69	0.61	0.65	0.97	0.81	0.87
II-R2-3	1.17	1.43	1.19	1.27	1.17	0.91	0.79	0.72	0.75	1.11	0.95	1.01
II-NR-1	0.95	0.58	0.51	0.54	0.58	0.55	0.58	0.51	0.54	0.58	0.51	0.54
II-NR-2	0.78	0.47	0.39	0.43	0.46	0.43	0.47	0.39	0.43	0.47	0.39	0.43
II-NR-3	0.94	0.52	0.45	0.48	0.51	0.48	0.52	0.45	0.48	0.52	0.45	0.48
III-H2-3	1.41	1.52	1.28	1.36	0.98	0.98	0.85	0.77	0.81	1.18	1.03	1.08
IV-28-1	1.62	1.63	1.39	1.46	1.08	1.07	0.91	0.83	0.87	1.27	1.11	1.16
IV-28-2	1.70	1.73	1.49	1.57	1.16	1.15	0.97	0.90	0.93	1.35	1.20	1.25
IV-28-3	1.47	1.41	1.17	1.24	0.93	0.92	0.78	0.70	0.74	1.09	0.93	0.99
IV-56-1	2.34	2.14	1.90	1.98	1.44	1.43	1.22	1.14	1.18	1.68	1.52	1.58
IV-56-2	2.40	2.24	2.00	2.07	1.50	1.49	1.27	1.20	1.23	1.76	1.60	1.65
V-A2-1	1.23	1.15	0.91	0.98	0.71	0.70	0.62	0.54	0.58	0.88	0.72	0.78
V-A2-2	1.21	1.29	1.05	1.12	0.81	0.80	0.70	0.63	0.67	1.00	0.84	0.89
V-A2-3	1.24	1.28	1.04	1.12	0.81	0.80	0.70	0.63	0.66	0.99	0.83	0.89
V-A3-2	0.70	0.89	0.65	0.61	0.56	0.55	0.46	0.29	0.36	0.67	0.39	0.49
V-A3-3	0.85	1.04	0.80	0.74	0.67	0.66	0.55	0.36	0.44	0.80	0.48	0.59
RMSE	—	0.58	0.64	0.61	0.45	0.43	0.29	0.28	0.26	0.30	0.42	0.37
R^2	—	0.84	0.80	0.82	0.90	0.91	0.96	0.96	0.97	0.96	0.92	0.93

Note: τ_{test} is interface shear resistance obtained from slant shear test; τ_{AASHTO} is nominal interface shear resistance calculated from AASHTO LRFD; τ_{ACI} is nominal interface shear resistance calculated from ACI 318-19; τ_{CSA} is nominal interface shear resistance calculated from CSA A23.3:19; τ_{Euro} is nominal interface shear resistance calculated from Eurocode 2; τ_{fib} is nominal interface shear resistance calculated from *fib* MC 2010; 1 ksi = 6.8948 MPa.

$$RMSE = \sqrt{\frac{\sum_{i=1}^n (Y_{pred} - Y_{ref})^2}{n}} \quad (4)$$

$$R^2 = 1 - \frac{\sum_{i=1}^n (Y_{pred} - Y_{ref})^2}{\sum_{i=1}^n (Y_{ref})^2} \quad (5)$$

Rough interface assumption (RI)

Over-conservative results are obtained when the intentionally roughened interface is considered as an untreated interface due to insufficient amplitude according to the code expressions in AASHTO LRFD,³ ACI 318-19,⁴ and CSA A23.3:19.⁵ Therefore, the interface shear resistance of the specimens was recalculated by assuming the cold joints as roughened interfaces, referred to as RI approach. This approach used expressions from AASHTO LRFD,³ ACI 318-19,⁴ and CSA A23.3:19,⁵ and the results are presented in Table 6 and Fig. 6. Although the R^2 value was improved to 0.96, 0.96, and 0.97, respectively, by the intentionally roughened interface factors in AASHTO LRFD,³ ACI 318-19,⁴ and CSA A23.3:19,⁵ the amount of

unconservative cases observed in the RI approach indicated a need for refinement.

Proposed approach: linearly interpolated factors (LIF)

Because the shear resistance of the cold joints is underestimated by current design expressions and overestimated by the previously introduced RI approach in AASHTO LRFD,³ ACI 318-19,⁴ and CSA A23.3:19,⁵ a modified approach is proposed to obtain the coefficients of cohesion and friction. To better represent the shear resistance of cold joints with intermediary levels of interface roughness, linear interpolation was employed to derive the cohesion and friction coefficient based on the amplitude of the interface roughness. Coefficients of cohesion and friction obtained from the original and the LIF methods are demonstrated in Fig. 7. The interface shear resistance calculated using the linearly interpolated factors, referred to as the LIF approach, is listed in Table 6 and plotted in Fig. 6.

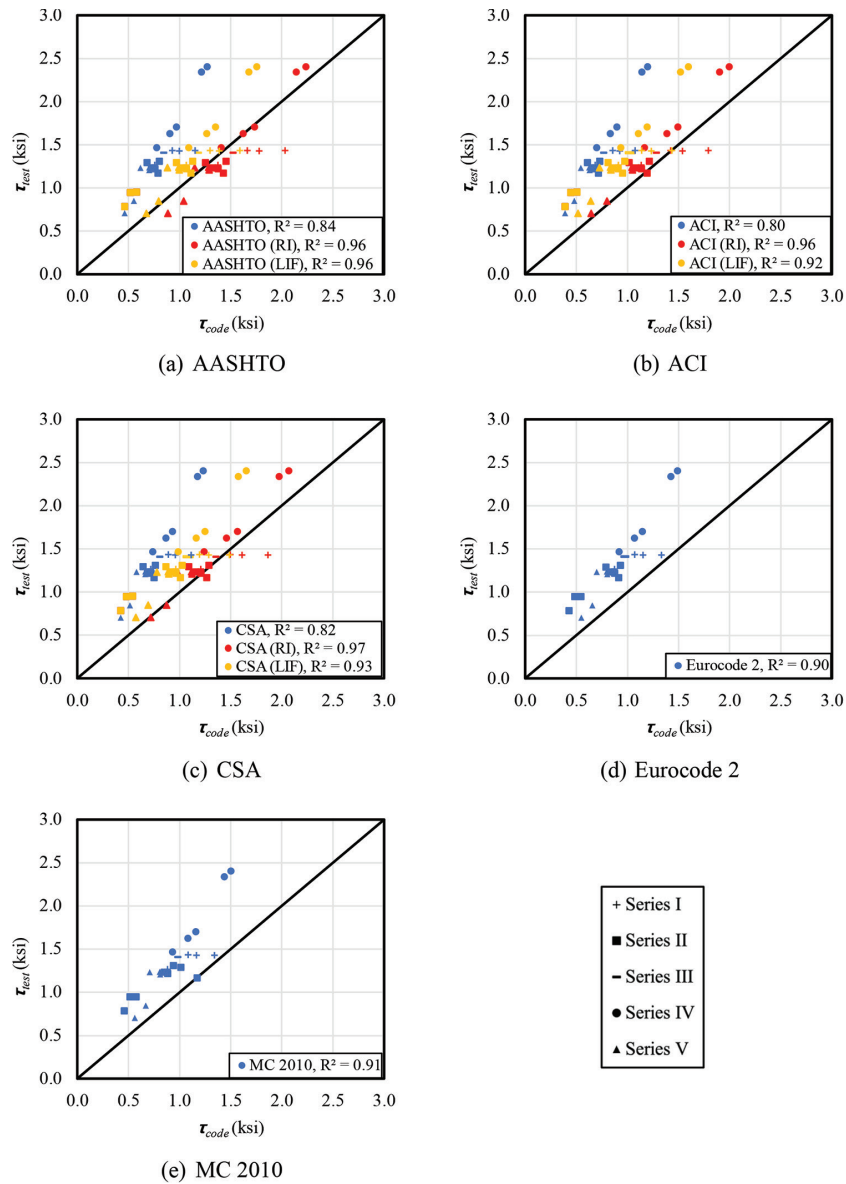


Fig. 6—Experimental and predicted results. (Note: 1 ksi = 6.8948 MPa.)

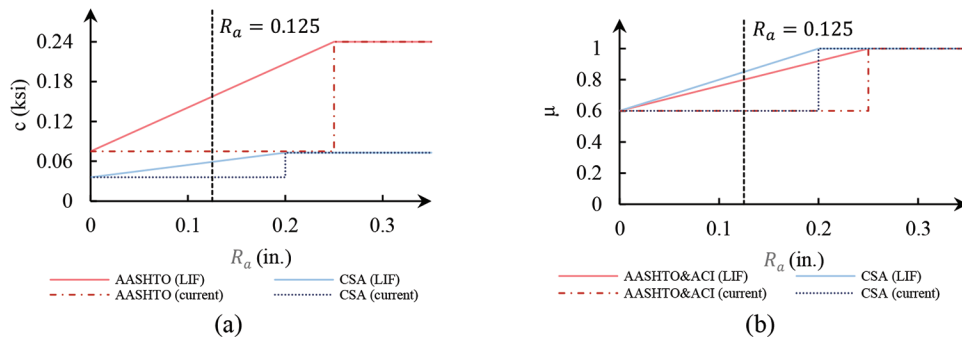


Fig. 7—Variation of: (a) cohesion factor c ; and (b) coefficient of friction μ with amplitude of interface roughening. (Note: 1 ksi = 6.8948 MPa.)

The interface shear resistance of the specimens is better predicted with the LIF method compared to current design expressions. The R^2 value of interface shear resistance calculated from applying LIF to AASHTO LRFD,³ ACI 318-19,⁴ and CSA A23.3:19⁵ was improved to 0.96, 0.92, and 0.93, respectively. This indicates that intentional interface

roughening should be considered even if the amplitude is smaller than the required height specified in these three design specifications to accurately predict the shear resistance of cold joints. Also, the total unconservative counts observed using the LIF approach decreased from 24 cases to three cases compared to the RI approach.

Table 7—Database investigation of slant shear tests

Source	<i>N</i>	<i>D</i> , in.	Geometry	θ_{ej} , degrees	<i>R_a</i> , in.	Roughening approach
Figueiredo et al. ¹¹	75	5.51	Cylindrical	30	0.034 to 0.22	Hand-scrubbing; vibrating
Santos and Júlio ¹²	150	5.91	Prismatic	30	0.01 to 0.048	Wire-brushing; sandblasting; shotblasting; hand-scrubbing
Diab et al. ¹⁴	150	5.91	Cylindrical, prismatic	30	0.12, 0.24	Patterned grooves
Saldanha et al. ¹³	12	5.91	Prismatic	30	0.20, 0.59	Hand-scrubbing; patterned grooves
Hu et al. ⁸	96	2.77	Cylindrical	30, 40	0.12, 0.24	Patterned grooves

Note: *N* is number of slant shear tests; *D* is diameter of cylindrical specimen, or side length of prismatic specimen; θ_{ej} is inclination of cold joint measured from vertical; *R_a* is amplitude of roughness; 1 in. = 25.4 mm.

VALIDATION STUDY

Database analysis

This section explores earlier experimental studies^{8,11-14} that examined the impact of various variables necessary for implementing the LIF approach. The database was constructed to include relevant studies that used slant shear tests and reported the following experimental data: roughness amplitude, cold joint inclination, and failure mode. The selected experimental programs are used to validate the proposed LIF method. The database covered common roughening methods and amplitudes; therefore, the applicability of the LIF approach could be well-justified in common scenarios. The range of variables and properties of specimens are listed in Table 7, while Appendix C includes the test results of the database. The normal stress and shear stress acting on the interface at peak load were computed according to the variables and test results reported.

Hu et al.⁸ conducted 24 traditional slant shear tests along with 72 dynamic slant shear tests. Rectangular indentions with two different depths were applied to the interface to quantify the surface roughness. The amplitude of interface roughening was 0.12 and 0.24 in. (3 and 6 mm). Additionally, the specimens had two cold joint inclinations from vertical—30 and 40 degrees—to investigate the interfacial behavior.

Figueiredo et al.¹¹ conducted 75 slant shear tests to study the bond performance between normalweight and sand-lightweight concrete. Five types of interface roughening approaches were applied to the specimens: smooth, vibrated, and three intentionally roughened methods. A laser-based displacement sensor was used to accurately measure the amplitude of roughness.

Santos and Júlio¹² performed 150 prismatic slant shear tests to investigate interface roughness and casting age difference. The interface shear stress for prismatic specimens is computed by considering the area of the cold joint as rectangular instead of elliptical. Different methods of interface preparation were applied to the substrate, which included wire-brushing, sandblasting, shotblasting, and hand-scrubbing. The roughness profile of the substrate was quantified using a laser roughness analyzer.

Saldanha et al.¹³ proposed a modified slant shear test approach on prismatic, reinforced concrete specimens. Traditional slant shear tests were conducted to validate the proposed method. Two interface roughening approaches were applied to both types of slant shear specimens, including hand scrub interface and wavy interface. The test results

of 12 slant shear experiments and the roughness profile measured by laser sensor were reported by this research.

Diab et al.¹⁴ tested cylindrical and prismatic slant shear specimens to investigate the interface behavior between self-consolidating concrete and old conventional concrete. Four types of interface roughness were applied to the specimens: hand brush, mechanical roughness, 0.12 in. (3 mm) depth x 0.12 in. width grooves, and 0.24 in. (6 mm) depth x 0.24 in. width grooves.

Force components to determine shear and normal stress acting on the cold joint could be clearly identified only when specimens were governed by the slant shear failure. Because the validation was conducted by comparing interface shear resistance calculated using the LIF approach and the experimental results, only experiments governed by slant shear failure are included in the validation to obtain the shear resistance of the interface. As a result, a total of 124 slant shear test results were included to validate the LIF approach.

Validation of LIF approach

Test results from database investigation are used to validate the LIF approach based on shear-friction expressions in AASHTO LRFD,³ ACI 318-19,⁴ and CSA A23.3:19.⁵ The validation was performed by comparing cold joint shear resistance obtained from previous experiments^{8,11-14} and interface shear resistance calculated using the LIF approach. The validation exclusively focuses on roughened interfaces with intermediary levels of amplitude to which the LIF approach is applicable. The test results of the slant shear database and experimental program in this research are presented in Fig. 8, along with the calculations from current design specifications and the LIF approach.

Based on the validation results, it is evident that the LIF approach improves the accuracy of shear-friction expressions with minor unconservative calculations that occurred only when applying LIF to AASHTO LRFD.³ The *R*² value of interface shear resistance calculated from applying LIF to AASHTO LRFD,³ ACI 318-19,⁴ and CSA A23.3:19⁵ was improved from 0.78, 0.72, and 0.77 to 0.91, 0.84, and 0.87, respectively.

The LIF approach appears to be more impactful under higher normal and shear stress. According to the conclusion from the experimental program, the shear resistance of cold joints with intentional roughening is expected to be higher than non-roughened interface. The shear resistance for cases reaching high peak normal and shear stress is proven to be sensitive to the contribution of interface roughness, even when the amplitude of roughness is lower than the

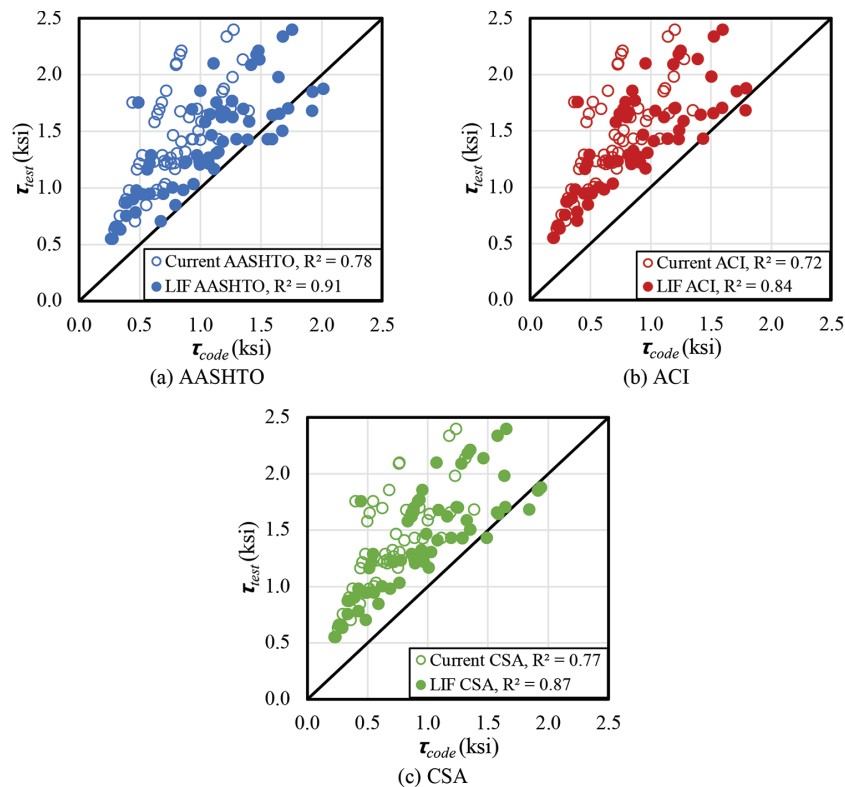


Fig. 8—Database validation for LIF approach based on different specifications.

requirement of specifications. The LIF approach improves the accuracy of calculation by directly reflecting the effect of roughening amplitude, while current design expressions fail to address the contribution of intermediary interface roughness. This implies that the LIF approach can lead to substantial improvements in calculating the shear resistance of cold joint with a roughened interface.

CONCLUSIONS

This study presents a comprehensive investigation into the behavior of cold joints through a series of slant shear experiments. The key experimental variables include inclination angle, interface roughness, concrete strength, casting age difference, and maximum aggregate size. The experimental results were compared against the interface shear capacities calculated using five design specifications³⁻⁷ to evaluate the accuracy of current interface shear expressions.

The conclusions drawn from this research are summarized as follows:

1. The application of interface roughening and the use of concrete with a larger maximum aggregate size significantly enhanced the shear resistance of cold joints. However, current design specifications only address the effect of interface roughness with no modification directly accounting for aggregate size.
2. The inclination angle and the compressive strength difference had a negligible effect on the shear resistance of roughened cold joints. However, the increase in the inclination angle shifted the failure patterns of cold joints from slant shear failure to crushing of concrete.
3. Eurocode 2⁶ and *fib* MC 2010⁷ provided reasonably accurate estimates of the cold joint resistance for specimens

in this research. This accuracy is achieved by including the effect of concrete tensile strength and the contribution of roughened interfaces. AASHTO LRFD,³ ACI 318-19,⁴ and CSA A23.3:19⁵ tend to underestimate the interface shear resistance of cold joints by considering the interfaces as untreated surfaces, neglecting the effect of interface roughening when the amplitude is lower than the required threshold.

4. The linearly interpolated factors (LIF) approach was proposed to reflect the actual interface roughness in AASHTO LRFD,³ ACI 318-19,⁴ and CSA A23.3:19.⁵ By linearly interpolating coefficients of cohesion and friction based on the roughening amplitude, the LIF approach improves the accuracy of calculated cold joint resistance.

5. The interface shear resistance calculated using the LIF approach demonstrated better alignment with the cold joint database comparing to the shear resistance calculated based on current AASHTO LRFD,³ ACI 318-19,⁴ and CSA A23.3:19⁵ specifications.

While the results in this paper provide promising advances in the evaluation of interface shear resistance of cold joints, two specific topics are suggested to complement this research. First, ongoing research efforts should address the minimum casting age difference needed for an interface to be considered as a cold joint. Because all specimens in IV-1/6 experienced crushing of concrete, these results suggest a lower limit for the casting age difference of a cold joint. Future research is needed to investigate the minimum time interval required for the formation of cold joints under the influence of environmental and material factors, such as humidity and concrete mixture. Second, the experimental results reveal that aggregate size and casting age difference is influential

to the interface shear resistance of a cold joint. However, current design expressions only consider the effect of interface roughness. Further research is required to quantify the influence of different levels of aggregate size on interface shear resistance and incorporate relevant modifications into design specifications.

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Cement-Cellulose Excelsior Composites: Accounting for Moisture

by Naveen Krishnan, O. Burkan Isgor, and W. Jason Weiss

Cellulose excelsior (CE), sometimes referred to as wood wool, is a shredded wood product with a strand thickness of 0.12 to 0.64 mm, a width that is up to three times the thickness, and a straw-like consistency. CE can be combined with cementitious materials to form cellulose-cement composite (C3) materials. The production of C3 materials requires accurate methods to quantify the moisture in the CE. This paper describes a methodology to obtain the surface-dry state repeatably by using a centrifuge and an approach to control water content, which can be used to provide consistency in C3 production. The mixture water absorbed by the CE can be estimated from the desorption isotherm when the relative humidity of the CE is greater than 80%. Isothermal calorimetry was used to confirm the accuracy of using the desorption model to quantify the water uptake.

Keywords: absorption; cellulose; cellulose-cement composites (C3); centrifuge; desorption isotherm; isothermal calorimetry; moisture content.

INTRODUCTION

Cellulose excelsior (CE), or wood wool, has been used as a building material in various nonstructural applications. For example, CE has been used as an acoustic material, a thermal insulation material, or as nonstructural boards.¹ However, the use of CE as a structural cellulose-cement composite (C3) material has been limited. CE has significant benefits as a sustainable building material for residential and light commercial construction. When mixed with a mixture-optimized cementitious binder and water, compacted and cured, C3 has the potential to be used as an alternative construction material to dimensional lumber, metal studs, and sheet products such as plywood and drywall.^{2,3}

This paper focuses on one such structural C3 material system.⁴ The main constituents of C3 are CE, cementitious binder, and water. C3 is designed to be a carbon-negative material with a targeted CO₂ factor of -0.09 kg CO₂e/kg.⁴ A primary component of the C3 material is CE, made from low-value, small-diameter trees that can be harvested as a part of forest management to reduce the potential for fuel in forest fires.⁵ The carbon stored in CE during tree growth through photosynthesis contributes to the overall CO₂ reduction.⁶⁻⁸ However, the main reduction arises from the carbonation potential of the binder phase. The binder system in C3 is formulated using low-cement-content and low-carbon materials such as limestone, fly ash, and calcined clay, which are capable of gradually absorbing CO₂ from the atmosphere over time.⁹⁻¹² In addition, the cementitious binder makes the C3 resistant to rot, fungi, and fire, and makes the composite durable against chemical, biological, and physical attacks from the environment.

The CE material is cellulose consisting of strands with a thickness of 0.15 to 0.64 mm, a width up to three times its thickness, and a length of 10 to 50 mm,¹³ as shown in Fig. 1. CE has the fibrous and porous nature of wood, as well as a large surface area, which influences both the rate and amount of water absorption, which in turn affects the C3's fresh properties, hydration/pozzolanic reactions, and eventual mechanical properties.¹³ Water plays a fundamental role in cementitious reactions. CE primarily consists of cellulose but also includes significant amounts of hemicellulose (10 to 20%) and lignin (20 to 30%), along with minor quantities of extractives and inorganic compounds.^{14,15} The cellulose, which makes up approximately 40 to 50% of the material, has the structural formula (C₆H₁₀O₅)_n composed of repeating glucose units linked by β(1 → 4) glycosidic bonds.^{16,17} The hydrophilic nature of cellulose is attributed to the numerous hydroxyl (-OH) groups along its chains, which form hydrogen bonds with water molecules, facilitating effective water uptake.¹⁵ This absorption is further enhanced by the fibrous and porous structure of CE, allowing it to draw in and retain water through capillary action.

The water that is available for cementitious reactions significantly influences the properties (for example, porosity, strength, and so on) of the paste. CE, in its as-received state, contains inherent moisture, typically ranging from 8 to 12%. Water is then added to the CE and cementitious binders to achieve the desired fresh and hardened performance requirements. The amount of water that is available for cementitious reactions is affected by CE's absorption and desorption characteristics. An unsaturated CE (that is, one with a moisture content below its absorption capacity) will absorb a portion of the mixing water. This can result in a lower amount of available water to react with the cement, effectively reducing the water-cementitious materials ratio (*w/cm*). Conversely, the CE water could also be released from the large pores to the mixture.^{18,19} This is particularly important because water released from the CE after setting can be effective in internally curing the mixture.²⁰ Therefore, accounting for the water and understanding the absorption/desorption properties are important in proportioning C3 systems and predicting their short- and long-term performance. However, this can be rather challenging because, as

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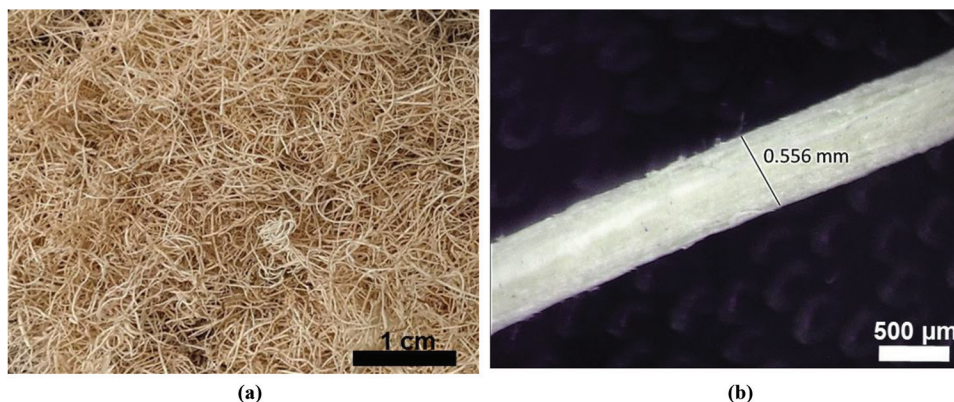


Fig. 1—CE: (a) photograph of bulk material; and (b) microscopic view of single strand.

reported by Tabarsa and Ashori,²¹ the tree species used to prepare CE and the preparation procedures impact the water sorption properties.

There is limited literature available on accounting for/controlling the water content and understanding the absorption characteristics of the CE during its mixing stage with cementitious binders. It is critical to quantify the amount of water that is absorbed by the CE in the C3 mixtures to maintain a predetermined w/cm . The main objective of this paper is to investigate the water desorption characteristics of CE in C3 mixtures, with the goal of informing mixture quality control procedures to produce consistent C3 materials.

RESEARCH SIGNIFICANCE

This paper outlines a procedure for C3 to account for the fluid sorption and desorption behavior of the cellulose. Segmenting the portion of the water in the paste from the portion in the CE at the time of setting is important for determining the pore structure of the paste, its w/cm , and the water that remains for internal curing. The paper applies this approach to the C3 materials. C3 materials can potentially remove fuel sources from the forest that are not readily used in other applications while creating a construction material with a low carbon footprint.

EXPERIMENTAL STUDY

Materials

The CE used in this study was obtained from Ponderosa pine from the state of Washington and consisted of strands with a thickness of 0.15 to 0.64 mm and a width of three times the thickness. The CE was prepared in two lengths (12.7 and 25.4 mm) using an N60 guillotine cutting machine. A chopping rate of 308 kg/h was used for the 12.7 mm cuts and 680 kg/h for the 25.4 mm cuts. Post-cut length analysis on 30 randomly selected fibers showed mean lengths of 13.0 ± 7.4 mm for the 12.7 mm batch and 22.9 ± 8.9 mm for the 25.44 mm batch. A Welch's t -test ($p \ll 0.05$) confirmed a statistically significant difference between the two batches.

The C3 mixtures were prepared using ordinary portland cement (OPC) as the binder. The oxide content of the OPC, as determined by X-ray fluorescence (XRF), is shown in Table 1. The clinker composition of the cement consists of 69% C_3S , 5.1% C_2S , 6.6% C_3A , and 6.9% C_4AF .²² The

Table 1—Oxide composition of cement from XRF analysis

Material	Cement
Oxide	Mass %
Na ₂ O	0.21
MgO	1.43
Al ₂ O ₃	3.95
SiO ₂	19.95
P ₂ O ₅	0.10
SO ₃	2.55
Cl	0.00
K ₂ O	0.48
CaO	63.32
TiO ₂	0.19
Mn ₂ O ₃	0.07
Fe ₂ O ₃	2.28
ZnO	0.01
LOI	2.71
Limestone	4.31

Note: LOI is loss on ignition.

specific gravity (SG) of the cement, found using a Le Chatelier flask experiment, was 3.05.²³

Methods

Determination of CE moisture state—An experimental methodology used a centrifuge to condition the CE to arrive at a “surface-dry (SD)” state (that is, all pores within the material are filled with water, but no free water is present on the surface). The CE (approximately 100 g of as-received CE) was first soaked under water for 24 hours, after which time the material was placed in an 11.4 cm radius bowl and spun in the centrifuge at 2000 rpm for 3 minutes to use centrifugal force to remove excess water from the fibers,^{24–26} as shown in Fig. 2. This method was helpful for the reproducible preparation of C3.

The speed of the centrifuge was selected based on prior studies on porous materials.^{24,27,28} Cheng et al.²⁸ demonstrated that centrifugal speeds between 2000 and 2500 rpm were

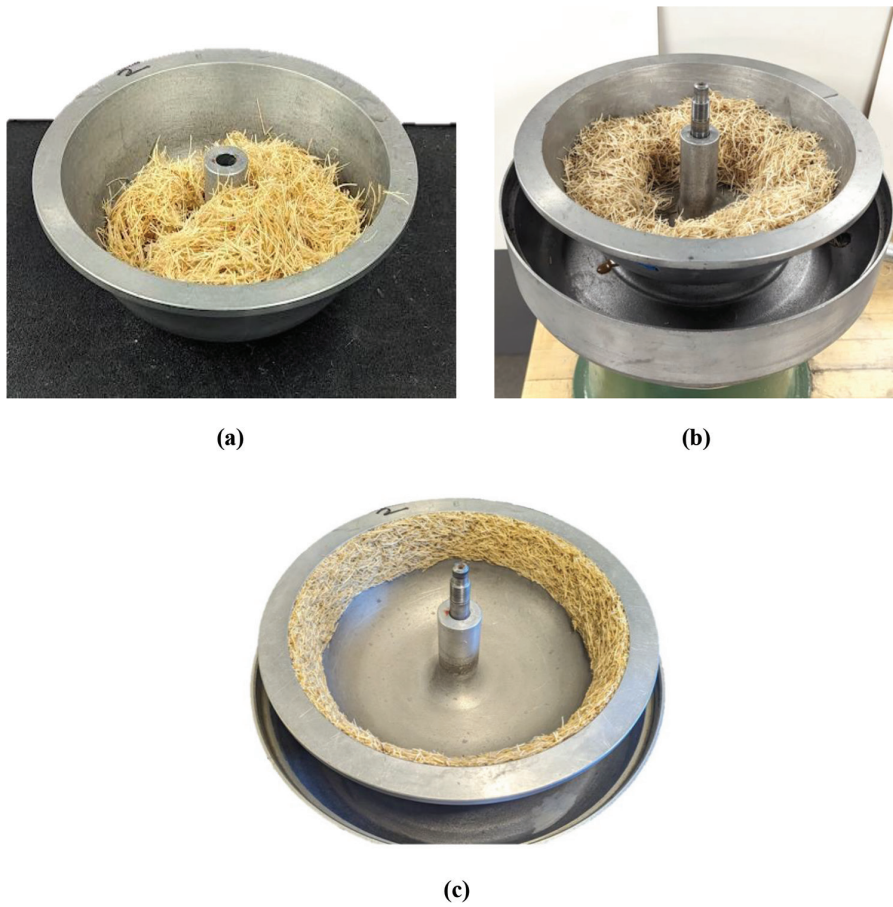


Fig. 2—Centrifuging procedure for measuring water absorption in CE: (a) OD samples are shredded and weighed; (b) shredded samples are soaked in deionized water for 24 ± 4 hours before being placed into centrifuge bowl; and (c) excess water drains out after centrifuging for 30 minutes at 2000 rpm.

effective in removing free water from pulp and microcrystalline cellulose without damaging fiber integrity. Operating at higher speeds (for example, >4000 rpm) risks structural degradation of CE strands and was avoided. By operating the centrifuge at a speed of 2000 rpm, pore sizes larger than $\sim 296 \mu\text{m}$ were emptied.²⁴ The optimal time required to reach a consistent moisture state was evaluated and determined to be 15 minutes, which was needed to allow the water leaving the system to come to equilibrium, approaching no water removal for the last minute, as discussed later in the document.^{24,26} Experiments were conducted in triplicate to determine duration by observing the variations in water absorption by the CE at each interval.

The moisture content of the CE was calculated by obtaining the mass of the CE at the SD state and at the oven-dried (OD) state, achieved by oven-drying the CE at the SD state for 24 hours at 105°C . The recorded mass using an analytical balance with a precision of $\pm 0.001 \text{ g}$ ($\pm 1 \text{ mg}$) was then used in Eq. (1) to calculate the moisture content, θ (%)²⁹

$$\theta = \frac{M_{SD} - M_{OD}}{M_{OD}} \times 100 \quad (1)$$

where M_{SD} and M_{OD} are the mass of the CE at the SD state and OD state, respectively. The absorption of both the 12.7 and 25.4 mm CE was measured.

The SG of CE was measured using the pycnometer method.³⁰ The SG was calculated using the formula shown in Eq. (2)

$$\text{SG} = \frac{\Delta M_{\text{sample}}}{(\Delta M_{\text{water}} - \Delta M_{\text{sample, water}})} \quad (2)$$

where ΔM_{sample} is the mass of the sample; ΔM_{water} is the mass of water filling the empty pycnometer; and $\Delta M_{\text{sample, water}}$ is the mass of water added to the sample in the pycnometer. An analytical balance with a precision of $\pm 0.01 \text{ g}$ ($\pm 10 \text{ mg}$) was used to ensure accurate mass determination. For the SG of CE in the SD condition (SG_{SD}), the CE samples were first soaked in deionized water for 24 hours and then centrifuged to remove excess water before weighing. Approximately 30 to 35 g of CE, occupying approximately one-third of the pycnometer jar volume, was used for each measurement. All measurements were conducted in triplicate, and the average values were calculated. The SG_{SD} of the CE used in this study was 0.98.

Developing desorption isotherm—Moisture desorption is a well-established method for assessing the relationship between pore volume and pore size.³¹ These desorption isotherms provide the equilibrium moisture content of the CE at different humidity levels (that is, the volume of pores of a specific size filled with water), which is useful

for understanding how water is retained or expelled from the pores during the mixing and curing stages. To develop the desorption isotherm, the CE was dried from an SD state to equilibrium at different relative humidity (RH) levels. For this, approximately 200 g of OD CE was first soaked in deionized water for 24 hours. The soaked samples were then centrifuged to remove excess water and placed in Petri dishes, where the initial mass was recorded. An analytical balance with a precision of ± 0.001 g (± 1 mg) was used to measure the mass of the samples in the Petri dishes. Approximately 50 g of centrifuged CE samples were placed in each Petri dish. Then, the samples were exposed to the following RH levels: 50%, 80%, 85%, 94%, and 98%. To create the RH chambers, specific salts were chosen based on the desired RH levels as listed in ASTM E104-20a.³² For the RH levels used in this study, high-RH salts such as potassium carbonate (K_2CO_3) for 50% RH, sodium bromide (NaBr) for 80% RH, potassium chloride (KCl) for 85% RH, potassium nitrate (KNO_3) for 94% RH, and potassium sulfate (K_2SO_4) for 98% RH were used. The selected salts were placed in noncorrosive, non-hygroscopic containers to a depth of approximately 1.5 cm. Water was added incrementally, with thorough stirring after each addition, until the solution was saturated, as indicated by the presence of undissolved salt and a small amount of free liquid. The containers were sealed and allowed to stabilize at the desired temperature (typically $23 \pm 0.1^\circ\text{C}$) for 1 hour to ensure equilibrium conditions. The CE samples in Petri dishes were then placed into these RH chambers, which were kept airtight to maintain the constant humidity environment. In addition, a fan was installed in the RH chamber to support active air circulation and facilitate rapid moisture exchange. The mass of the CE samples was monitored until equilibrium was reached, defined as a mass change of less than 0.01% per day. The OD CE samples were considered to be at 0% RH, and centrifuged CE samples were considered to be at 100% RH. After reaching equilibrium at each RH level, the samples were oven-dried to determine the final OD mass, M_{OD} . Then, the moisture content at each equilibrium RH level was calculated.

Isothermal calorimetry study of cementitious reactions of C3—Isothermal calorimetry was used^{31,33-35} to quantify the influence of water absorbed or desorbed from the cellulose. Water absorbed before setting would lead to a lower w/cm , and desorbed water after setting will internally cure the sample. This technique measures the heat released during cement hydration under sealed conditions with controlled temperature conditions. This approach enables the determination of the w/cm of the paste. The experimental approach used here is similar to that described in Castro et al.³⁶: 1) establishing a baseline relationship between pastes with various w/cm and heat release to understand how different heat values are associated with specific w/cm values; and 2) finding the heat release of a mixture of CE with varying moisture content to obtain the w/cm .

To establish a baseline for comparison and develop a relationship between heat release and w/cm , reference OPC cement paste samples (without CE) were prepared across a range of w/cm : 0.25, 0.26, 0.27, 0.28, 0.29, 0.30, 0.33, 0.35, 0.37, 0.40, 0.45, 0.50, and 0.60. For each w/cm , cement paste

was thoroughly mixed using a vacuum mixer at 250 rpm for 4 minutes. The paste (approximately 7 g) was then transferred into two individual glass ampules and immediately sealed to prevent moisture loss. An analytical balance with a precision of ± 0.0001 g (± 0.1 mg) was used to accurately weigh the samples in the ampules for all isothermal calorimetry measurements. These ampules were then lowered into the isothermal calorimeter, which was stabilized at $23 \pm 0.1^\circ\text{C}$. After time for baseline correction (~ 40 minutes), the heat values were recorded continuously for 7 days. The cumulative heat of hydration for each sample over the first 7 days (average of results from two samples) was used to construct a reference curve, relating cumulative heat release to w/cm .

Afterward, isothermal calorimetry was performed using CE + paste samples. The CE samples were preconditioned at various RH levels (0%, 50%, 80%, 85%, 94%, 98%, and 100%) from an SD state. For each RH condition, preconditioned CE was first placed in a glass ampule, and its mass was recorded. Then, cement paste with a w/cm of 0.33 was added to the ampule containing the CE to increase the total mass in the ampule to approximately 7 g. The mixture was then mixed in the ampule for 4 minutes. The final mass was recorded, and the samples were sealed and placed in the calorimeter. The recorded masses were used to normalize the heat results by weight of the binder. The heat measured after 7 days was compared to the plot from the paste samples to estimate the w/cm . It is to be noted that, while CE-containing samples may continue to hydrate beyond 7 days due to internal curing, the focus of this analysis is on early hydration. The 7-day cumulative heat release is an adequate indicator³⁶ of initial water availability and hydration behavior, which is essential for understanding the effective w/cm (w/cm^*) during the strength development phase of the C3 system.

RESULTS AND DISCUSSION

CE moisture state

Figures 3(a) and (b) show the θ (%) of the CE after different centrifuge testing durations for the bulk and cut CE, respectively. Figure 3(a) shows the θ (%) of the bulk (uncut) CE plotted as a function of centrifuge time and indicates a significant reduction in θ (%) within the first 10 minutes. After 15 minutes, the θ (%) values approach and stabilize around a constant value, $115 \pm 5\%$. The decrease in absorption rate over time highlights the efficacy of the centrifuge process in achieving a consistent saturated state of the CE, which is crucial for its application in composite materials. This moisture content is consistent with, though lower than, the pore volume of the CE, which would correspond to a maximum moisture content of approximately 156% (assuming 70% porosity and an SG of 0.98).³⁷⁻⁴⁰ This is due to the cellulose having very large pores (greater than 40 μm) where the water is not tightly bound and behaves like free water in concrete. As such, the experimentally observed value of $115 \pm 5\%$ after centrifugation corresponds well with the expected SD moisture state used in mixture proportioning.

Figure 3(b) shows the comparative analysis of water absorption for the 12.7 and 25.4 mm CE fibers. A statistical comparison of moisture content values at 15 to 60 minutes

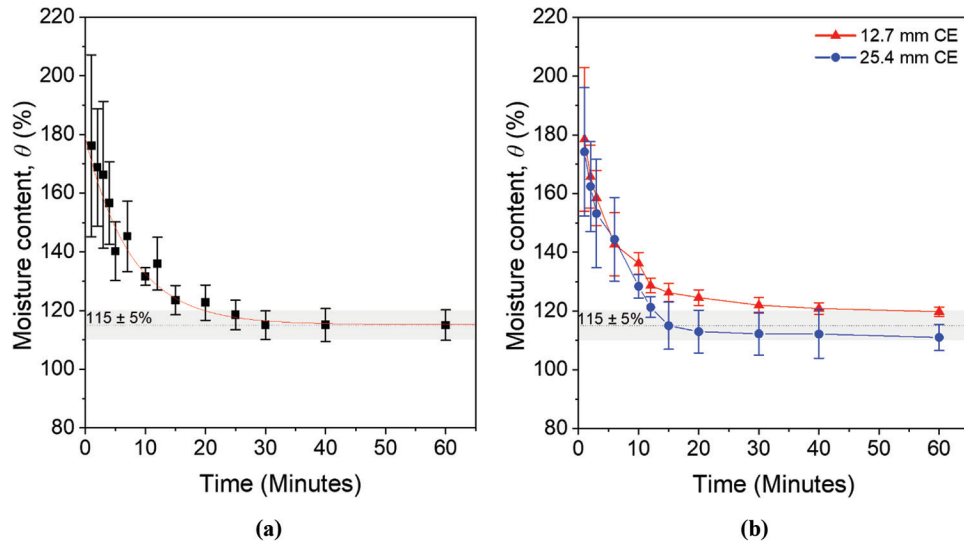


Fig. 3—Moisture content θ (%) versus time for: (a) uncut CE; and (b) 12.7 and 25.4 mm CE.

using Welch's t -tests revealed no significant differences ($p > 0.05$) between the 12.7 and 25.4 mm CE fibers, supporting the conclusion that fiber length does not significantly affect moisture retention during centrifugation. However, the 25.4 mm CE fibers showed greater variability, likely due to heterogeneous packing and orientation effects, which are more pronounced at longer strand lengths. The results indicated a negligible difference in the moisture retention capabilities of the two fiber lengths. The θ (%) of both cut fiber lengths appeared to be $\approx 115\%$, demonstrating that the fiber length does not significantly affect the maximum water retention of the CE.

CE desorption isotherm

Figure 4 shows the variation in the θ (%) of the CE samples over time at various RH. The θ (%) is expressed as the percentage of the mass of water in the CE to the mass of OD CE. Figure 4 indicates that the moisture content tends to reach equilibrium in relation to the surrounding humidity condition after approximately 200 hours. In this study, the CE was assumed to be in equilibrium if a change of less than 0.01% per day in the mass was observed. At higher RH levels, the moisture content curves for the CE samples plateaued closer to the original moisture content, suggesting a lower rate of moisture loss as the ambient RH increased. This trend was particularly pronounced at the highest tested RH conditions (94 and 98%), where the moisture content remained well above 90%, indicating CE's tendency to retain moisture at high ambient humidity conditions.

The Mualem-van Genuchten (MVG) model^{41,42} was used to obtain the desorption isotherm from the drying data presented in Fig. 4. As per the MVG model, the effective saturation (S_e) and capillary suction are expressed as shown in Eq. (3)

$$S_e = \left[\frac{1}{1 + (ah_c)^n} \right]^{(1 - \frac{1}{n})} \quad (3)$$

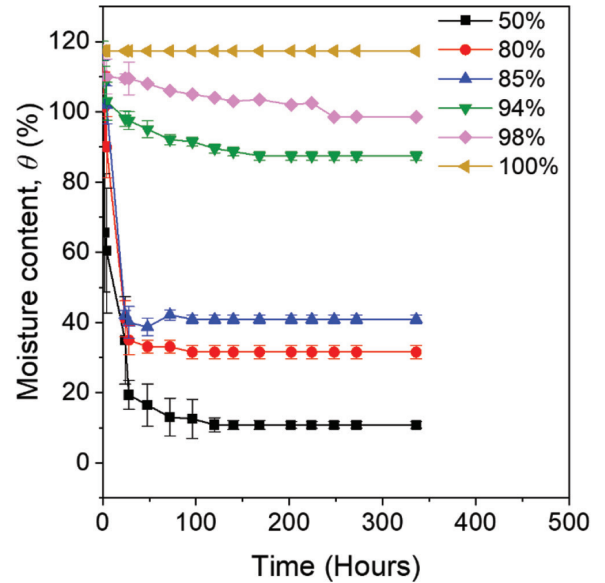


Fig. 4—Variation of moisture content of CE at various RH states with time.

where α and n are the dimensionless model fitting parameters; and h_c is the capillary suction head. This MVG model delineates effective saturation (S_e), as shown in Eq. (4)

$$S_e = \frac{\theta - \theta_r}{\theta_s - \theta_r} \quad (4)$$

where θ_r is residual moisture content; θ_s is saturated moisture content; and θ is the current moisture content. While oven drying at 105°C defines 0% moisture content gravimetrically, some amount of water may still be tightly bound within the nanopores of the CE.^{27,43,44} However, such residual moisture is typically excluded from desorption calculations and becomes undetectable in sorption isotherms when the moisture content is less than 5%.⁴³⁻⁴⁶ Therefore, the residual moisture content can be considered negligibly small, and θ_r can be assumed to be 0. Thus, Eq. (5) can be simplified as

$$S_e = \frac{\theta}{\theta_s} \quad (5)$$

The capillary suction head, h_c , can be expressed as a function of the matric suction head ψ_c as shown in Eq. (6)⁴⁷

$$h_c = \frac{\psi_c}{\rho g} \quad (6)$$

where ρ is the density of water (kg/m³); and g is the acceleration constant (m/s²).

The total suction, ψ , is the sum of the matric suction, ψ_c , and the osmotic suction, π . In scenarios where osmotic effects are negligible, which is often the case for pure water systems where solute concentrations are low, π can be assumed to be exceedingly small or zero, effectively eliminating osmotic pressure from the total suction.^{48,49} Thus, the total suction head (ψ) can be equated to the matric suction head (that is, $\psi \approx \psi_c$). Total suction is related to vapor pressure through the equilibrium thermodynamic relationship,⁴⁷ as shown in Eq. (7)

$$\psi = \frac{-RT}{gM_w} \times \ln\left(\frac{P_v}{P_0}\right) \quad (7)$$

where ψ is total liquid suction; R is the universal (molar) gas constant (J/mol·K); M_w is the molar mass of water (kg/mol); T is absolute temperature (K); P_0 is vapor pressure at saturation (Pa); and P_v is water vapor pressure (Pa). Hence, Eq. (2) can be rewritten as shown in Eq. (8)

$$\theta = \theta_s \times \left[\frac{1}{1 + \frac{\left(-\alpha RT \ln\left(\frac{P_v}{P_0} \right) \right)^n}{\rho g^2 M_w}} \right]^{\left(1 - \frac{1}{n} \right)} \quad (8)$$

where $(P_v/P_0) = (RH/100)$.

Figure 5 shows the desorption isotherm at 23°C (296.15 K), fitted from the drying test data in Fig. 4 using Eq. (8) along the 95% confidence band. The values of the fitted parameters α and n were obtained as 11.35 and 1.97, respectively.

Effect of CE moisture state on cementitious reactions of C3

As discussed earlier, the cementitious reactions in C3 are highly influenced by the moisture state of the CE. To better understand this relationship, a series of isothermal calorimetry studies was performed. Figure 6(a) illustrates the cumulative heat of hydration for neat OPC paste samples across a range of w/cm , from 0.25 to 0.60. This set of data is intended to serve as a reference for the hydration behavior of OPC, showing an increase in heat release with higher w/cm (due to self-desiccation). Subsequently, as depicted in Fig. 6(b), the calorimetry tests conducted on OPC paste ($w/cm = 0.33$) mixed with CE equilibrated at various levels of RH revealed how the pre-equilibrated moisture state of CE

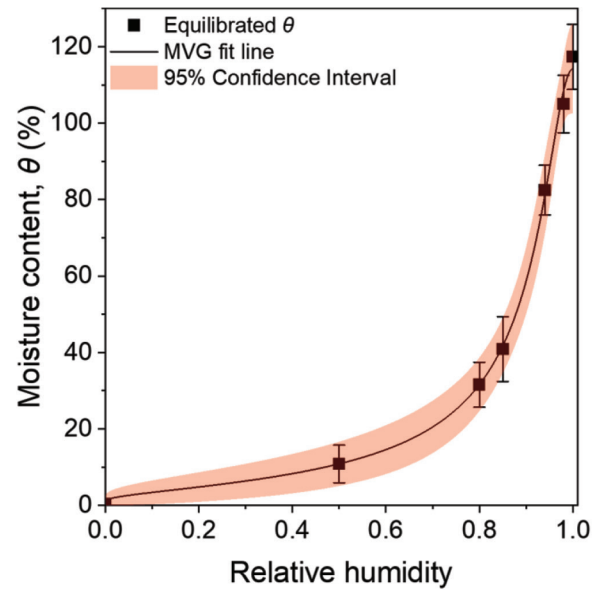


Fig. 5—Fitted desorption isotherm for CE at 23°C.

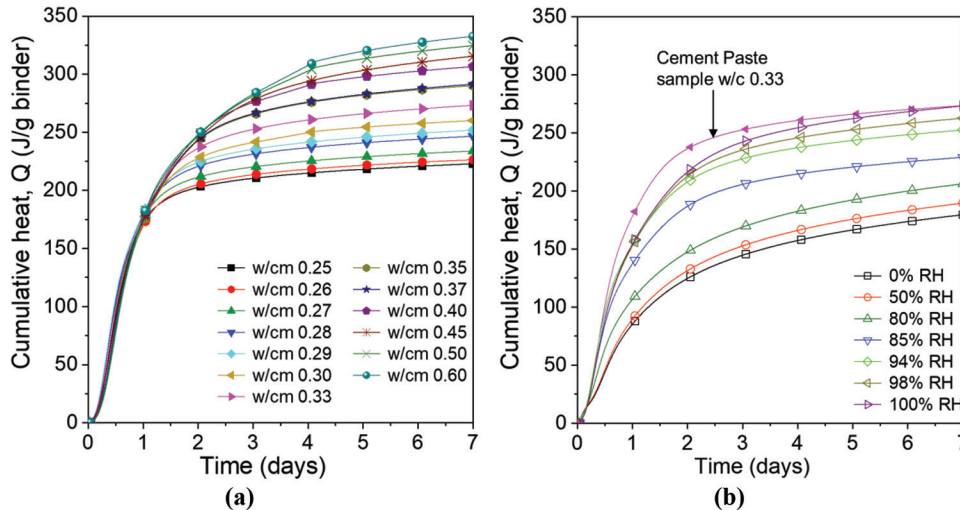


Fig. 6—Isothermal calorimetry results from: (a) paste samples; and (b) C3 samples ($w/cm = 0.33$).

Table 2—Calculation of mixture water that would be absorbed by CE and resulting w/cm^*

RH, %	w/cm	Material added to isothermal calorimetry ampule			CE_{OD} , g	θ , %	Water absorbed per g of CE_{OD}	Water absorbed by CE, g	Δw (g water/g cement)	w/cm^*
		Cement, g	Water, g	CE, g						
0	0.33	6.38	2.10	0.50	0.50	0.33	1.17	0.585	0.092	0.237
50	0.33	5.94	1.96	0.46	0.41	10.85	1.06	0.439	0.074	0.256
80	0.33	6.22	2.05	0.64	0.49	31.56	0.86	0.416	0.067	0.263
85	0.33	5.82	1.92	0.61	0.43	40.89	0.76	0.329	0.056	0.273
94	0.33	5.95	1.96	0.91	0.50	82.46	0.35	0.175	0.029	0.300
98	0.33	5.59	1.84	1.26	0.61	105	0.12	0.074	0.013	0.316
100	0.33	4.66	1.54	0.55	0.26	117.33	0.00	0.000	0.000	0.330

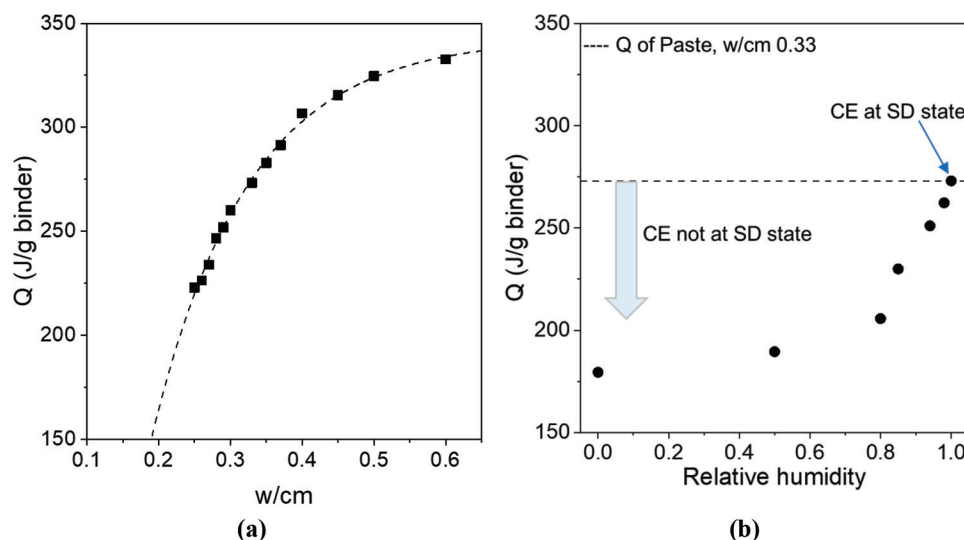


Fig. 7—(a) Cumulative heat release (Q) of OPC paste sample on seventh day versus w/cm ; and (b) maximum cumulative heat release by OPC + pre-equilibrated CE samples after 7 days.

affects the OPC hydration. As the RH in the preconditioned CE decreased, a corresponding decrease in heat evolution was observed, indicating a reduction in the amount of water available for cement hydration because the CE fibers absorb part of the added mixture water, effectively decreasing the water available for cementitious reactions. Samples pre-equilibrated to higher RH levels, notably those at 97 and 99.9% RH, showed higher heat evolution than the heat evolution of the samples with CE. This is consistent with having a higher w/cm . The results underscore the importance of considering the moisture condition of CE at various RH in the mixture design, as its capacity to retain water at high RH levels can influence the composite material's curing and strength gain.

Table 2 presents the calculated delta water (Δw), which represents the amount of water exchanged between the CE and the cement paste. It is determined by calculating the OD weight of the CE sample (CE_{OD}) using the equilibrated θ values derived from the desorption curve and subtracting the equilibrated θ at the specific RH from the equilibrated θ at 100% RH, and multiplying the result by the CE_{OD} . The w/cm^* can be calculated using Δw , as shown in Eq. (9)

$$w/cm^* = \left(\frac{w - \Delta w}{c} \right) \quad (9)$$

where w is the added mixture water; Δw is the water absorbed by the CE; and c is the mass of cement in the mixture. This calculated w/cm^* accounts for the water that could be absorbed or desorbed by the CE, affecting the hydration process. Table 2 shows that the w/cm^* of the C3 mixtures are lower than the intended w/cm (0.33) when the RH of the system is less than 100%. This demonstrates the need for additional water for cementitious reactions.

Figure 7(a) presents the relationship between the cumulative heat release for the OPC paste and the w/cm for 7 days. The data, a fitted exponential equation, show that the cumulative heat release increases as the w/cm rises. At lower w/cm , there is a lower heat release due to limited water available for reaction caused by self-desiccation. As the w/cm increases, the heat release rises, reflecting an increase in hydration products as more water is available to react with the cement particles. This baseline can help understand how CE addition may exchange water with the cement paste.

Figure 7(b) presents the maximum cumulative heat release by paste with a w/cm of 0.33 for a reference system (dashed line), as well as the paste mixed with CE that has been pre-equilibrated at various RH levels (circular dots). As the RH of the pre-equilibrated CE decreased, a reduction in the cumulative heat was observed. At the highest humidity level (approximately 98% accounting for ionic

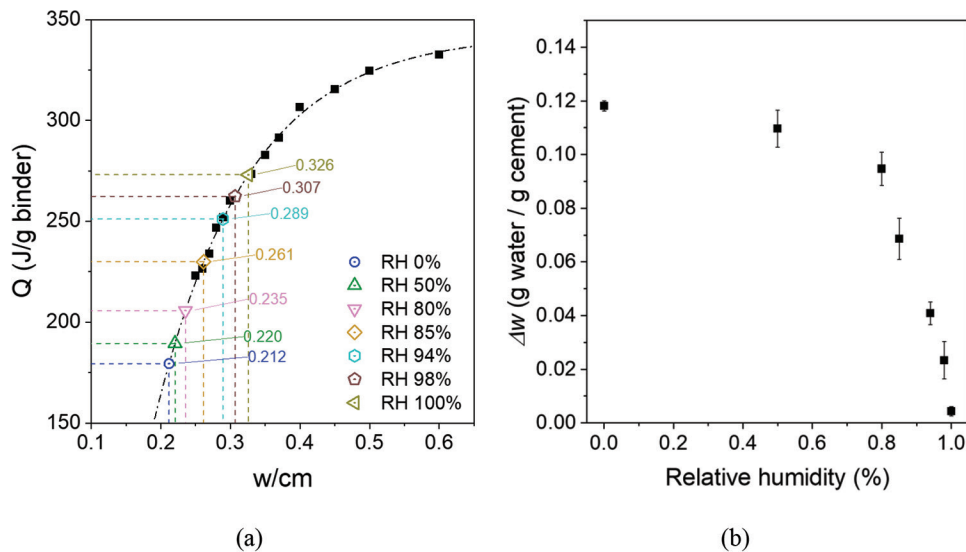


Fig. 8—(a) w/cm^* corresponding to heat release from C3 mixtures; and (b) amount of water absorbed by CE equilibrated at different RH. (Note: Mixtures shown contain 4 to 7.6% CE_{OD} by mass.)

species using Raoult's law³¹), the cumulative heat release approximately matched that of plain OPC paste, indicating that the CE was in an SD state and did not absorb water from the surrounding paste. This allowed hydration to proceed similarly to the plain OPC paste with 0.33 w/cm . At lower RH levels, however, the CE absorbed water from the cement paste, resulting in a reduction in water available for hydration and a corresponding reduction in heat release.

Because there is an exchange between CE water and mixed water, depending on the moisture state of the CE and the RH of the environment, there is a need to find a w/cm^* for the C3 that would provide the same degree of cementitious reactions as the neat OPC paste. Figure 8(a) represents the w/cm^* for C3 (OPC paste samples mixed with CE), which were pre-equilibrated to different RH levels. These w/cm^* values correspond to the cumulative heat released from the C3 mixtures after 7 days, mapped onto the baseline (Fig. 7(a)) established from the pure OPC samples. The w/cm values corresponding to the heat release of the cement and CE (equilibrated at various RH levels) mixtures are indicated on the curve. This approach provided a quantitative measure of how the absorbed and retained water within the CE contributed to the overall hydration process of the cement when the two materials were combined. It also offered insight into the adjusted w/cm necessary to achieve a specific hydration level when incorporating CE pre-equilibrated at various RH conditions. Notably, as the RH in the preconditioned CE increased, the w/cm^* also increased.

Figure 8(b) shows the relationship between RH and the amount of water absorbed by the CE, denoted as Δw (g water/g cement). At 100% RH, the amount of water absorbed by the CE was 0.008 g water/g cement for approximately 4% CE_{OD} content. Theoretically, this would be expected to be zero for the SD condition; however, this small, measured difference is likely due to water lost from the large pores where the capillary stresses holding the water in the pores are very low. If the RH of the CE was decreased, water from the mixture was absorbed by the CE. The water absorbed by the

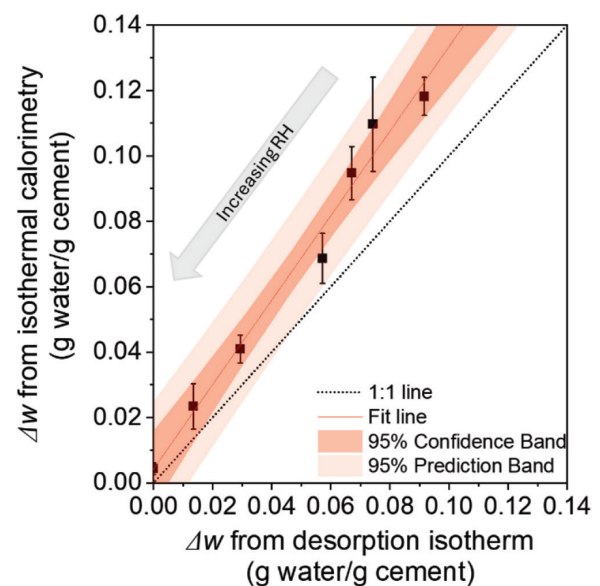


Fig. 9—Correlation between observed Δw of CE as determined from isothermal calorimetry and desorption isotherm.

CE ranged from 0.008 g at 100% RH to 0.08 g at 80% RH (assuming $6 \pm 1\%$ CE). At lower RH levels, the CE exhibits a higher Δw , indicating a substantial uptake of water from the mixture. As discussed earlier, the water absorbed in the CE could later serve as an internal reservoir, which would gradually release the water after setting to counteract the effects of self-desiccation associated with the cement hydration process, promoting internal curing.

Figure 9 compares the Δw from the calorimetry experiment (y-axis) with the Δw from the desorption isotherm (x-axis). Data points are fitted using a linear model, excluding error bars. The shaded area represents the 95% confidence interval, and the dashed lines indicate the 95% prediction limits. The strong linear correlation ($R^2 = 0.98$) displayed in Fig. 9 demonstrates a high degree of agreement between the CE desorption isotherm and the calorimetry, suggesting that the desorption isotherm can be used to predict water

absorption when the CE begins with an RH in the 98 to 100% range. The w/cm^* observed in isothermal calorimetry for CE less than the SD case was consistently lower than that predicted from the desorption data, with the difference between calorimetry and desorption ranging from approximately 1 to 10%. Specifically, the reductions observed at 100%, 98%, 94%, and 85% RH were 1.21%, 2.85%, 3.67%, and 4.40%, respectively. At lower RH levels (80, 50, and 0%), the average reduction increased to approximately 12%. This difference may arise from dynamic mixing effects during the calorimetry test, where water can access larger or reopened pores that are not accounted for in equilibrium desorption measurements.^{37,50-52} The desorption isotherm (Fig. 4) was obtained at equilibrium, an idealized condition assuming complete capillary-driven desorption, which may not occur in the time provided in the isothermal calorimetry, potentially overestimating water release from the CE under practical, non-equilibrium conditions. Thus, these considerations explain the offset while still supporting the use of desorption isotherms as predictive tools under near-saturated initial conditions (98 to 100% RH).

The fitted desorption curve can be used to calculate the Δw (g water/g cement) for any mixture proportions (that is, for differing w/cm or differing CE content). Figure 10 shows the predicted Δw as a function of RH for various CE_{OD} contents in the mixture. The predicted Δw is shown as lines, and the circular dot indicates the observed Δw from isothermal calorimetry. In the isothermal calorimetry test, the w/cm of the paste was 0.33; however, the percentage of CE added was not constant, that is, the mixture proportions varied in each isothermal calorimetry ampule (cement = $71 \pm 0.7\%$, water = $23.4 \pm 0.2\%$, and $CE_{OD} = 5.6 \pm 1\%$). Hence, Δw was calculated for mixture proportions with 4, 5, 6, 7, and 8% CE_{OD} to reflect this variability.

It can be observed that the Δw values from the isothermal calorimetry test closely follow the predicted desorption curves when matched with the actual CE content used. For instance, the obtained Δw at 85% RH corresponded to 5.3% CE_{OD} and fell just above the 6% curve. The Δw at 94% RH (5.9% CE_{OD}) aligned with the 8% curve, and the value at 98% RH (7.6% CE_{OD}) lay just above the 8% curve. This means that the desorption curve obtained could serve as useful data to predict the Δw in these systems. The isothermal calorimetry method, which relies on heat release as an indicator of hydration, may suggest that at lower RH, additional factors may need to be considered as the water absorption by CE may not be complete.

These findings have significant implications for the design of cementitious materials that incorporate CE, particularly when the CE is not in SD condition. Although the CE_{OD} content was relatively low by mass (typically 4 to 8% in this study), its low SG resulted in a disproportionately large volume fraction, often greater than 50% of the cement volume. This volumetric dominance of CE amplifies its impact on water absorption. Therefore, accounting for water absorbed by CE provides the potential for control over the properties of C3. The practical implication is that adjustments to the mixture design might be required to account for the water absorbed by CE, ensuring that the desired cement

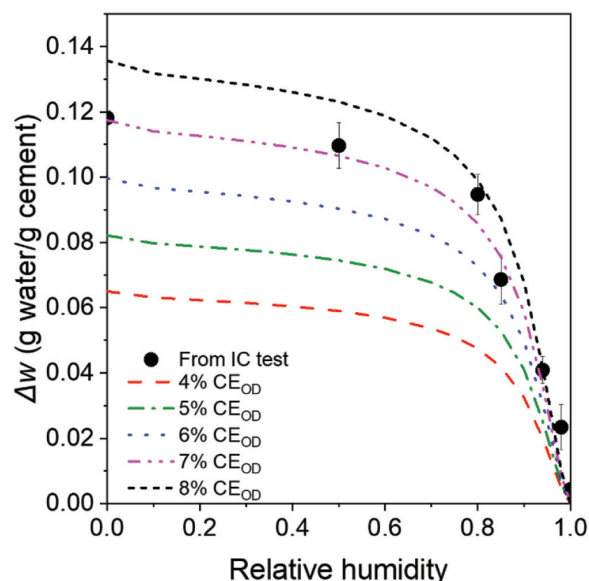


Fig. 10—Predicted Δw as function of RH for various CE contents in mixture.

hydration and concrete properties are achieved. This would be similar to adjustments made for aggregate absorption in a conventional concrete mixture design; however, the impact of absorption in CE would be much greater due to the reduced paste volume of C3 and the higher absorption of the CE (115% compared to 0 to 5% for most aggregates).

CONCLUSIONS

The development of cellulose-cement composite (C3) materials using cellulose excelsior (CE) offers potential for manufacturing a sustainable construction material alternative with a low carbon footprint. The production of C3 materials requires accurate methods to quantify the moisture in the CE. A methodology is presented to determine the surface-dry (SD) condition of the CE repeatably by using a centrifuge. The moisture content of CE at its SD state was found to be $115 \pm 5\%$. This approach can be used to provide consistency in C3 production by controlling water. The desorption isotherm was measured for CE and fitted using the Mualem-van Genuchten (MVG) model. This desorption can be used to quantify the water absorbed during the mixing process, especially if the relative humidity (RH) of the CE is greater than 80%. Isothermal calorimetry was used to confirm the validity of using the desorption model to quantify the water uptake by the CE from the mixture water. CE may provide internal curing water, which would be released gradually into the mixture, thereby enhancing the hydration process.

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Properties of Tunnel Fireproof Mortar: Effect of Alkali Equivalent

by Shuanghui Wen, Wei Zhao, Xiaowen Zhao, Yongning Liang, and Tao Ji

Desulfurization gypsum—alkali-activated slag-based tunnel fireproof mortar (GAM)—is a lightweight, cement-based fireproof material. A series of tests were conducted to investigate the effect of varying Na₂O equivalents (2, 4, 6, and 8%) on the wind and vibration resistance of GAM, along with its bond strength, and so on. Through X-ray diffraction (XRD), thermogravimetric-derivative thermogravimetric analysis (TG-DTG), scanning electronic microscope (SEM), and mercury intrusion porosimetry (MIP) analyses, the underlying mechanisms were elucidated. The study indicates that as the Na₂O equivalent increases, the weight loss of C-(A)-S-H gel and Aft phase exhibits an initial increase followed by a decrease. The weight loss of CaSO₄·2H₂O crystals, the crystal-to-gel ratio, and total porosity first decrease and then increase. Therefore, the fire resistance of GAM first decreases and then increases, while the dry density, compressive strength, bond strength, and wind and vibration resistance of GAM initially increase and subsequently decrease with increasing Na₂O equivalent. GAM with an Na₂O equivalent of 4% demonstrates optimal wind and vibration resistance.

Keywords: alkali-activated slag; desulfurization gypsum; Na₂O equivalent; tunnel fireproof mortar; wind and vibration resistance.

INTRODUCTION

In the case of a tunnel fire, serious consequences such as traffic disruptions, concrete structure damage, and potential loss of life and property can occur.¹⁻³ To avoid these risks, tunnel fireproof mortar (TFM) is often applied to enhance the fire resistance of tunnel linings.⁴ Ordinary portland cement (OPC) as binder is frequently used in fireproof materials due to its non-combustibility when exposed to heat.^{5,6} However, due to the decomposition of calcium hydroxide, TFM with OPC is prone to spalling at high temperatures, namely a rapid and brittle failure, which is caused by the severe deterioration of mechanical properties of TFM.^{7,8} Compared to OPC, alkali-activated slag (AAS) demonstrates superior high-temperature resistance⁹⁻¹¹ and stronger bond strength.^{12,13} However, AAS is also associated with higher autogenous and drying shrinkage.^{14,15} Desulfurization gypsum (DG) offers advantages such as light weight,¹⁶ good fire resistance,¹⁷ and low shrinkage.¹⁸ DG can compensate for the drawbacks of AAS. Therefore, a binder composed of DG and AAS has performances of high temperature resistance, high bond strength, and low shrinkage, which is expected to be suitable for TFM.^{19,20} Wind suction and vibration generated by running vehicles in tunnels can easily cause TFM spalling, posing a hazard to traffic safety and deteriorating the tunnel's fire protection function. Therefore, the study for wind and vibration resistance of TFM is very important.

Several studies²¹⁻²⁵ have explored the effect of Na₂O equivalent on the performance of alkali-activated cement (Appendix A). The effect of Na₂O equivalent on the performance of TFM has also been investigated. As the Na₂O equivalent increases from 0.7 to 17.5%, the cumulative pore volume, average pore diameter, and harmful pores of TFM decrease, and the interfacial transition zone of TFM becomes dense; therefore, the bond strength of TFM after wind and vibration resistance testing is enhanced²⁶ and the bond strengths of TFM before and after the freezing-and-thawing cycle increase.²⁷ However, the effect of Na₂O equivalent on the performance of desulfurization gypsum—alkali-activated slag-based tunnel fireproof mortar (GAM) and its wind and vibration resistance—is not fully understood.

This study aims to investigate the impact of Na₂O equivalent (2, 4, 6, and 8%) on various macroscopic properties, including compressive strength, bond strength, dry density, fire resistance, wind, and vibration resistance of GAM. The underlying mechanisms are explored by X-ray diffraction (XRD), thermogravimetric-derivative thermogravimetric analysis (TG-DTG), scanning electronic microscope (SEM), and mercury intrusion porosimetry (MIP), focusing on hydration phases and pore structure.

RESEARCH SIGNIFICANCE

As a fire barrier in tunnels, GAM plays a crucial role in mitigating serious consequences such as traffic disruptions and infrastructure damage. By exploring the impact of Na₂O equivalent on the properties of GAM and understanding its underlying mechanisms through in-depth analyses, this study aims to contribute to the broader application of GAM in tunnel projects. The expected outcomes include improved fire resistance, enhanced bond strength, and environmental friendliness of GAM. Ultimately, the findings from this research have the potential to advance the effectiveness and durability of GAM, ensuring the safety and resilience of tunnel infrastructures.

EXPERIMENTAL INVESTIGATION

Material

The binder used in this study includes DG and AAS. DG is composed of 94.6% CaSO₄·0.5H₂O, 2.8% CaSO₄·2H₂O,

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Table 1—Chemical compositions of DG and BFS, %

Precursor	CaO	SO ₃	SiO ₂	MgO	Al ₂ O ₃	P ₂ O ₅	Fe ₂ O ₃	K ₂ O	MnO	TiO ₂	Loss
DG	47.55	45.87	2.66	1.65	0.70	0.48	0.24	0.19	—	—	0.66
BFS	39.91	3.71	31.59	5.85	13.99	0.43	1.13	0.66	0.88	1.10	0.75

Note: “—” indicates not-measured items.

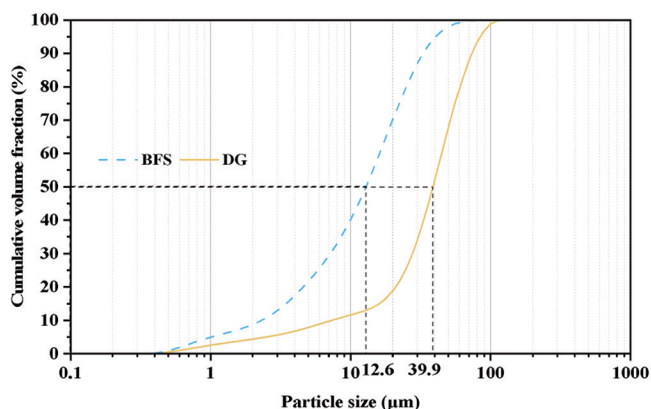


Fig. 1—Particle size distribution of DG and BFS.

and 2.6% CaSO₄. The specific surface area of DG is 2960 cm²/g and its average particle size is 39.9 μm. AAS is prepared by blast-furnace slag (BFS), and a combined alkaline activator which is composed of calcium hydroxide (CH) and sodium carbonate (SC). CH and SC are analytical pure reagents with a purity of more than 99%. The slag is S95 BFS powder, whose specific surface area is 4290 cm²/g and average particle size is 12.6 μm. The specific surface areas of DG and BFS were measured according to GB/T 8074-2008.²⁸ The chemical compositions of DG and BFS obtained with an X-ray fluorescence analyzer are shown in Table 1. The particle size distributions of DG and BFS are shown in Fig. 1. The XRD spectra of DG and BFS are shown in Fig. 2.

Thermal insulation materials include expanded vermiculite (EV), expanded perlite (EP), and hollow glass bead (HB). EV is a reddish-brown particle, and EP is a white particle, and HB is irregular spherical particle with a smooth surface. Aluminum hydroxide (AH) as a flame-retardant material effectively inhibits combustion after dehydration. Redispersible latex powder (RP) is an auxiliary adhesive, which can rapidly form a film-forming substance to enhance the cohesion of GAM.²⁹ Additives are used to improve the construction performance of TFM. The additives include gypsum retarder (GR), thixotropic agent (TA), cellulose ether (CE), and silicone hydrophobic powder (SP).

Mixture proportion

Tables 2 and 3 show the mixture proportion of desulfurization gypsum—alkali-activated slag-based tunnel fireproof paste (GAp) and GAM with different Na₂O equivalents, respectively. The definition of Na₂O equivalent is shown in Eq. (1). “GApN” and “GAMN” represent GAp and GAM with Na₂O equivalent (namely 2%, 4%, 6%, and 8%), respectively. For example, GAp2 and GAM2 represent GAp and GAM with Na₂O equivalent of 2%, respectively.

$$N = \frac{m_o}{m_{ar} + m_s} \quad (1)$$

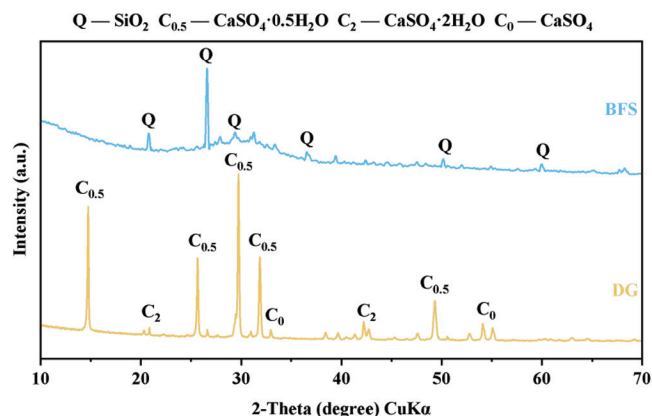


Fig. 2—XRD spectra of DG and BFS.

Sample preparation

To prepare GAp specimens, the components of DG, BFS, CH, and SC listed in Table 2 were accurately weighed and poured into a mortar mixer. The mixture was slowly stirred for 60 seconds, and deionized water (DW) was added. It was then slowly stirred for 60 seconds and rapidly for another 60 seconds until it became homogenous. The mixture was then poured into a mold and cured under the condition of temperature of 23 ± 2°C and relative humidity of 65 ± 5%. GAp specimens were broken into pieces 5 to 10 mm in size after curing for 28 days. These pieces were soaked in isopropyl alcohol for 7 days, cleaned with anhydrous ethanol, and then dried at 45°C for 48 hours.³⁰ The dried specimens were ground into powder with a particle size less than 45 μm. GAp specimens were used for XRD, TG-DTG, and MIP tests.

For the preparation of GAM specimens, the binder was poured into the mixer followed by the addition of the thermal insulation and flame-retardant materials, auxiliary adhesive, and additives. The mixture was slowly stirred for 120 seconds, and DW was added. It was then slowly stirred for 120 seconds and rapidly for 60 seconds until it became homogenous. GAM specimens were used for SEM, compressive strength, dry density, fire resistance, bond strength, and wind and vibration resistance tests.

Test method

The compressive strength, bond strength, dry density, fire resistance, and wind and vibration resistance of GAM were tested, and the SEM of GAM, and XRD, TG-DTG, and MIP of GAp were explored to reveal the underlying mechanism.

XRD—An X-ray diffractometer was used to detect the phase composition of GAp powder. The operating conditions included CuKα X-ray radiation, 2θ measurement range of 5 to 55 degrees, scanning step size of 0.01313 degrees,

Table 2—Mixture proportions of GAp, g

Specimen	DG	BFS	CH	SC	DW
GAp2	800	188.4	4.8	6.8	550
GAp4		176.8	9.5	13.7	
GAp6		165.2	14.3	20.5	
GAp8		153.5	19.1	27.4	

Table 3—Mixture proportions of GAM, g

Specimen	DG	BFS	CH	SC	RP	EV	EP	HB	AH	GR	TA	CE	SP	DW
GAM2	516	121.5	3.1	4.4	20	70	100	50	100	1	1	4	9	750
GAM4		114.0	6.2	8.8										
GAM6		106.5	9.2	13.3										
GAM8		99.1	12.3	17.6										

scanning steps of 3427, step time of 118.32 ms, and scanning speed of 0.01 deg/s.

The Rietveld method was used to perform quantitative XRD analysis on the GAp powder. The Rietveld method is a quantitative XRD analysis technique based on full-spectrum fitting, which lay in iterative optimizing and calculating the matching degree between the diffraction spectrum and experimental data through the least-square method.³¹ The scanning speed of the X-ray diffractometer was set to 2 deg/min in this test. α -Al₂O₃ (purity: 99.99%) was used as an internal standard, and α -Al₂O₃ and GAp were uniformly mixed at a mass ratio of 1:2.

TG-DTG—GAp powder placed in nitrogen was used to analyze hydration phases with a synchronous thermal analyzer. The rising temperature range of the analyzer was 30 to 1000°C with a heating rate of 10°C/min.

SEM—A tungsten-filament scanning electron microscope was used to analyze clean GAM specimens after the bond strength test. The process included impregnating the cut slice specimens with resin, grinding and polishing them using grinding equipment, and applying a gold-palladium coating to the surface of the specimen. These steps allowed for clear observation of the microstructure of hydration phases at 100× and 4000× magnifications. The working distance of the scanning electron microscope was maintained within a range of 10 to 16 mm, and the acceleration voltage was set to 15 kV.

MIP—The pore structure of GAp pieces of 5 to 10 mm in size was characterized by a mercury intrusion porosimeter with a pressure range of 0.17 to 413.69 MPa. The minimum pore diameter that could be measured was approximately 3.6 nm.

Compressive strength—Compressive strengths at the ages of 7, 14, and 28 days were tested using an automatic flexure and compression testing machine with a loading rate of 180 N/min in accordance with GB 14907-2018.³² The size of a specimen was 70.7 x 70.7 x 70.7 mm.

Dry density and fire resistance—The dry density test was conducted with reference to JT/T 1308-2020,³³ and the size of specimen used in this test was 70.7 x 70.7 x 70.7 mm. The fire resistance of GAM was tested according to the chamber combustion method after 28 days of curing.³⁴ GAM with a thickness of 20 ± 1 mm was placed on a fiber-reinforced slab with a size of 150 x 70 x 5 mm. The fire resistance of GAM

**Fig. 3—Tunnel fireproof mortar combustion test machine.**

was evaluated by conducting a 2-hour combustion test with a tunnel fireproof mortar combustion test machine (Fig. 3). The temperature on the bottom of the slab was monitored during the fire resistance test. A multichannel temperature tester was used to record the temperature change of GAM during the combustion test, and the interval was set to 1 minute.

Bond strength—According to JC/T 626-2008,³⁵ a fiber-reinforced slab with the size of 70.7 x 70.7 x 10 mm was prepared, and then a steel molding frame with an inner diameter of 40 x 40 mm, an outer diameter of 70 x 70 mm, and a thickness of 5 mm was placed on the slab. The fresh GAM was placed into the steel molding frame and flattened, and the bond specimen was obtained by removing the molding frame after the fresh GAM had been initially set, as shown in Fig. 4(a). The bond strength σ between GAM and fiber-reinforced slab at ages of 7, 14, and 28 days was tested with an intelligent bond strength meter (Fig. 4(b)) at a rate of 5 mm/min in accordance with JG/T 24-2018.³⁶ σ is calculated according to Eq. (2).

$$\sigma = \frac{P}{A} \quad (2)$$

Wind and vibration resistance—Wind suction and tunnel vibration are generated by vehicles or trains moving through

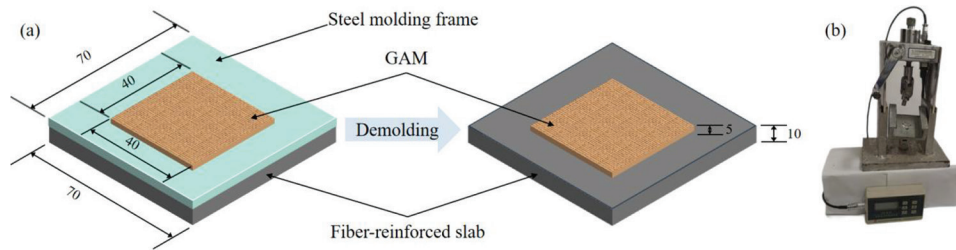


Fig. 4—Bond strength test: (a) preparation of bond specimen; and (b) bond strength meter. (Note: Units in mm.)

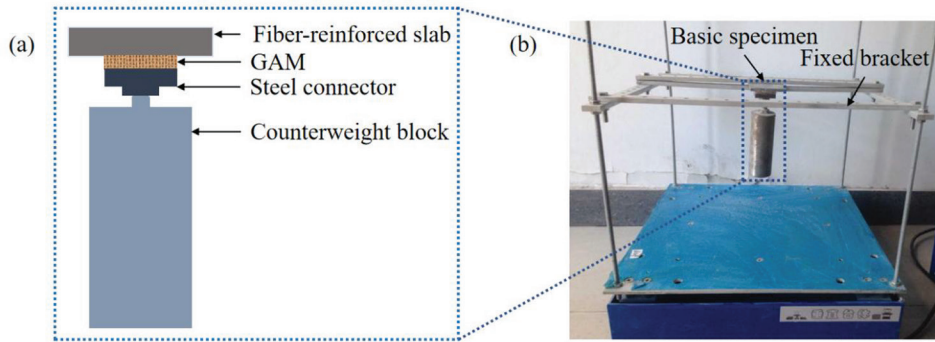


Fig. 5—Wind and vibration resistance test: (a) basic specimen; and (b) electromagnetic vertical shake table.

tunnels, and these effects have negative impacts on the bond properties of GAM.²⁶ There are primary microcracks in GAM, which will be expanded by wind suction and vibration and adversely affect the bond strength of GAM. To assess the ability of GAM to withstand these adverse effects, a wind and vibration resistance test was conducted according to T/CECS 901-2021,³⁷ and the real conditions about wind suction and vibration encountered in tunnels were simulated.

The basic specimen for the wind and vibration resistance test was assembled by fiber-reinforced slab, GAM, steel connector, and counterweight block, as shown in Fig. 5(a). The wind suction force and the weight of the counterweight block both act perpendicularly to the GAM (Fig. 5(a)). The wind suction force is dynamic relative to the GAM, while the counterweight block's gravitational force remains static. According to theoretical relationships established by other researchers, the wind suction force within the test area (that is—the base area of the GAM) is 0.9 N (equivalent to 91.8 g).²⁶ To simulate dynamic wind suction forces and accelerate experimental progress, the wind suction force applied to the GAM needs to be conservatively amplified by approximately 20 times (1840 g). Therefore, when the weight of steel connector is 112 ± 2 g, the counterweight block's weight should be 1728 ± 5 g. The basic specimen was then suspended under a fixed bracket on an electromagnetic vertical shake table (Fig. 5(b)), and tested with a vibration frequency of 50 Hz and amplitude of 3 mm for a duration of 30 minutes.

Immediately upon completion of the vibration, the basic specimen was removed from the fixed bracket and used for the bond strength test. σ included σ_b and σ_a , which were the bond strength of GAM before and after a 30-minute wind and vibration resistance test, respectively. The bond strength loss rate μ was calculated by Eq. (3).

$$\mu = \frac{\sigma_b - \sigma_a}{\sigma_a} \quad (3)$$

EXPERIMENTAL RESULTS AND DISCUSSION

Microscopic properties

Qualitative XRD—Figure 6 shows that $\text{CaSO}_4 \cdot 2\text{H}_2\text{O}$ crystal, ettringite (AFt) phase, calcium (aluminum) silicate hydrate (C-(A)-S-H) gel, and calcite are hydration products of GAP. The diffraction peaks of $\text{CaSO}_4 \cdot 2\text{H}_2\text{O}$ crystals and C-(A)-S-H gel overlap at $2\theta = 11.5, 20.5,$ and 29 degrees. Several AFt phases are detected at $2\theta = 9, 15.8, 22.8,$ and 35.8 degrees. As the Na_2O equivalent increases, rising concentration of OH^- ions in solution accelerates the depolymerization of tetrahedral $[\text{H}_3\text{SiO}_4]^-$, tetrahedral $[\text{H}_3\text{AlO}_4]^{2-}$, and octahedral $[\text{Al}(\text{OH})_6]^{3-}$ ions in BFS.³⁸ These ions combine with Ca^{2+} ions to promote the formation of C-(A)-S-H gel. C-(A)-S-H gel mainly exists in the form of amorphous gel with a dispersion peak because of its poor crystal morphology.³⁹ $[\text{H}_3\text{AlO}_4]^{2-}$ and $[\text{Al}(\text{OH})_6]^{3-}$ react with Ca^{2+} and SO_4^{2-} ions to form agglomerated AFt phases. A small amount of calcite is detected at $2\theta = 29.5$ degrees, which may be caused by carbonation during natural curing.²³

TG-DTG and quantitative XRD—Figure 7(a) shows that the TGA-DTG curves of GAP with different Na_2O equivalents after curing for 28 days. As shown in Fig. 7(b) and (c), the DTG curves are taken for local amplification in the temperature ranges of 30 to 200°C and 600 to 950°C, respectively. There are several distinct endothermic peaks of GAP. The first weight loss peak (60 to 110°C) is due to the evaporation of free water, dehydroxylation of C-(A)-S-H gel, and decomposition of the AFt phase.⁴⁰ Figure 7(b) shows that the GAP4 has the highest peak followed by GAP6, and then GAP2 and GAP8. The second weight loss peak appears at 110 to 200°C due to the dehydration of $\text{CaSO}_4 \cdot 2\text{H}_2\text{O}$ transferring into $\text{CaSO}_4 \cdot 0.5\text{H}_2\text{O}$.⁴¹ The decomposition peak between 600 and 700°C is caused by the decarburization of

the calcite.⁴² The existence of the calcite influences the filling and compaction of pores in GAP. The weight losses of the calcite increase with increasing Na₂O equivalent. The peak of approximately 700 to 900°C is caused by the conversion of C-(A)-S-H gel into β -wollastonite.⁴³

Table 4 shows that the total weight losses of hydration products (Δm) increase initially and subsequently decrease with the increase of Na₂O equivalent. When the Na₂O equivalent is 4%, Δm is the highest, indicating that the hydration degree of GAP4 is the largest. The weight losses of C-(A)-S-H gel and AFt phase at the temperature of 60 to 110°C (Δm_{CA}) also increase first and then decrease with increasing Na₂O equivalent. When the Na₂O equivalent is

4%, Δm_{CA} is the largest. However, with the increase of Na₂O equivalent, the weight loss of CaSO₄·2H₂O at the temperature of 110 to 200°C (Δm_{CS}) initially decreases and subsequently increases. When the Na₂O equivalent increases from 2 to 4%, the CaSO₄·2H₂O crystal is gradually consumed by calcium aluminum hydrate (C-A-H) gel and subsequently transformed into AFt phase.⁴⁴ At this stage, a small amount of C-(A)-S-H gel along with a significant quantity of AFt phase are produced, which is expected to enhance the compressive strength. However, as the Na₂O equivalent rises from 4 to 8%, the content of OH⁻ ions increases and the hydration reaction intensifies. The C-(A)-S-H gel is adsorbed onto the BFS particle surface, inhibiting the depolymerization of BFS,^{21,45} while the hydrolysis reaction of CaSO₄·0.5H₂O continues, leading to a reduction in the product of C-(A)-S-H gel and AFt phase, but an increase in the formation of CaSO₄·2H₂O crystal.⁴⁴ With increasing Na₂O equivalent, the weight loss of calcite at the temperature of 600 to 700°C (Δm_{CC}) increases because of the increasing content of the alkaline activator (CH + SC).

As shown in Fig. 7(b), the peaks of C-(A)-S-H gel and AFt crystal overlap partly in the range of 60 to 110°C. A Rietveld method is used to calculate the content of each phase in GAP shown in Table 5.⁴⁶ Table 6 shows that the crystal-to-gel ratio K obtained by Eq. (4) initially decreases and then increases as the Na₂O equivalent rises. When the Na₂O equivalent is 4%, K reaches a minimum of 7.93.

$$K = \frac{\sum w_c}{w_g} \quad (4)$$

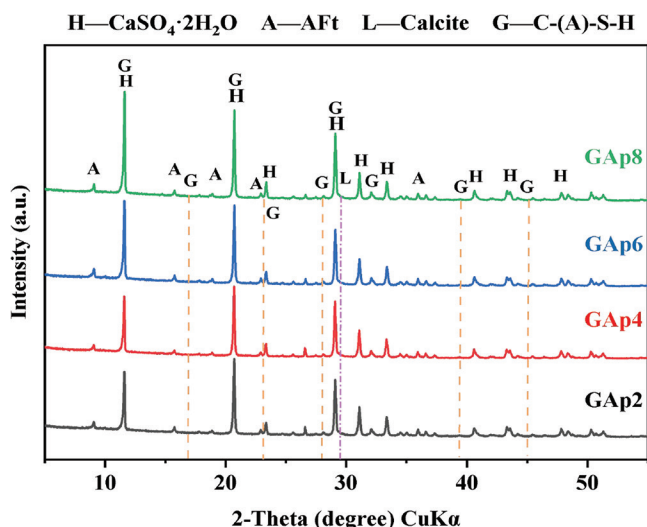


Fig. 6—XRD spectra of GAP.

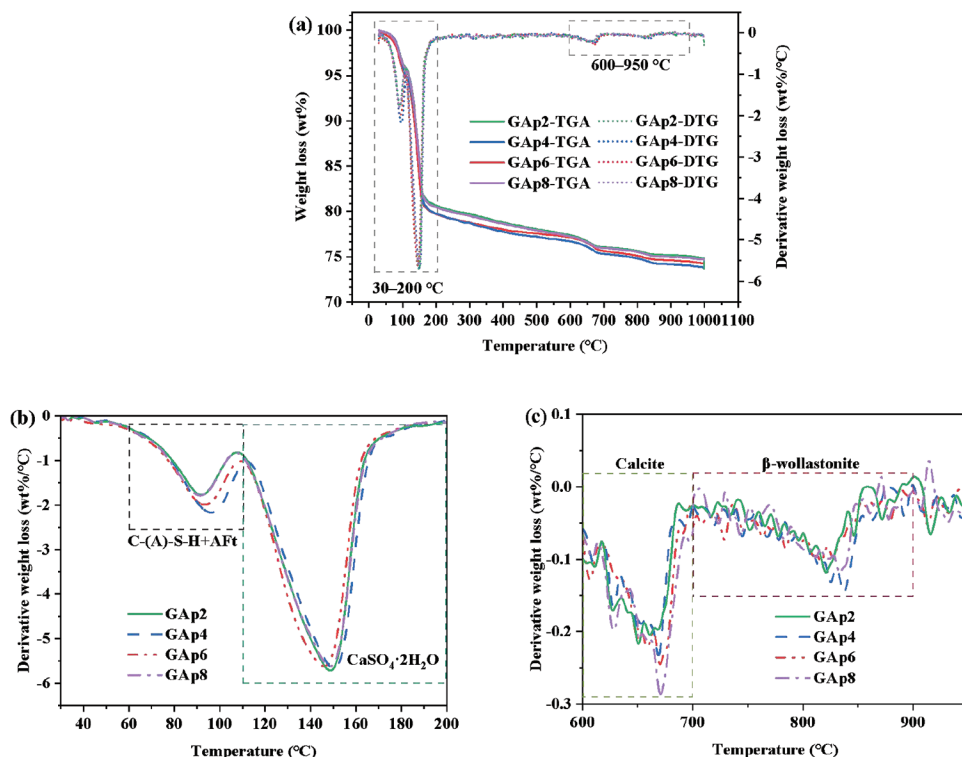


Fig. 7—TG-DTG curves and DTG amplification curves of GAP: (a) TG-DTG of 30 to 1000°C; (b) DTG of 30 to 200°C; and (c) DTG of 600 to 950°C.

Table 4—Weight losses of main phases in GAP, wt. %

Specimen	Δm_{CA}	Δm_{CS}	Δm_{CC}	Others	Δm
	60 to 110°C	110 to 200°C	600 to 700°C		
GAp2	3.75	15.28	1.46	4.47	25.16
GAp4	4.63	15.02	1.47	5.09	26.21
GAp6	4.47	15.17	1.58	4.55	25.77
GAp8	3.89	15.24	1.73	4.46	25.32

Table 5—Content of main phases in GAP obtained by XRD quantitative analysis, %

Specimen	CaSO ₄ ·2H ₂ O	C-(A)-S-H	AFt	Calcite	Others
GAp2	85.7	3.1	9.1	1.2	0.9
GAp4	75.4	11.2	11.0	1.4	1.0
GAp6	76.9	11.0	9.6	1.6	0.9
GAp8	78.3	9.2	9.4	2.2	0.9

Table 6—*K* calculated according to Tables 4 and 5

Specimen	$\Sigma w_c, \%$				$w_g, \%$	<i>K</i>
	CaSO ₄ ·2H ₂ O	AFt	Calcite	Others	C-(A)-S-H	
GAp2	21.56	2.29	0.30	0.23	0.78	31.26
GAp4	19.76	2.88	0.37	0.26	2.94	7.93
GAp6	19.82	2.47	0.41	0.23	2.83	8.09
GAp8	19.83	2.38	0.56	0.23	2.33	9.87

SEM—The microstructure of the bonding failure surfaces (100×) is shown in Fig. 8(a), (c), (e), and (g). As the Na₂O equivalent increases, the pores on the failure surface of GAM after the bond strength test become dense and then loose. When the Na₂O equivalent is 4%, the failure surface is the most compact with only a small number of pores, macroscopically showing the optimal bond strength. However, when the Na₂O equivalent is 6 and 8%, many pores appear on the failure surface (Fig. 8(e) and (g)), ultimately resulting in lower bond strength.

The microstructure of the hydration products (4000×) is shown in Fig. 8(b), (d), (f), and (h). When the Na₂O equivalent is 2%, the hydration products consist mainly of intersecting and long rod-shaped CaSO₄·2H₂O crystals. A small amount of flocculent C-(A)-S-H gel adheres to the surface of CaSO₄·2H₂O crystals, and some needle-shaped AFt phases intersperse into voids. The bond properties between needle-shaped AFt phases and expanded perlite are poor, resulting in the formation of loose structure.⁴⁷ Compared to 2% Na₂O equivalent, 4% Na₂O equivalent results in increasing contents of the flocculent and needle-shaped products in Fig. 8(d), leading to denser network structure and higher bond strength of GAM. However, when the Na₂O equivalent rises from 4 to 8%, the hydration reaction is accelerated and the honeycomb pores are generated (Fig. 8(e) and (g)), and the shrinkage of matrix increases, which leads to the transformation of CaSO₄·2H₂O crystals from short column to long rod and the decreasing formation of needle-shaped AFt phases (Fig. 8(f) and (h)). C-(A)-S-H gel is scattered between the skeleton structure composed of CaSO₄·2H₂O

crystal and AFt phase, and the size of CaSO₄·2H₂O crystal increases significantly, which loosens the microstructure.^{48,49}

MIP—The pore structure has a great influence on the macroscopic properties of GAM. Figures 9 and 10 show that the order of the cumulative pore fraction (total porosity) first decreases and then increases with the increase of Na₂O equivalent. When the Na₂O equivalent increases from 2 to 4%, increasing Na₂O equivalent promotes the depolymerization of BFS³⁸; the generated C-(A)-S-H gel and AFt phases fill the pores (Fig. 8(b) and (d)) and the total porosity decreases. However, when the Na₂O equivalent increases from 4 to 8%, the total porosity increases due to an increased amount of microcracks. When the Na₂O equivalent is 4%, the total porosity is the lowest.

The pores of GAP are divided into micropore (<50 nm), mesopore (50 to 100 nm), and macropore (>100 nm).⁵⁰⁻⁵² Figure 11 appears that the predominant pore of GAP is the macropore. When the Na₂O equivalent is 2%, the pore distribution of GAP is mainly concentrated at approximately 1000 nm, which is characterized by the loose structure and poor strength of hardened cement paste. As the Na₂O equivalent increases, the peak of the pore distribution moves toward the micropore—namely, the curves shift to the left in Fig. 11, indicating the increase of pore pressure and autogenous shrinkage of the matrix.⁵³ When the Na₂O equivalent is 4%, the macropore volume fraction (M_a) (Fig. 10) is the least while the micropore volume fraction is the largest, meaning the best pore structure, which is beneficial to the compressive strength and wind and vibration resistance.

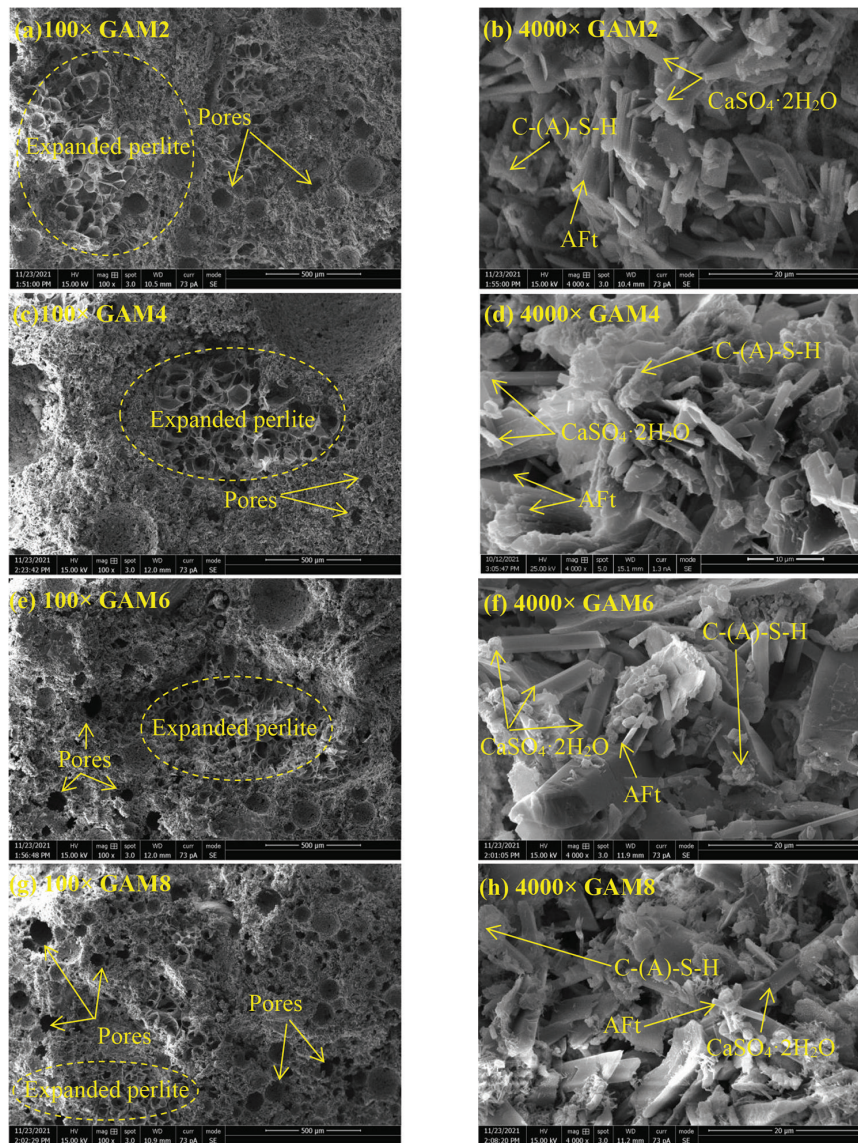


Fig. 8—SEM micrograph of GAM: (a) 100× GAM2; (b) 4000× GAM2; (c) 100× GAM4; (d) 4000× GAM4; (e) 100× GAM6; (f) 4000× GAM6; (g) 100× GAM8; and (h) 4000× GAM8.

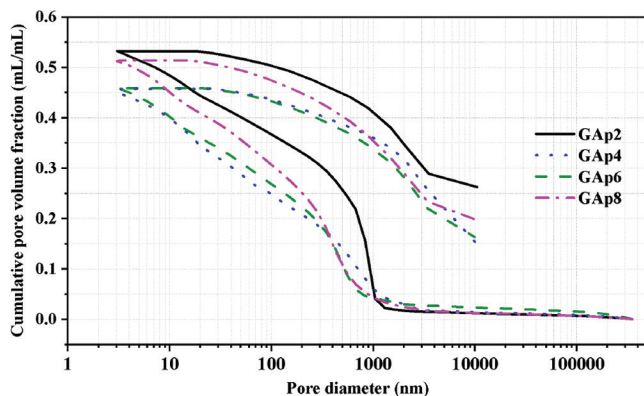


Fig. 9—Cumulative pore volume fraction of Gap.

Macroscopic properties

Compressive strength—Figure 12(a) shows the compressive strength of GAM. The compressive strength of GAM initially increases and then decreases as the Na_2O equivalent rises. When the Na_2O equivalent is 4%, the 28-day

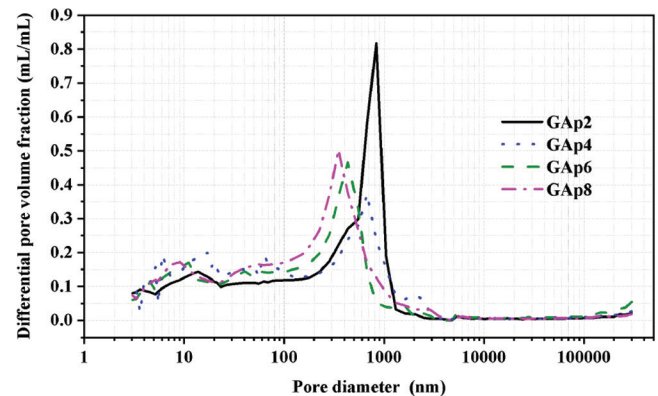


Fig. 10—Pore size classification of Gap.

compressive strength of GAM reaches the maximum value of 3.17 MPa. The relationship among Δm_{CA} (Table 4), total porosity (Fig. 10), and 28-day compressive strength of GAM is shown in Fig. 12(b). A lower Δm_{CA} and a larger total porosity lead to a lower compressive strength. With the increase of

Na_2O equivalent, Δm_{CA} first increases and then decreases, while total porosity first decreases and then increases, thereby giving rise to an initial increase and subsequent decrease for the compressive strength of GAM. When the Na_2O equivalent increases from 4 to 8%, the excessively high Na_2O equivalent inhibits the depolymerization of BFS, resulting in the reduction of C-(A)-S-H gel and AFt phase (Table 5) and an increase in total porosity, which leads to a decrease in the compressive strength of GAM. When the Na_2O equivalent is 4%, GAM has the highest compressive strength, as it has the largest Δm_{CA} and the lowest total porosity.

Dry density and fire resistance—Figure 13 shows the relationship among the dry density, Δm_{CS} (Table 4), total porosity (Fig. 10), and the back surface temperature of the fiber-reinforced slab of GAM. As depicted in Fig. 13, with the increase of Na_2O equivalent, both the dry density of GAM and the back surface temperature of the slab initially rise and then fall. Higher dry density and back surface temperature correspond to lower fire resistance. Therefore, the fire resistance of GAM initially decreases and then improves as the Na_2O equivalent increases. The GAM with an Na_2O equivalent of 4% demonstrates the highest dry density of 0.684 g/cm^3 and the highest back surface temperature of the slab, and therefore the lowest fire resistance of GAM.

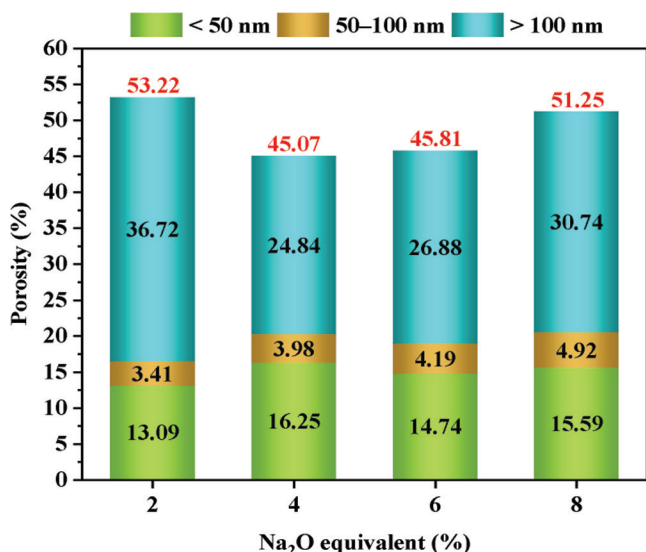


Fig. 11—Pore distribution of GAM.

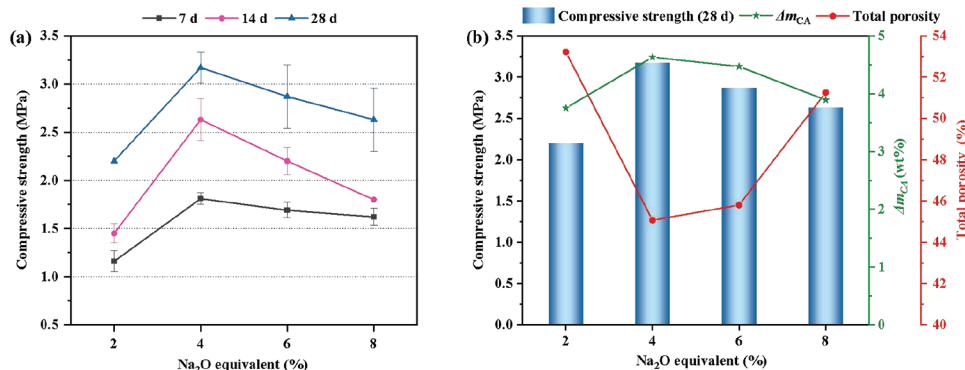


Fig. 12—(a) Compressive strength with increasing ages; and (b) relationship among Δm_{CA} , total porosity, and 28-day compressive strength of GAM.

The fire resistance of GAM correlates with Δm_{CS} and total porosity. By losing combined water and transforming into $\text{CaSO}_4 \cdot 0.5\text{H}_2\text{O}$ at elevated temperatures, the $\text{CaSO}_4 \cdot 2\text{H}_2\text{O}$ crystal contributes significantly to enhancing fire resistance of GAM⁴¹ (Table 4). Lower total porosity leads to higher thermal conductivity, thereby reducing fire resistance.⁵⁴ As the Na_2O equivalent increases, both Δm_{CS} and total porosity first decrease and subsequently increase, indicating that the fire resistance of GAM follows a similar trend (Fig. 13). GAM with an Na_2O equivalent of 4% has the lowest Δm_{CS} and total porosity, and therefore the poorest fire resistance (Fig. 13).

Bond strength—As shown in Fig. 14(a), the bond strengths of GAM at ages of 7, 14, and 28 days initially increase and then decrease as the Na_2O equivalent increases. When the Na_2O equivalent is 4%, the 28-day bond strength of GAM reaches the maximum value of 0.47 MPa. Figure 14(b) shows the relationship among C_g (Table 5), K (Table 6), total porosity (Fig. 10), and 28-day bond strength of GAM. A larger C_g leads to lower total porosity, resulting in greater bond strength. As the Na_2O equivalent rises, C_g initially increases and then decreases while K and total porosity decrease and then increase; therefore, the bond strength of GAM increases first and then decreases. C-(A)-S-H gel fills the internal pores and evenly distributes among AFt phase and $\text{CaSO}_4 \cdot 2\text{H}_2\text{O}$ crystal (Fig. 8(d)). GAM with an Na_2O equivalent of 4% has the largest C_g , the lowest K , and lowest total porosity, resulting in the densest internal structure of GAM. Thus, GAM with an Na_2O equivalent of 4% has the largest bond strength.

The failure mode of GAM can be classified as internal failure and bond interface failure. Internal failure occurs within GAM, indicating that the bond strength between the matrix and internal aggregate of GAM (σ_{ai}) is smaller than the bond strength between GAM and the fiber-reinforced slab (σ_{ab}). In contrast, bond interface failure occurs at the interface of GAM when σ_{ai} is larger than σ_{ab} . Figure 15 shows that GAM2 experiences bond interface failure, characterized by a smooth failure surface. In practical engineering applications of GAM, such damage of bond interface failure easily leads to the overall spalling of GAM, posing a significant threat to traffic safety in tunnels. The failure modes of GAM4, GAM6, and GAM8 are internal failures, with lightweight aggregate

visible on the fracture surfaces, which achieves higher wind and vibration resistance and is safer for vehicles in tunnels.

Wind and vibration resistance—The wind and vibration resistance of GAM is evaluated by parameters of σ_b , σ_a , and μ . Higher σ_b , σ_a , and lower μ result in higher wind and vibration resistance of GAM. σ_b is directly related to initial defects within GAM. σ_a is determined by both σ_b and μ (Eq. (3)). As depicted in Fig. 16, the wind and vibration resistance (σ_a) of GAM depends on M_a (Fig. 10) and C_g (Table 5). There are defects including macropores (quantified by M_a) and microcracks presented in the weak internal structure of GAM. Under cyclic wind and vibration loads, these defects gradually accumulate and propagate, eventually leading to the internal structural damage of GAM.⁵⁵ With increasing Na₂O equivalent, M_a initially decreases and then increases, which results in an initial increase and subsequent decrease in σ_b . A higher M_a indicates greater initial damage in GAM before wind and vibration resistance testing, which contributes to lower σ_b , thereby higher μ and lower σ_a . With increasing Na₂O equivalent, C_g rises first and then declines, leading to a corresponding increase and subsequent decrease in σ_b . Therefore, as the Na₂O equivalent increases, the wind and vibration resistance of GAM exhibits an initial increase followed by a decrease. When the Na₂O equivalent is 4%, σ_b and σ_a achieve the maximum values of 0.47 and 0.33 MPa, respectively, and μ is the smallest value of 29.8%, resulting in the highest wind and vibration resistance due to the lowest M_a and the highest C_g .

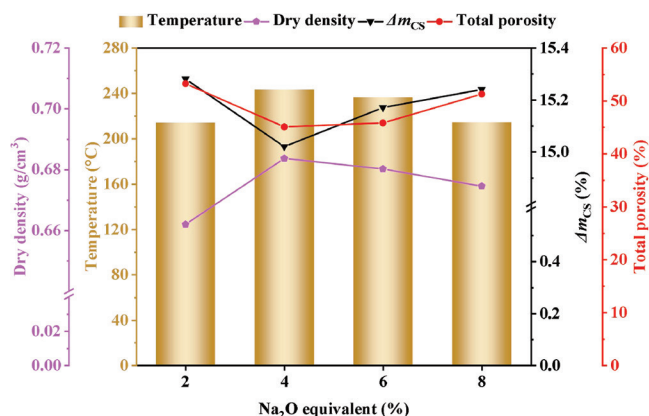


Fig. 13—Influences of dry density, Δm_{CS} , and total porosity on fire resistance of GAM.

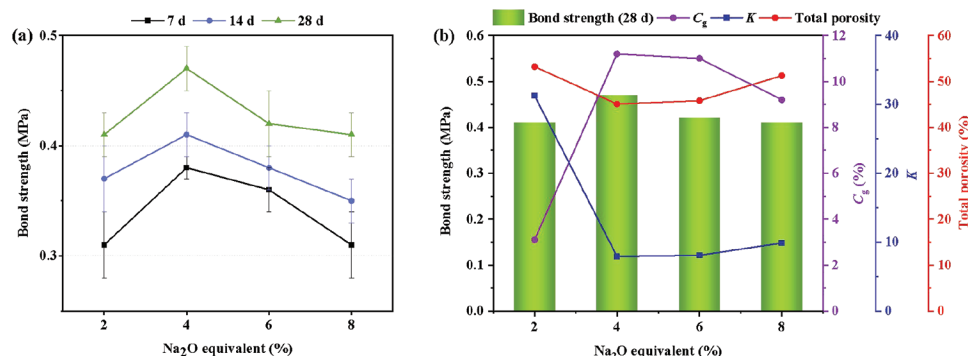


Fig. 14—(a) Bond strength with increasing ages; and (b) relationship among C_g , K , total porosity, and 28-day bond strength of GAM.

CONCLUSIONS

The influence of Na₂O equivalent on the microscopic and macroscopic properties of desulfurization gypsum—alkali-activated slag-based tunnel fireproof mortar (GAM) using calcium hydroxide (CH) and sodium carbonate (SC) as activators—was thoroughly examined in this paper. The following conclusions were drawn:

1. As the Na₂O equivalent increases, the weight losses of calcium (aluminum) silicate hydrate (C-(A)-S-H) gel and Aft phase (Δm_{CA}) initially increase and then decrease, while the total porosity initially decreases and then increases. Consequently, the compressive strength of GAM first increases and then decreases. GAM with an Na₂O equivalent of 4% has the highest Δm_{CA} and the lowest total porosity, resulting in optimal compressive strength.

2. With increasing Na₂O equivalent, the weight loss of CaSO₄·2H₂O crystals (Δm_{CS}) and total porosity first decrease and then increase, while the dry density initially increases and then decreases. As a result, the fire resistance of GAM initially decreases and then increases. GAM with an Na₂O equivalent of 4% demonstrates the lowest Δm_{CS} and lowest total porosity, but the highest dry density, yielding the poorest fire resistance.

3. With rising Na₂O equivalent, the content of C-(A)-S-H gel (C_g) first increases and then decreases, while the macropore volume fraction (M_a) initially decreases and then increases, therefore the wind and vibration resistance of GAM initially increases and then decreases. GAM with an Na₂O equivalent of 4% shows the highest C_g and the lowest M_a , achieving optimal wind and vibration resistance.

4. GAM with an Na₂O equivalent of 4% demonstrates adequate dry density and fire resistance, optimal compressive strength, and wind and vibration resistance, which is recommended for the application in tunnels.

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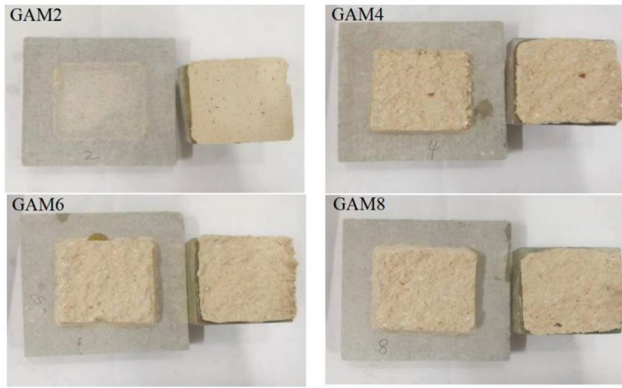


Fig. 15—Bond failure surface of GAM (28 days).

Tao Ji is a Professor in the College of Civil Engineering at Fuzhou University.

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NOTATION

A	=	bond area, mm ²
C_g	=	content of C-(A)-S-H gel, %
K	=	crystal-to-gel ratio
M_a	=	macropore volume fraction, %
m_{ar}	=	mass of activator, g
m_C	=	mass of C, g
m_o	=	mass of Na ₂ O, g
m_p	=	mass of precursor, g
m_{SC}	=	mass of SC, g
m_s	=	mass of BFS, g
N	=	Na ₂ O equivalent, %
N_0	=	mass ratio of Na ₂ O to Na ₂ O and BFS, %
N_1	=	mass ratio of Na ₂ O to precursor, %
N_2	=	mass ratio of activator to precursor, %
N_3	=	mass ratio of activator to activator and precursor, %
P	=	tensile load, N
w_g	=	$C_g \times \Delta m$, %
Δm	=	total weight losses of hydration products, wt. %
Δm_{CA}	=	weight losses of C-(A)-S-H gel and Aft phase at 60 to 110°C, wt. %
Δm_{CC}	=	weight loss of calcite at 600 to 700°C, wt. %
Δm_{CS}	=	weight loss of CaSO ₄ ·2H ₂ O at 110 to 200°C, wt. %
μ	=	bond strength loss rate, %
Σw_c	=	content of crystal (CaSO ₄ ·2H ₂ O + Aft + calcite + others) $\times \Delta m$, %
σ	=	bond strength between GAM and fiber-reinforced slab, MPa
σ_a	=	bond strength of GAM after 30-minute wind and vibration resistance test, MPa
σ_{ab}	=	bond strength between GAM and fiber-reinforced slab after 30-minute wind and vibration resistance test, MPa
σ_{ai}	=	bond strength between matrix and internal aggregate of GAM after 30-minute wind and vibration resistance test, MPa
σ_b	=	bond strength of GAM before wind and vibration resistance test, MPa

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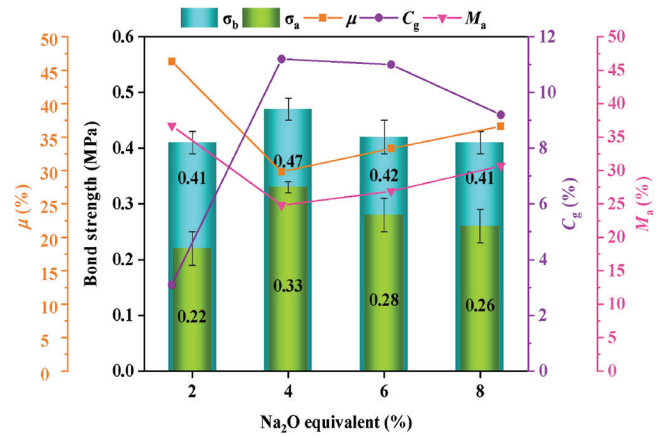


Fig. 16—Influences of C_g and M_a on wind and vibration resistance (σ_b , σ_a , and μ) of GAM.

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Appendix A—Effects of Na₂O equivalent on performance of alkali-activated cement

Researcher	Precursor	Activator	Variable	Performance
Choi and Lee ²¹	BFS	WG + SH	$N_0 = \frac{m_o}{m_o + m_p} = 4\%, 6\%, \text{ and } 8\%$	The flowability of AAS paste increases while the initial and final setting time decrease with increasing N_0 . When N_0 is 6%, the 24-hour compressive strength of AAS made from WG-activated BFS is similar to that at 8%, but much larger than that at 4% (<1 MPa), which is consistent with the result that the peak point of the calorimetric curves for the heat release rates of 6% and 8% during 0 to 12 hours is much higher than that of 4%.
Marvila et al. ²²	BFS	SH	$N_1 = \frac{m_o}{m_p} = 2.5\%, 5\%, 7.5\%, 10\%, 12.5\%, \text{ and } 15\%$	Both 7- and 28-day compressive strengths of AAS cured in the laboratory (approximately 25°C with relative humidity of 60%) increase first and then decrease with increasing N_1 , and reach the maximum value at 10%.
Wang et al. ²³	BFS	WG + SH	$N_1 = \frac{m_o}{m_p} = 4\%, 6\%, 8\%, \text{ and } 10\%$	When the WG modulus is 1.2, as the N_1 increases, the 28-day compressive strength of AAS first increases and then decreases, and achieves the highest value at 6%.
Langaro et al. ²⁴	ABFS and BBFS	SH	$N_2 = \frac{m_{ar}}{m_p} = 4\%, 5\%, \text{ and } 6\%$	As the N_2 increases, the initial and final setting time of AAS with ABFS and BBFS decrease. The maximum heat flow and 28-day compressive strength of AAS with ABFS initially increase and then decrease, and achieve the highest value at 5%. For the AAS with BBFS, the increasing N_2 is not able to apparently improve the compressive strength at any age; however, the maximum heat flow consistently increases with the increasing N_2 .
Zhao et al. ²⁵	G + BFS	OPC + C or OPC + SC	$N_3 = \frac{m_C \text{ or } m_{SC}}{m_{ar} + m_p} = 1.5\%, 3\%, \text{ and } 4.5\%$	When the ratio of OPC + C to GSC is 5 wt. %, as the N_3 increases, the initial and final setting time of GSC increase, while the 3-, 7-, and 28-day compressive strengths of GSC decrease.

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Portland-Limestone Cement Concrete Incorporated with Clay Pozzolana

by Mark Bediako and Timothy Kofi Ametefe

Portland-limestone cement (PLC) currently has gained widespread use as the most accessible and sustainable blended cement in the market. However, in many African countries, including Ghana, the use of clay pozzolana (CP) in the concrete industry has primarily relied on ordinary portland cement (OPC). In this study, PLC Type II/B-L was partially replaced with CP at levels ranging from 10 to 50% by weight. The investigation included compressive strength testing, nondestructive evaluations using electrical surface resistivity, pulse velocity, and chloride penetration tests, targeting a characteristic strength of 30 MPa (4351.13 psi). Additionally, an environmental impact assessment based on the carbon footprint of both control and CP concretes was conducted. The mixture design followed the EN 206 standard. A total of 72 cubic molds were produced for the strength test. The results showed that CP concretes with between 10 and 20% replacement achieved strength values of 35 and 33 MPa (5076.4 and 4786.32 psi), respectively, higher than the target of 30 MPa (4351.13 psi) strength at 28 days. However, mixtures with 30 to 50% replacement required extended curing periods of 60 to 90 days to reach the desired strength. At extended curing, 10 to 50% CP replacement attained strength between 32 and 41 MPa (4641.28 and 5946.64 psi). Nondestructive test results showed no direct correlation with compressive strength, confirming that different factors govern strength, resistivity, and pulse velocity. The environmental impact assessment revealed a 14 to 51% reduction in carbon strength index (CSI) and a 19 to 36% increase in carbon durability index (CRI) with 10 to 50% CP (for CSI) and 10 to 40% (for CRI). The thermodynamic modeling also revealed that pozzolana contents below 30% primarily promoted pozzolanic reactions, enhancing performance compared to the control mixture. Based on these results, 20 to 30% CP replacement is recommended to ensure reliable performance, while higher levels (>30%) require further durability evaluation for long-term use.

Keywords: carbon-chloride penetration index; carbon-strength index; clay pozzolana; environmental impact assessment; portland-limestone cement; thermodynamic modeling.

INTRODUCTION

Portland-limestone cement (PLC) is a type of blended cement that includes a proportion of finely ground limestone in its composition. The widespread use of PLC began in the early 1970s, during the oil crisis, when the French government urged the cement industry to adopt less energy-intensive materials to replace traditional portland cement.^{1,2} Concurrently, the production capacities for other known alternative materials, such as fly ash from coal-fired power plants and slag from steel manufacturing, were reduced. PLC is now used globally and standardized in European and American standards,^{3,4} permitting up to 15% limestone content in the United States and up to 35% in Europe. The introduction of

PLC as a blended cement is gaining widespread popularity, primarily due to its lower carbon footprint. Consequently, in many countries across the Global South, ordinary portland cement (OPC) is becoming increasingly rare in the construction industry.⁵ PLC aligns closely with the global sustainability agenda, as it can reduce the carbon footprint of concrete by approximately 15%⁶ and decrease the demand for primary raw materials for cement production by approximately 10%.⁷

Population growth and societal development have significantly increased the demand for construction materials, including portland cement and concrete.⁸ High population growth is expected to be concentrated in low-income countries, and Ghana, as a developing nation, is no exception. The demand for cement in Ghana is very high to meet the infrastructure needs associated with this growth. However, like many other West African countries, Ghana relies on imported clinker for cement production. The rising cost of clinker and a weak currency present substantial challenge for the cement industry in West Africa. In Ghana, for example, cement prices have increased by over 220% in the last decade. On the other hand, increasing cement consumption has dire consequences on the environment due to its associated carbon dioxide (CO₂) emissions. Reducing the clinker factor in cement with supplementary cementitious materials (SCMs) is an appropriate strategy to reduce the carbon emissions of the cement industry⁹⁻¹¹ and reduce the cost of cement. Before PLC was introduced in many West African countries, OPC was the dominant binder choice. Efforts to reduce cement costs in Ghana have included using SCMs such as pozzolana produced from bauxite waste and clay, blending these with OPC. Today, PLC dominates the cement market, yet there is a lack of data regarding the performance and environmental impact of PLC and clay pozzolana (CP) blends.

Several studies have reported that fine limestone powder in cement improves particle packing efficiency (filler effect) and enhances cement hydration, especially at early ages.¹²⁻¹⁵ The filler effect reduces water demand, improves workability, and reduces bleeding in concrete.^{14,16-20} During hydration, limestone powder participates in the cement hydration process by producing calcium carboaluminate hydrates.^{2,21-24} This hydrate is reported to fill spaces and

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contribute to the strength gain of the cement.^{25,26} Up to 20% and 35% of limestone are allowed in the production of CEM II/A-L and CEM II/B-L, respectively, in Europe.²⁷ In the United States, a maximum of 15% is allowed for cement production.²⁸ Some studies have also indicated that limestone powder addition above 30% introduces a dilution effect.¹² Dilution results in reduced concrete strength, especially at late ages.

In recent years, a family of cement known as limestone calcined clay cement (LC3) has emerged.²⁹ Several researchers have performed extensive research on LC3 and it has been shown to perform similarly to OPC.³⁰⁻³³ Up to 50% clinker replacement can be achieved using LC3, making it an efficient means to not only decarbonize the cement industry but also to produce economical cement.^{34,35} The widely known LC3 produced in many countries contains 50% clinker, 30% CP, 15% limestone, and 5% gypsum.³⁴ LC3 is based on two hypotheses: first, the development of carbo aluminate (hemi and mono) from the reaction of limestone powder and the C₃A component from cement as well as from the aluminate phases in the CP.^{36,37} The second hypothesis is the formation of a pozzolanic reaction between portlandite and the reactive aluminosilicate phases from the pozzolana, forming calcium aluminosilicate hydrates (C-A-S-H).^{38,39} In a cement hydration system, limestone improves the early-age strength whereas the CP also improves the late age strength of concrete. Several studies have shown that the synergy between calcined clay and limestone in a ternary blended system produces enhanced mechanical and durability properties of concrete than plain portland cement or portland pozzolana concretes.^{26,40,41}

Previous studies have indicated that CP enhances long-term strength and durability, refines pore structure, and contributes to the overall reduction of CO₂ emissions. However, data on the combined use of CP with PLC, especially regarding mechanical properties, durability, and environmental performance, is scarce. Although LC3 shows promise, its adoption in West Africa lags behind that of PLC. However, the synergistic relationship between limestone and CP in cement suggests that optimizing CP in PLC systems could yield benefits. Githachuri and Alexander¹² posited that the synergy between limestones and a reactive pozzolana offers new options for innovative construction materials from which further economic and environmental gains can be accrued. This study investigates the strength, durability, and environmental performance of incorporating CP in PLC. It is hypothesized that, while limestone addition in PLC improves early-age properties through pore filling, CP addition will further refine the pore structure, enhancing strength, durability, and reducing carbon footprint.

RESEARCH SIGNIFICANCE

This research explores the potential of combining PLC with CP to create a more sustainable and cost-effective cement blend for Ghana and similar developing regions. Given the high carbon footprint and costs associated with clinker, a primary component in cement, this study aims to reduce clinker dependence by substituting it with locally sourced CP. By partially replacing clinker, this blend could lower carbon

Table 1—Oxide composition of PLC and CP

Component	PLC	CP
SiO ₂ , %	22.1	64.9
Al ₂ O ₃ , %	5.2	25.3
Fe ₂ O ₃ , %	4.04	5.84
CaO, %	61.1	0.21
MgO, %	3.3	2.91
SO ₃ , %	2.5	0.56
Na ₂ O+K ₂ O, %	0.683	0.08

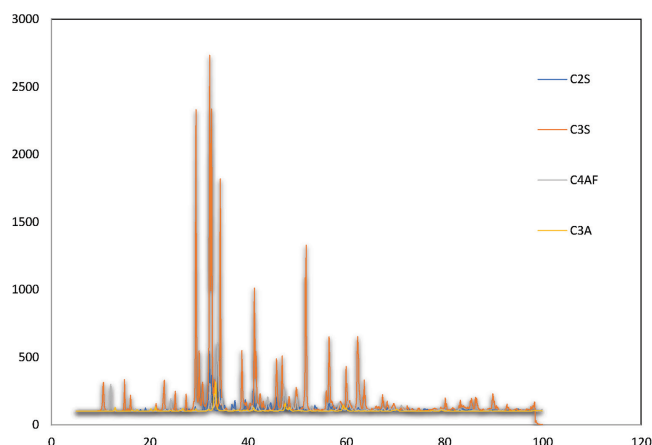


Fig. 1—XRD pattern of PLC.

emissions and production costs of concrete. It will further address economic and environmental challenges in regions with rapidly growing construction demands. Ultimately, the findings could shape regional guidelines for more sustainable construction practices that leverage locally available resources, offering environmental and economic benefits.

MATERIALS AND METHODS

Materials

The study used commercially available PLC conforming to the EN 197-1³ standard as CEM II/B-L 42.5R PLC. CP with 40% kaolinite content was obtained from the Ashanti region. Table 1 depicts the chemical composition of PLC and CP. Figures 1 and 2 show the mineralogical composition of the PLC as well as the raw clay and CP, respectively. The cement mineral in the PLC consisted of 10, 50, 11.7, and 3% of C₂S, C₃S, C₄AF, and C₃A, respectively. The calcite content was approximately 21.6%. In Fig. 2, the disappearance of kaolinite in the calcined clay about the raw clay shows a transformation from crystalline kaolinite into an amorphous phase. The PLC and CP had a specific gravity of 3.1 and 2.7, respectively. Granitic aggregate of nominal size 20 mm (0.79 in.) was sourced from the Agona area in the Ashanti region, and pit sand was obtained from the Juaben area in the same region. Specific gravities of the coarse and fine aggregates were 2.69 and 2.63, respectively. Figure 3 presents the particle-size distribution of the aggregates.

Methods

Concrete preparation and testing—The concrete mixture design was prepared following the EN 206⁴²

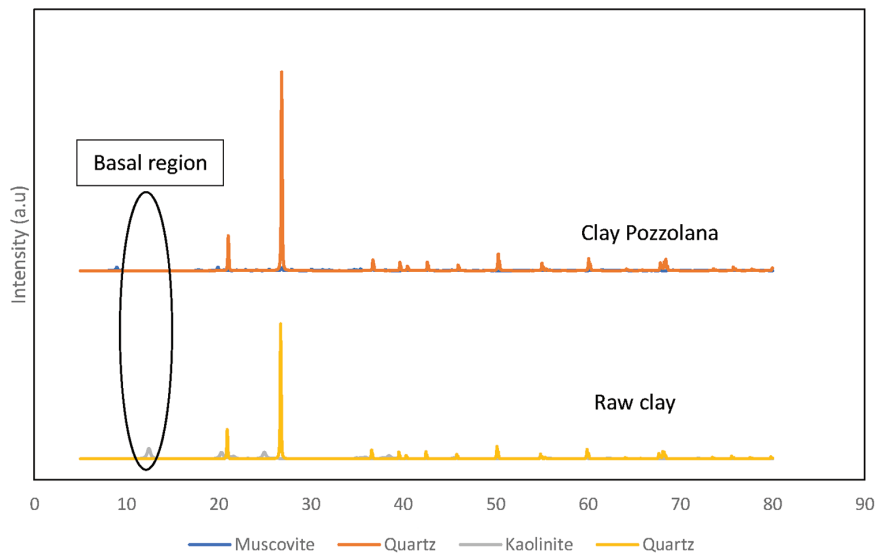


Fig. 2—XRD pattern of raw and calcined clay.

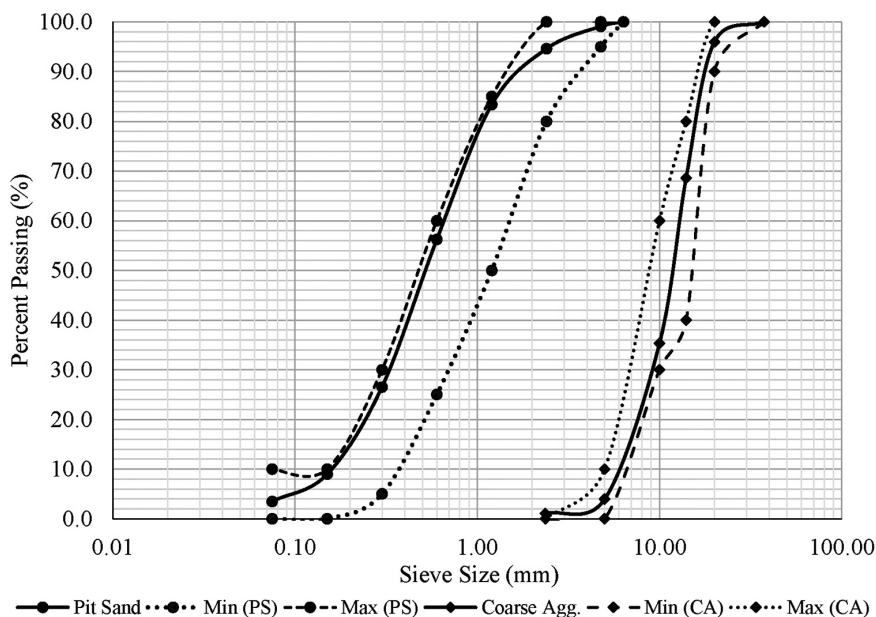


Fig. 3—Particle-size distribution of aggregates.

standard, designed with a characteristic strength of 30 MPa (4351.13 psi). CP was used to replace cement at 10, 20, 30, 40, and 50% by weight of the cement. Table 2 provides the detailed concrete mixture proportions. A polycarboxylate high-range water-reducing admixture (HRWRA) was used to achieve an S3 slump (100 to 150 mm [3.94 to 5.91 in.]).

Concrete production was carried out in a 0.05 m³ (1.77 ft³) capacity concrete mixing machine. The mixing process began with charging the mixer with coarse aggregate. Approximately 15% of the total water, mixed with the HRWRA, was gradually added to the aggregates. This was followed by the addition of fine aggregates and the binder. The initial mixing of these constituents lasted for approximately 90 seconds. The remaining water was then added, and the mixing continued for an additional 3 minutes.

After mixing, the slump was tested. The concrete was then placed into 100 mm (3.937 in.) cubic molds, compacted

on a vibrating table, and covered with polythene sheets with wet burlap on top. The molds were kept at room temperature for 24 hours before demolding. The demolded cubes were placed in a water bath maintained at $23 \pm 2^\circ\text{C}$ ($73.4 \pm 3.6^\circ\text{F}$) for continuous water curing until their respective test ages of 3, 7, 28, 60, and 90 days for compressive strength. Nondestructive testing (NDT) methods, including ultrasonic pulse velocity (UPV) and surface resistivity, were conducted by ASTM C597⁴³ and AASHTO T 358⁴⁴ standards, respectively, at 28, 60, and 90 days. The electrical resistivity equipment applies a uniform electric field using two electrodes that contact the bases of the saturated concrete specimen through sponges previously moistened with water. The ion transport mechanism through the concrete was monitored at the same intervals using the rapid chloride permeability test as prescribed by ASTM C1202.⁴⁵

Table 2—Concrete mixture design, per cubic meter

% replacement	PLC	CP	Fine aggregate	Coarse aggregate	Water	HRWRA	Slump, mm
0	385 (849)	0 (0)	628 (1385)	1167 (2573)	210 (463)	0 (0)	130
10	347 (765)	39 (86)	638 (1407)	1186 (2615)	179 (395)	2.2 (4.9)	120
20	308 (679)	77 (170)	638 (1407)	1186 (2615)	179 (395)	2.6 (5.7)	120
30	270 (595)	116 (256)	638 (1407)	1186 (2615)	179 (395)	3.1 (6.8)	100
40	231 (509)	154 (340)	638 (1407)	1186 (2615)	179 (395)	3.8 (8.4)	100
50	193 (426)	193 (426)	638 (1407)	1186 (2615)	179 (395)	4.6 (10.1)	105

Note: All units of PLC, CP, fine and coarse aggregates, water, and HRWRA are all in kg (lbf).

Environmental impact assessment—The common method used to quantify the environmental impact of concrete is the life cycle analysis (LCA), defined in the ISO 14040 and 14044.⁴⁶ It could be used to compute the environmental footprints in terms of the CO₂ emissions, global warming potentials, and many more. LCA consists of four basic phases that have been described by many authors including Dhandapani et al.⁴⁷ and Miller et al.⁴⁸ These include: 1) establishing the limit of the system; 2) analyzing the inventory; 3) evaluating the impact; and 4) interpreting and comparing the data. Either cradle-to-gate or cradle-to-cradle analysis is the emphasis of the system boundary. Cradle-to-gate boundaries were employed in this investigation. Cradle-to-gate carbon emission entails the total primary CO₂ emitted from direct and indirect processes associated with concrete within this defined boundary. They include material extraction, quarrying/mining, manufacturing, transportation, and all fabrication processes up to the point where the concrete is ready to leave the factory. This study excluded transportation of materials factors and emissions related to electricity and energy used in concrete production due to a lack of data in Ghana.

Inventory analysis involves using data from recognized sources such as the University of Bath, ecoinvent, the Environmental Policy Act (EPA), the Intergovernmental Panel on Climate Change (IPCC), and other secondary data from researchers; these databases provide information on the embodied energy and carbon of products. Embodied carbon refers to the total carbon emissions generated during the extraction and transportation of raw materials and their transformation into a final product.

CO₂ emissions, a key contributor to greenhouse gases (GHGs), were used as the primary indicator for the environmental impact assessment. The emissions data were calculated using conversion factors from various databases. Cement emissions were derived from the Institution of Civil Engineers (ICE) database,⁴⁹ while emissions for coarse and fine aggregates and water were sourced from the ecoinvent database.⁵⁰ Emissions for other admixtures, such as superplasticizers, were also considered. However, the estimation did not include CO₂ emissions related to the energy used in the cement production process due to a lack of available data. Such energy-related issues include on-site fuel and electricity consumption within the cement plant. Appendix A shows the emissions data and sources.

Finally, the interpretation phase involves comparing and analyzing the data collected. In this study, a carbon strength index (CSi) and carbon durability index (CRi) were used to assess the efficiency of the concrete in terms of its durability relative to its carbon emissions. The basis of these estimations was on a cubic meter of concrete. Equations (1) and (2) were used for the CSi and CRi estimations, respectively. The total embodied energy (EC) was calculated based on an equation provided by Bediako and Valentini.⁵³

The ratios provide a more comprehensive understanding of the material's environmental efficiency by balancing its structural performance against its environmental impact. This type of analysis helps in identifying opportunities for improving sustainability by optimizing concrete mixtures to reduce carbon emissions without compromising durability.

$$CSi = \frac{EC}{i\text{-day compressive strength}} \quad (1)$$

$$CRi = \frac{EC}{i\text{-day chloride penetration}} \quad (2)$$

Thermodynamic modeling—Thermodynamic simulations were conducted using the Gibbs Energy Minimization Software (GEMS) platform coupled with the Cemdata18 thermodynamic database.¹⁵ The modeling aimed to predict the phase assemblage and hydration product evolution in PLC-CP-blended systems at varying replacement levels.

The initial input compositions for each mixture were precisely defined based on the experimentally determined oxide compositions of PLC and CP (refer to Table 1). Thermodynamic simulations were conducted using the “single process calculation” module in GEMS to predict the equilibrium phase assemblage resulting from cement hydration. This approach enabled the estimation of stable hydration products and quantification of their relative amounts under controlled conditions, providing a detailed understanding of the microstructural evolution in the blended systems.

Phase equilibrium calculations were performed to estimate the formation and stability of major hydration products, including calcium silicate hydrate (C-S-H), C-A-S-H, portlandite, ettringite, monosulfate, hydrogarnet, and carboaluminate phases. The model results were compared with experimental observations to validate phase evolution trends and to provide mechanistic insights into the effects of high-volume CP on microstructural development.

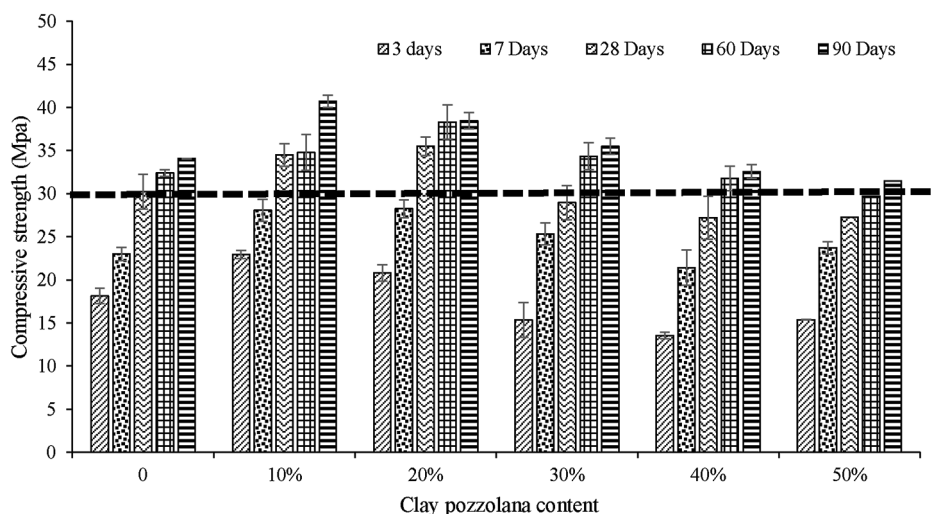


Fig. 4—Compressive strength of PLC concrete containing CP.

RESULTS AND DISCUSSIONS

Concrete properties

Compressive strength—Figure 4 illustrates the effect of replacing PLC with CP in concrete, targeting a characteristic strength of 30 MPa (4351.13 psi). At 3 and 7 days, concretes with 10% and 20% CP content exhibited strengths surpassing the control by 28% and 17%, respectively. By 7 days, concretes containing 10%, 20%, and 30% pozzolana achieved strengths that were 22%, 22%, and 9% higher than the control concrete. At 28 days, the concretes with 10% and 20% pozzolana content demonstrated strengths 13% and 20% greater than the control. The figure also shows that a characteristic strength of 30 MPa (4351.13) at 28 days was reached with up to a 30% reduction in PLC. Further, extended curing periods of 60 and 90 days resulted in superior strength even with a 30% reduction in PLC content. At 60 days, the 10%, 20%, and 30% pozzolana concretes exceeded the control concrete's strength by 9%, 19%, and 9%, respectively, while at 90 days, they surpassed the control by 21%, 12%, and 3%, respectively. The extended curing period also revealed that even with a 50% replacement of PLC with CP, the targeted characteristic strength of 30 MPa (4351.13) could still be achieved.

The early performance of the 10, 20, and 30% concrete mixtures at 3 and 7 days can be attributed to the increased hydration of alite (C_3S), which is influenced by the sulfate source in the cement. The influence of sulfate on hydration kinetics has been documented, indicating that sulfate modifies the surface charge of C-S-H, leading to enhanced hydration and stability of the microstructure.⁵⁴ The acceleration of the C_3S hydration caused by the right amount of calcium sulphate in the cement increases the initial mechanical strength.⁵⁵ Additionally, the presence of limestone in the cement contributes to the formation of stable AFt and AFm compounds. This same explanation could be used for the performance of the concretes at 28 days. The later-age performance of 10 to 30% CP concrete could be attributed to the refinement of the pore structure having space to accommodate hydrates AFm and C-A-S-H.

Ultrasonic pulse velocity—Figures 5 and 6 illustrate the UPV and normalized results of concrete mixtures containing

CP. As shown in Fig. 5, the pulse velocity values for all mixtures fall within the “very good” quality classification, according to BS EN 12504-4.⁵⁶ However, the replacement levels caused notable variations in the results. UPV values provide insight into the homogeneity of the concrete matrix, with higher pulse velocity indicating more homogeneous and better-quality concrete.

From the normalized results in Fig. 6, at 28 days, the 20% pozzolana concrete achieved the highest UPV, while the 30% mixture was similar to the control concrete. Both the 40% and 50% mixtures had lower UPV values than the control. At 60 days, the pulse velocity evolution closely mirrored that of the 28-day results. By 90 days, concretes with 20 to 40% CP showed higher UPV values than the control, while the 50% mixture recorded lower values.

A detailed examination of the pulse velocity evolution reveals that up to 40% pozzolana promotes matrix densification through pore refinement. This densification due to pozzolanic material addition is supported by the findings of Cheah et al.⁵⁷ The formation of cement hydration products, such as C-S-H, monocarboaluminate (Hc), and hemicarboaluminate (Hm), is well-known to enhance the concrete's density.⁵⁸ However, pozzolana levels above 40% may introduce voids into the concrete matrix. As the construction industry continues to seek sustainable and high-performance materials, these insights into the behavior of CP concrete mixtures will undoubtedly prove valuable in informing future research and practical applications.⁵⁹

Concrete resistivity—Figure 7 presents the electrical resistivity behavior of the control concrete and CP concrete mixtures. At 28 days, the concrete mixtures with 10%, 20%, and 30% CP replacement exhibited electrical resistivities that were approximately 20%, 67%, and 13% lower than the control, respectively. In contrast, the 40% and 50% CP replacement mixtures showed resistivities that were 13% and 10% higher, respectively, compared to the control concrete.

At 60 days, the electrical resistivity of the 20% CP concrete was approximately 25% lower than that of the control. However, the resistivities of the 10% and 30% pozzolana concretes were almost comparable to the control. For the 40% and 50% pozzolana concretes, resistivities were approximately

26% and 56% higher, respectively, compared to the control. By 90 days, the resistivity kinetics exhibited a progressive increase across all pozzolana mixtures, with values surpassing the control concrete. The CP concretes demonstrated resistivity increases ranging from 28 to 213%, with the 50% pozzolana concrete showing the highest resistivity.

Electrical resistivity measurement serves as an indirect method for assessing pore connectivity and pore size in concrete. Generally, electrical resistivity is inversely related to porosity. Performance data for concrete with 40 and 50% content of CP at 28 and 60 days show that a finer pore network

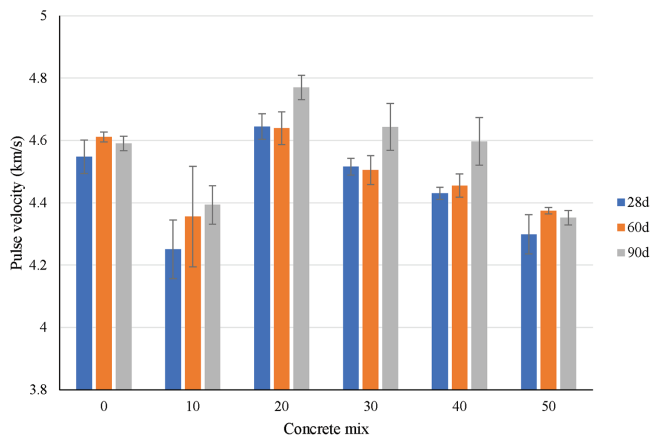


Fig. 5—UPV of concrete mixtures containing CP.

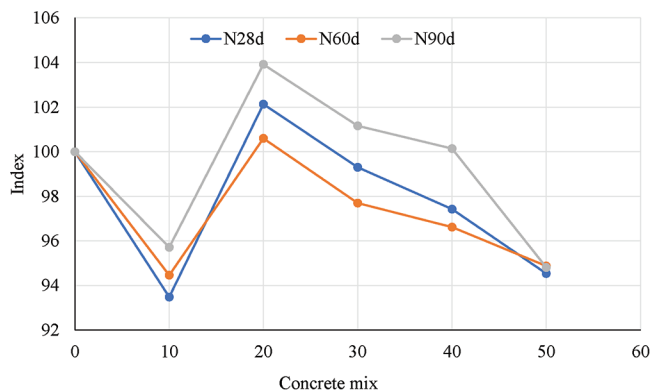


Fig. 6—Normalized UPV of concrete mixtures.

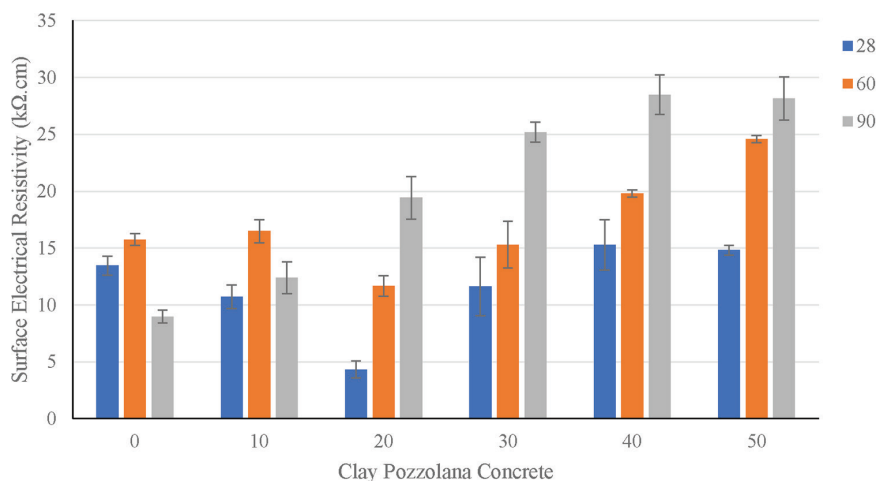


Fig. 7—Surface electrical resistivity of concrete containing CP.

and reduced permeability resulted in lower porosity. This reduction in porosity is attributed to the formation of hydration products that fill gaps, thereby making the concrete denser. This observation aligns with the findings of Layssi et al.⁶⁰ and Garcés-Vargas et al.⁶¹ Additionally, the performance of CP concrete at 90 days suggests that prolonged curing enhances gap-filling due to the formation of hydrates from the interaction between limestone powder and aluminate phase from the cement and the CP, as well as from pozzolanic reactions.⁶² This combination likely contributes to the observed decrease in internal porosity.

Environmental impact assessment—Figure 8 demonstrates the relationship between carbon emissions, compressive strength, and chloride penetration resistance in concrete. Figures 8(a), (b), and (c) illustrate a comprehensive view of environmental impact with concrete performance. These figures assess the carbon emission indices associated with the strength and durability of concrete. Appendix A shows the emission data of the components of the concrete.

Figure 8(a) presents the results of the carbon emission-CSI and carbon emission-CRi ratios at 28 days. The data shows that the CSI values for all CP concrete mixtures were between 19 and 36% lower than those of the control concrete. On the other hand, the CRi values were generally higher, ranging from 14 to 51%, except for the 50% clay pozzolan concrete mixture, which exhibited a CRi value approximately 13% lower than the control. Similar trends were observed in Fig. 8(b) and (c), which display the environmental impact at 60 and 90 days, respectively.

The findings suggest that, in terms of carbon emissions and compressive strength, incorporating CP reduces the environmental burden. However, when considering durability as measured by resistance to chloride penetration, the trend indicates a higher environmental burden. This highlights the distinct factors influencing strength and chloride penetration. While compressive strength is largely governed by the interfacial transition zone (ITZ) between the paste and the aggregates, chloride penetration resistance is more closely tied to the chemical composition of the pore solution in the concrete matrix.⁶³ These differing mechanisms underscore the complexity of balancing environmental sustainability with material performance in concrete formulations. In line

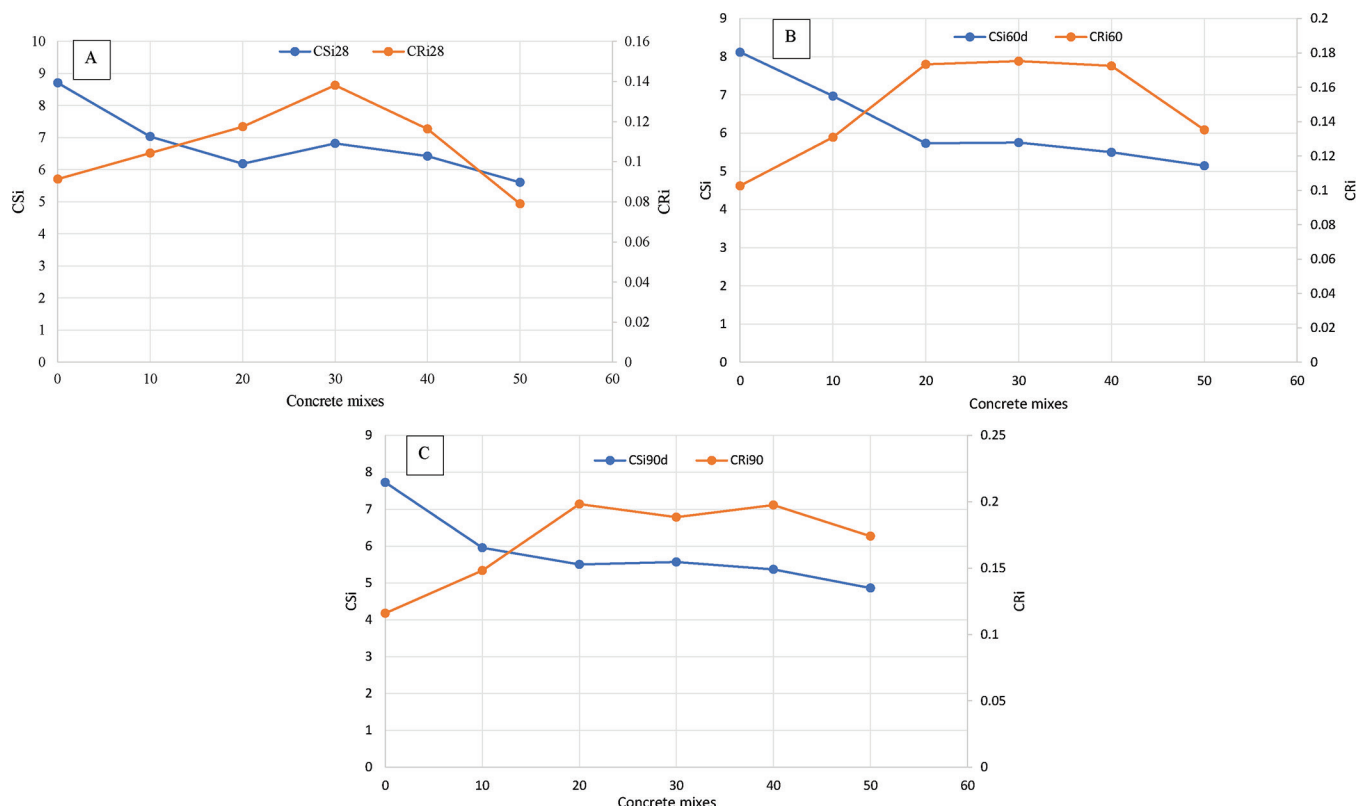


Fig. 8—Carbon emission-strength and carbon emissions-chloride penetration indexes of various concrete mixtures at: (a) 28 days; (b) 60 days; and (c) 90 days.

with this performance, it is clear that strength performance cannot be used as an indicator for chloride penetration.

Phase assemblage from thermodynamic modeling

Figure 9 illustrates the predicted phase assemblage evolution in PLC concrete as the proportion of CP increases from 0 to 50%, simulated using thermodynamic modeling (GEMS software with Cemdata database).

As shown, C-S-H remains the dominant hydration product across all replacement levels. However, its relative content gradually decreases with increasing CP content, reflecting the lower availability of clinker-derived calcium silicates as cement replacement increases.

The content of AFm phases (monosulfate and carboaluminate phases) also decreases progressively, while strätlingite diminishes sharply and nearly disappears beyond 20% CP replacement. This reduction indicates a shift in aluminum-bearing hydration products due to changes in alumina and sulfate availability when pozzolana replaces cement.^{26,64,65}

On the other hand, phases such as C_3AH_6 (hydrogarnet) and $C_3(AF)S_{0.84}H$ show a moderate increase with higher CP content. This increase can be attributed to the reactive alumina introduced by the calcined clay, which favors the formation of these hydrated aluminate phases.^{26,65-67}

Portlandite ($Ca(OH)_2$) content decreases significantly with higher CP incorporation. This is a direct result of the pozzolanic reaction, where portlandite is consumed to form additional C-S-H and C-A-S-H gels, leading to a denser microstructure and potentially enhanced durability.

The presence of $C_4Ac_{0.5}H_{12}$ (carboaluminate phases) remains relatively stable but slightly increases due to the synergistic interaction between clay-derived alumina and limestone filler in PLC, which promotes the stabilization of carboaluminates.^{26,64}

Aqua (representing free water) remains relatively constant, suggesting a similar degree of hydration water retention across mixtures.

Relating phase assemblage to mechanical and NDT performance—At lower replacement levels (10 to 20%), early-age strength gains are primarily attributed to the retained clinker reactivity, which ensures sufficient production of C-S-H from the hydration of C_3S and belite (C_2S). Additionally, the initial portlandite content remains adequate at these levels, facilitating partial early-stage pozzolanic reactions while also supporting matrix densification through nucleation effects. This synergistic mechanism explains the enhanced early compressive strengths that even surpass those of the control mixture.

The gradual formation of additional hydrates, notably C-A-S-H gels and C_3AH_6 , compensates for the initial strength lag, leading to substantial strength gains at later ages. This delayed but steady microstructural refinement underlies the eventual achievement of the design strength target even at high pozzolana contents. The NDT results further corroborate these thermodynamic predictions. The observed increase in UPV with curing time and pozzolana content reflects a continuous improvement in matrix homogeneity and internal density, consistent with the formation of additional hydration products predicted by the model. On the side of electrical resistivity, the significant rise in

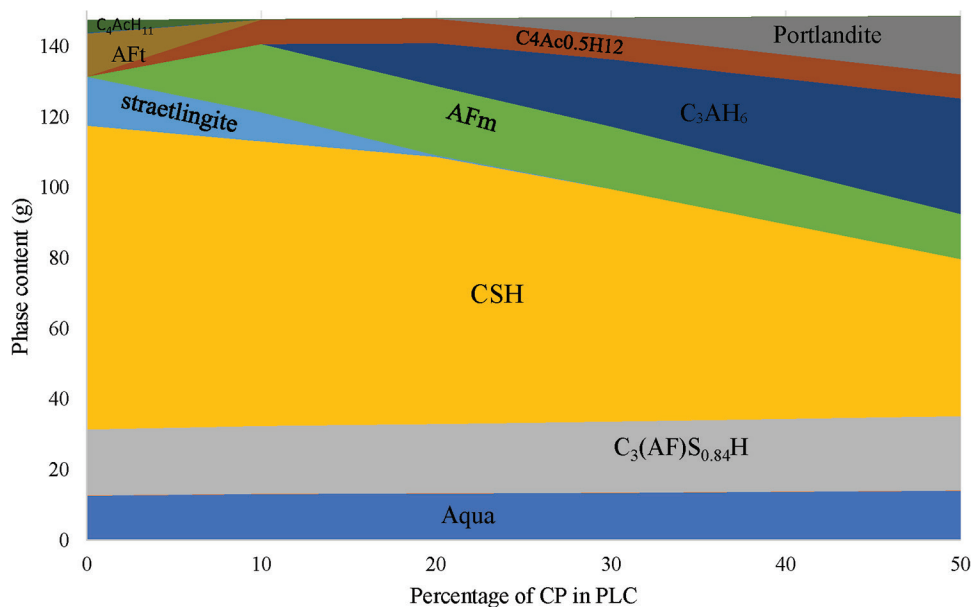


Fig. 9—Phase assemblage of PLC containing CP.

surface electrical resistivity indicates reduced ionic permeability and enhanced pore structure refinement, attributable to the extensive consumption of portlandite and pore-filling by secondary hydrates. The model-predicted reduction in portlandite and increase in alumina-bearing phases directly align with these durability indicators.

CONCLUSIONS AND RECOMMENDATIONS

Conclusions

The study explored the performance and environmental impact of incorporating clay pozzolana (CP) as partial replacement of portland-limestone cement (PLC) at 10 to 50% by weight. The performance characteristics were based on compressive strength, electrical surface resistivity, and ultrasonic pulse velocity as nondestructive testing mechanisms and environmental impact assessment which used two indexes, carbon strength (CSi) and carbon durability (CRi). From the evaluation of the strength, durability, and environmental impact analysis, these conclusions were made.

1. The incorporation of CP in PLC concrete between 10 and 50% not only meets but exceeds the targeted characteristic strength requirements, particularly with extended curing above 60 days. However, with traditional curing of 28 days, 20% CP content in PLC is most ideal.

2. Replacing PLC with CP up to 40% promotes matrix densification and improves the quality and homogeneity of concrete through pore refinement especially with extended curing periods. However, pozzolana levels above 40% may introduce voids into the concrete matrix, potentially reducing homogeneity and quality.

3. Lower levels of CP reduce surface resistivity initially, but higher levels enhance it over time. This trend differs from pulse velocity, suggesting different factors influence concrete behavior in these two nondestructive tests.

4. Different factors affect the environmental impact based on compressive strength and chloride penetration. While CP lowers carbon emissions for compressive strength, it may

increase the environmental burden by reducing resistance to chloride penetration.

5. At lower replacement levels (10 to 20%), the retained clinker content ensures strong early-age strength, while the gradual formation of secondary hydrates at higher pozzolana contents compensates for delayed strength gain. This synergy enables mixtures to eventually meet or exceed design strength targets even at higher pozzolana levels.

6. The progressive consumption of portlandite and the formation of additional hydrates (such as calcium aluminosilicate hydrate [C-A-S-H] gels and hydrogarnet) lead to a denser microstructure. This microstructural refinement improves durability indicators, as reflected by higher electrical resistivity and increased ultrasonic pulse velocity, indicating reduced permeability and enhanced internal compactness.

Recommendation

The study recommends conducting comprehensive investigations to establish correlations between strength performance, chloride penetration, and nondestructive testing methods. Such research could provide deeper insights into the underlying factors that influence concrete behavior, further improving our understanding of its performance characteristics.

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DECLARATION OF GENERATIVE AI AND AI-ASSISTED TECHNOLOGIES IN WRITING PROCESS

During the preparation of this work, the author(s) used ChatGPT to improve language and readability. After using this tool/service, the author(s) reviewed and edited the content as needed and take(s) full responsibility for the content of the publication.

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Appendix A

Material	Carbon equivalent, kgCO _{2eq} /kg	Reference
PLC (42.5R), CEM II/B-L	0.664	Hammond and Jones ⁴⁹
Coarse aggregate	0.00419	ecoinvent ⁵⁰
Sand	0.00419	ecoinvent ⁵⁰
HRWRA	0.767	Gursel and Horvath ⁵¹
Calcined clay pozzolan	0.07	Zhou et al. ⁵²
Water	0.0000658	ecoinvent ⁵⁰

Impact of Corrosion on Mechanical Properties of Thermomechanically Treated Steel Bars

by Sher Khan, Muhammad Masood Rafi, Humberto Varum, and Bruno Briseghella

Corrosion in reinforcing steel bars is a critical factor influencing the durability and structural performance of reinforced concrete structures. This paper investigates the effects of corrosion on the mechanical properties of thermomechanically treated steel bars. The study parameters included bar diameter, corrosion technique, and varying corrosion levels (CLs). The impressed current technique was used to accelerate corrosion. Load-displacement data from uniaxial tensile tests were analyzed to determine stress-strain relationships of corroded bars. The results showed that the mechanical properties of the bars were unaffected by diameter or corrosion technique. However, a consistent reduction in both nominal yield strength and ultimate strength was observed with increasing CLs, while the elastic modulus remained unchanged. The strength factors for yield strength and ultimate strength of the corroded bars varied in the range of 0.0013 to 0.015 and 0.0032 to 0.012, respectively, which were higher than reported in the literature. The fracture strain of the bars decreased at higher CLs. Predictive models were developed to estimate the residual mechanical properties, which are crucial for defining the constitutive relations needed to determine analytical stress-strain behavior. Analytical methods for determining these constitutive relations were also proposed, showing a good correlation with the experimental stress-strain curves.

Keywords: accelerated corrosion; corrosion level (CL); diameter loss (DL); residual strength; strain hardening; stress-strain curve; thermomechanically treated (TMT) bar.

INTRODUCTION

Corrosion of steel reinforcement is one of the most pervasive and costly threats to global infrastructure today, endangering the safety, durability, and serviceability of countless reinforced concrete (RC) structures. Despite over a century of reliance on RC in bridges, buildings, and critical lifelines, the degradation of embedded steel due to environmental exposure continues to cause premature failures, posing significant risks to public safety and resulting in billions of dollars in annual repair costs.¹

Once the alkaline environment surrounding steel reinforcement is compromised, oxidation initiates rapidly. This could happen due to various reasons, such as insufficient cover, construction defects, or exposure to aggressive agents such as chlorides or carbonation.² The resulting expansion cracks the concrete, weakens the bond, and reduces the steel's cross-sectional area, all of which can severely impair load-bearing capacity. This is particularly dangerous in aging infrastructure subjected to seismic or cyclic loading, where undetected corrosion can trigger brittle failure without warning.

While design codes and maintenance strategies have improved, current structural assessment practices often rely

on simplified assumptions, such as uniform section loss,³ which overlook key mechanical property changes. The urgent need for accurate, quantitative methods to evaluate the residual strength of corroded RC elements is more pressing than ever,⁴ particularly as climate-driven environmental aggressors intensify and infrastructure continues to age.

Although extensive research has addressed the electrochemical processes of corrosion and its impact on bond behavior,⁵⁻¹² far less attention has been paid to the mechanical degradation of the steel itself, particularly its residual yield strength (f_y), ductility, and stress-strain behavior.¹³ This knowledge gap is even more pronounced in the case of thermomechanically treated (TMT) reinforcing bars, which have become the preferred reinforcement in seismic regions due to their strength and ductility, but whose performance under corrosion remains underexplored.¹⁴

While a number of studies have examined corrosion effects on plain or low-carbon deformed bars,^{2,4,13,15-19} data on TMT bars are limited and fragmented.²⁰⁻²³ This is a critical oversight, considering the growing global reliance on TMT bars for resilient construction. Experimental evidence on how corrosion affects their mechanical properties (particularly under tensile loading) is essential for developing reliable predictive models.

In response to this urgent need, the present study investigates the tensile behavior of corroded TMT steel bars across various diameters and corrosion levels (CLs), using multiple exposure techniques to simulate realistic deterioration scenarios. Simplified predictive models and constitutive stress-strain relationships are derived to support seismic performance assessments and structural reliability evaluations of corroded RC elements.

Although this study focuses exclusively on tensile properties, it lays essential groundwork for broader residual capacity modeling. Long-term effects such as creep, Poisson's ratio, and multidimensional material behaviors are beyond its current scope. However, by isolating and quantifying the tensile degradation due to corrosion, this work directly addresses a critical data gap at a time when infrastructure resilience is both a national and global priority.

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RESEARCH SIGNIFICANCE

The determination of post-corrosion residual mechanical properties of TMT steel reinforcing bars is a relatively understudied area, with limited findings in the existing literature. Previous studies on the mechanical properties of other types of steel bars have yielded inconsistent and sometimes conflicting results. For example, Du et al.¹⁶ reported a 29% decrease in ultimate strength at a 10% CL, whereas Haris and Gadve²⁴ observed only a 13% decrease at a low CL. This study offers a comprehensive investigation of corroded TMT bars of various diameters exposed to different CLs ranging from 10 to 45%, employing both accelerated and simulated corrosion techniques to replicate realistic conditions. The chemical composition and microstructure of the TMT bars used are consistent with those reported by Rafi et al.,^{25,26} providing a solid basis for interpreting their corrosion behavior and mechanical response. The data obtained allowed for the reevaluation of current models predicting corrosion-induced strength loss, contributing valuable insights for the seismic assessment of corrosion-damaged RC structures. This study stands out for its novelty, as no previous published contributions to date (to the best of the authors' knowledge) have systematically investigated the mechanical properties of corroded reinforcing steel bars in Pakistan. The analytical models developed here improve predictions of residual mechanical properties, enhancing structural assessment accuracy and guiding the design of more resilient, corrosion-resistant RC structures.

EXPERIMENTAL PROGRAM

The experimental program included TMT bars of five different diameters (d_b): 12 mm (0.47 in.) (D12), 16 mm (0.63 in.) (D16), 19 mm (0.75 in.) (D19), 25 mm (1 in.) (D25), and 32 mm (1.26 in.) (D32). The bars were cut into 800 mm (32 in.) lengths each and were arranged in three series. While the bars in Series 1 and 2 were subjected to the accelerated corrosion technique, the simulated corrosion method was applied to the bars in Series 3.

The bars in Series 1 were machined according to ASTM E8/E8M-15a²⁷ within the gauge length of $4d_b$ before being exposed to artificial corrosion. This machining ensures that the bar fracture occurs within the specified zone during the tension testing.²⁶ The nomenclature MC was used for these types of bars. The bars of all the aforementioned diameters were used in Series 1 and 3 specimens and were tested to obtain an average of bar characteristics. Control specimens of uncorroded machined reinforcing bars were also tested to compare their properties with those of corroded reinforcing bars. The study examined a total of 50 reinforcing bar specimens.

D16 bars without machining were included in Series 2 and are designated by the nomenclature UM. This testing was conducted to compare the bar tensile behavior with bars in Series 3, which underwent simulated corrosion through machining to achieve a reduced area corresponding to a specific level of corrosion of the bars in Series 2. The bars in Series 3 are designated as UCM bars. Three specimens were tested at each CL to assess variability and ensure data consistency for the D16 bars in Series 1. Based on the consistent

results obtained from these triplicate tests, the number of specimens used to determine average values was reduced to two for all other bar diameters in Series 1, as well as for specimens in Series 2 and 3.

Instead of defining fixed CLs (or mass loss), diameter loss (DL) was used as an indicator of uniform corrosion in this study. The corresponding CLs were determined using Eq. (1)⁴

$$d_f = d_i \sqrt{1 - CL/100} \quad (1)$$

where d_i and d_f are the diameters of the uncorroded and corroded bars, respectively.

The approach of controlling DL allows for a more precise evaluation of corrosion effects on the bars by directly relating the observed DL to the electrochemical processes described by Faraday's law.²⁸ Further, a clearer understanding of the relationship between physical degradation and electrochemical corrosion phenomena is provided by this method.²⁹

The overall scheme of bar testing is given in Table 1 along with the target CLs. The bar d_i values are also provided in Table 1. The typical nomenclature is as follows: the first set of letters indicates the testing series based on the bar surface treatment, followed by the bar diameter, and the letter at the end is the target DL. For example, MC-D16DL1 is a machined D16 bar in Series 1 subjected to a 1 mm (0.04 in.) target DL. Further, DL0 signifies control specimens.

It is noted in Table 1 that D16 bars in Series 1 were subjected to five DL levels: 0.5, 1, 1.5, 2, and 2.5 mm (0.02, 0.04, 0.06, 0.08, and 0.1 in.). The DL levels for the bars of the remaining diameters were selected randomly to investigate the influence of reinforcing bar diameter on corrosion behavior and tensile properties. Further, the target CLs ranged from 10 to 44.7% to cover a broad spectrum of conditions. A CL up to 15% is considered low, while CLs of 16 to 25% and 26 to 35% correspond to medium and high levels, respectively.²⁴ Further, the bars UM-D16DL3 (Series 2) and UCM-D16DL3 (Series 3), as well as MC-D16DL1 (Series 1) and UCM-D16DL4 (Series 3), were used to study the influence of the corrosion technique on bar tensile behavior. The mass and diameter of the bar were recorded both before and after machining (where applicable), as well as at the end of the induced corrosion process.

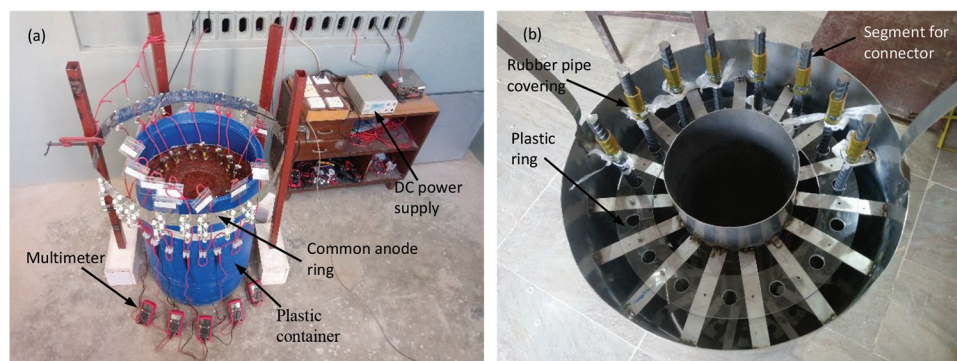
Accelerated corrosion method

The accelerated corrosion of the steel bars was induced and monitored using a galvanostatic accelerated corrosion test, which is an electrochemical corrosion technique. This method allows for the achievement of desired CLs in a short period³⁰⁻³² and is suitable for rapid testing and analysis of the corrosion behavior and mechanical properties of the steel bars.^{33,34}

A specially designed corrosion cell made from a plastic container was used for this accelerated corrosion test (Fig. 1). An integrated system of external adjustable direct current (DC) power supply (rated at 60 V and capable of delivering a current between 0 to 10 A) and multimeters (to monitor the current) was constructed to supply DC to the test specimen bars.

Table 1—Summary of bar diameters and corrosion levels

Series	Specimen ID	d_s , mm (in.)	d_b , mm (in.)		CL, %		Exposure duration, hours
			Target	Observed	Target	Observed	
Series 1	MC-D12DL1	10 (0.39)	9 (0.35)	9.04 (0.36)	19	18.3	179
	MC-D12DL2	10 (0.39)	8 (0.31)	8.25 (0.32)	35	31.9	330
	MC-D16DL0.5	13 (0.5)	12.5 (0.49)	12.33 (0.49)	7.5	10	92
	MC-D16DL1	13 (0.5)	12 (0.47)	11.88 (0.47)	15	16.5	184
	MC-D16DL1.5	13 (0.5)	11.5 (0.45)	11.47 (0.45)	22	22.2	270
	MC-D16DL2	13 (0.5)	11 (0.43)	11.03 (0.43)	28	28	344
	MC-D16DL2.5	13 (0.5)	10.5 (0.41)	10.47 (0.41)	35	35.1	430
	MC-D19DL1	16 (0.63)	15 (0.59)	14.93 (0.59)	12	12.9	181
	MC-D19DL2	16 (0.63)	14 (0.55)	14.03 (0.55)	23	23.2	347
	MC-D25DL2	22 (0.87)	20 (0.79)	20.06 (0.79)	17	16.8	353
	MC-D32DL2	29 (1.14)	27 (1.06)	26.93 (1.06)	13	13.8	356
Series 2	UM-D16DL1	16 (0.63)	15 (0.59)	14.67 (0.58)	12	15.9	181
	UM-D16DL2	16 (0.63)	14 (0.55)	13.67 (0.54)	24	27	363
	UM-D16DL3	16 (0.63)	13 (0.5)	12.67 (0.50)	34	37	514
Series 3	UCM-D16DL1	16 (0.63)	15 (0.59)	14.74 (0.58)	12	15.1	—
	UCM-D16DL2	16 (0.63)	14 (0.55)	13.95 (0.55)	24	24	—
	UCM-D16DL3	16 (0.63)	13 (0.5)	13.04 (0.51)	34	34	—
	UCM-D16DL4	16 (0.63)	12 (0.47)	11.90 (0.47)	44	44.7	—

**Fig. 1—View of galvanostatic accelerated corrosion test setup; (a) corrosion cell; and (b) drum-in-drum cathode.**

A 5% sodium chloride (NaCl) solution (salt water) was used as an electrolyte (Fig. 2). The positive terminal of the power supply was connected to a Grade 316 stainless steel ring, which served as the common anode. The stainless steel (Grade 316) drum-in-drum (surrounding the steel bars) acted as the cathode, which was connected to the negative terminal of the power supply. The drum-in-drum cathode connected to stainless steel fins ensures uniform corrosion of the bars along their perimeter. The bars were connected in parallel to the common anode (stainless steel ring), and resistors were placed between the common anode ring and the steel bars to control the desired electric current (Fig. 2). Figure 2 shows that the current flowing to each bar was held constant and monitored by connecting a multimeter between the bar and a resistor linked to the common anode. Alternatively, the current flow could be calculated using the total applied voltage and the resistor's value, though an ohmic drop occurs due to the inherent resistance of the electrolyte.³⁵

The corrosion cell could accommodate up to 15 bars simultaneously. The electrolytic solution was filled into the corrosion cell before installing the reinforcing bars into a plastic holder (Fig. 1). The exposed length of the bars in Series 1 was 700 mm (28 in.), although the primary interest was in the reduced section of the bar ($4d_b$). This step was to maintain the proportionality between the original and reduced sections before and after corrosion.²⁵

A 50 mm (2 in.) section of the top 100 mm (4 in.) of the bar (above the electrolyte level) was coated with silicone sealant, wrapped in polyvinyl chloride (PVC) waterproof electrical insulated sealing tape, and covered with a rubber pipe to prevent intrusion of rising rust, which could disrupt the connector segment (Fig. 3). The remaining top 50 mm (2 in.) of bar length was reserved for the connector for applying electric current to the bar. The bottom surface was also protected using epoxy to prevent longitudinal corrosion of the bar. The bars were thoroughly washed multiple

times in soapy water before applying a protective coating to ensure they were free from dust and oil contamination on the surface.

Conversely, a 70 mm (2.76 in.) section in the middle of the reinforcing bar was exposed to corrosion for the bars in Series 2. The remaining bar was carefully sealed using a multilayer process (Fig. 3). The bar was first wrapped with PVC waterproof electrical insulated sealing tape, followed by an application of silicone caulk. Finally, two additional layers of the tape were applied to ensure complete protection of the non-corroded regions.

The duration of the test was determined using Faraday's law (Eq. (2))

$$\Delta_m = \frac{t \times I \times M}{z \times F} \quad (2)$$

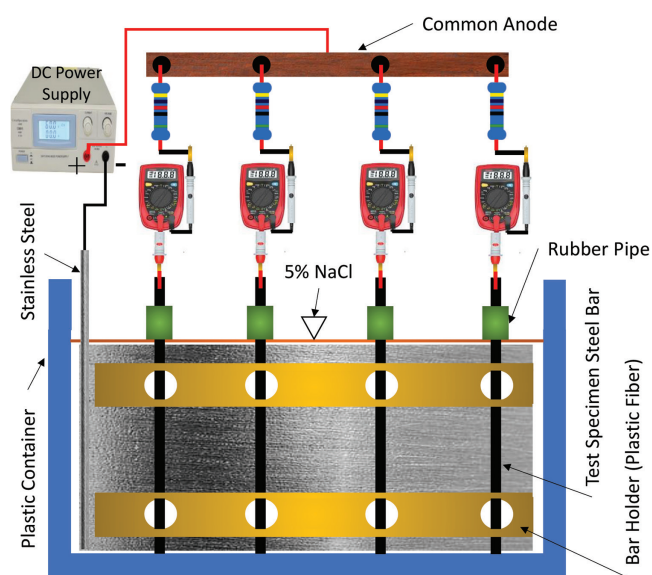


Fig. 2—Arrangements of induced bar corrosion using electrochemical corrosion method.

where Δ_m is the target mass loss of reinforcing bar (gm); I is the electric current (A); t is the time (seconds); M is the atomic weight of the metal; z is the ionic charge ($= 2$); and F is the Faraday's constant (96,487 C/mol [26.8 A·h/mol]).

A wide range of current densities has been employed in the literature to accelerate the corrosion of bare reinforcing bars using the impressed current method. However, there is increasing recognition that excessively high current densities may lead to corrosion products and morphologies that differ from those formed under natural exposure conditions. Andrade³⁶ cautioned that current densities exceeding approximately 200 $\mu\text{A}/\text{cm}^2$ (1.29 $\text{mA}/\text{in.}^2$) can create unrealistic conditions due to high local acidity, affecting the distribution and nature of corrosion products. As a result, several researchers recommend limiting the applied current density to below this threshold to better simulate natural corrosion behavior.

Nonetheless, a review of prior studies reveals considerable variation in the applied current densities, reflecting differing objectives, test durations, and trade-offs. For instance, El Maaddawy and Soudki³² noted that the mass loss predicted by Faraday's law depends on the total charge passed and is therefore independent of the specific current density applied. Others^{7,32,33,37-44} have used values ranging from 55 to 10,400 $\mu\text{A}/\text{cm}^2$ (355.84 to 67,031.36 $\mu\text{A}/\text{in.}^2$). Luo et al.⁴⁵ applied current densities between 1.16 and 2.56 mA/cm^2 (7.48 and 16.52 $\mu\text{A}/\text{in.}^2$), while Ahmed et al.¹⁹ noted that values between 0.01 and 2.4 mA/cm^2 (0.0645 and 15.48 $\text{mA}/\text{in.}^2$) have been used in past studies.

A current density of 2 mA/cm^2 (12.90 $\text{mA}/\text{in.}^2$) was selected in the present work to achieve the target mass loss (Eq. (2)) within a feasible experimental time frame. While this value exceeds the threshold proposed by Andrade³⁶ for replicating natural corrosion morphology, it was considered appropriate given the study's aim to accelerate corrosion for comparative assessment. This objective (along with the use of bare reinforcing bars) justified the selection of a higher current density. This approach (while not replicating field

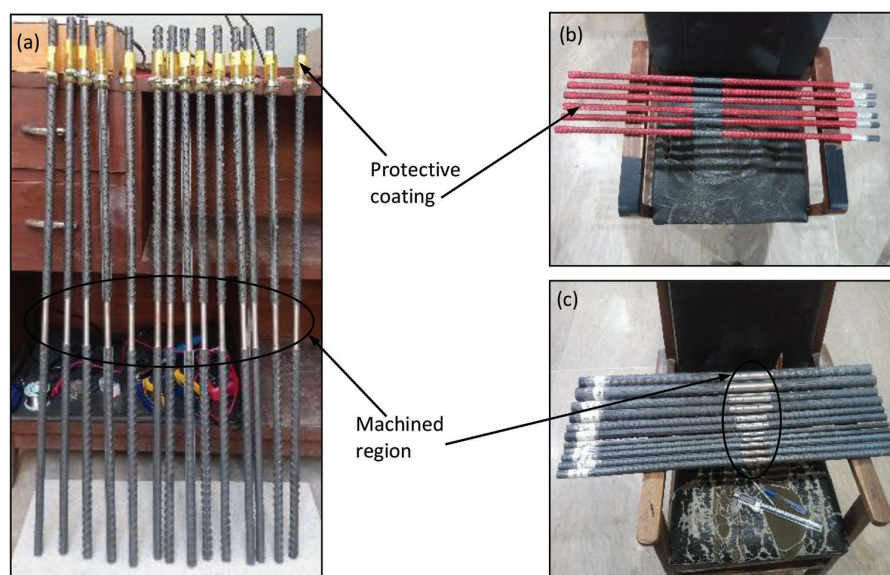


Fig. 3—View of bars ready for corrosion testing: (a) MC bars; (b) UM bars; and (c) UCM bars.



Fig. 4—Tensile strength testing of reinforcing bar.

conditions in full) is consistent with accelerated corrosion protocols adopted in several previous investigations.

After the corrosion process was completed, the bars were first brushed in tap water and then cleaned mechanically in soapy water using a brush, following ASTM G1-90(1999)e1⁴⁶ guidelines for removing corrosion products from reinforcing bar surfaces. Chemical cleaning was not performed because bare bars (unlike concrete-embedded bars) do not have tightly adherent corrosion products.

Tensile strength testing

The tensile strength of the bars was evaluated using a universal testing machine capable of a maximum load of 500 kN (112 kip), in accordance with ASTM E8/E8M.²⁷ This machine operates under displacement control, with adjustable rates ranging from 0.5 to 12 mm/min (0.02 to 0.47 in./min). The testing was conducted at a consistent machine head movement rate of 4 mm/min (0.16 in./min), as described by Rafi et al.²⁶ To measure the bar extension within the gauge length of $4d_b$, two linear variable differential transducers (LVDTs) were used (Fig. 4). Data on load and extension were continuously recorded using a data acquisition system.

The average results from these two or three bars were used to determine the mechanical properties of the bars. Control specimens, which were not subjected to corrosion, were also tested to compare the properties of corroded and non-corroded bars. In total, the study examined 50 reinforcing bar specimens.

RESULTS AND DISCUSSION

The tensile properties of the tested bars were determined using the stress-strain curves. The recorded load and elongation data were used to calculate the stress and strain of the bars, respectively. As mentioned earlier, the gauge length was taken as $4d_b$ for strain calculation.

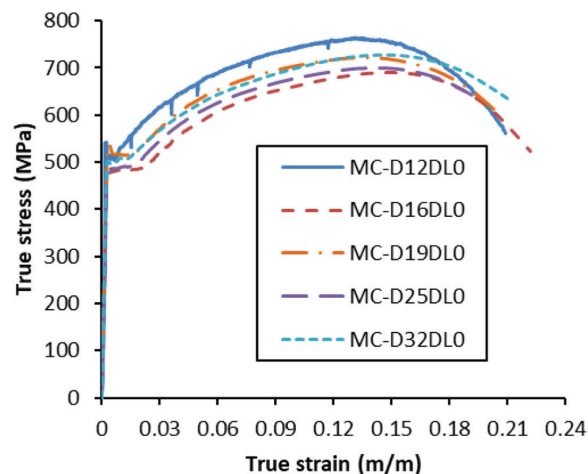


Fig. 5—Stress-strain curves for control specimens. (Note: 1 MPa = 145 psi.)

Behavior of control specimens

The average stress-strain curves for three control specimens of each bar diameter are shown in Fig. 5. The values of true stress and true strain are plotted to account for the material's nonlinear behavior and plastic deformation under applied loads. An inconsistent yield plateau is seen for bars of different diameters. The D12 (MC-D12DL0) bar exhibited a distinct but relatively short yield plateau. The D19 (MC-D19DL0) bar also had a distinct yield plateau, but its length was longer and comparable to that of the D32 (MC-D32DL0) bar. The longest yield plateau was observed for the D16 (MC-D16DL0) bar, followed by the D25 (MC-D25DL0) bar. This behavior differs from the findings of Rafi et al.,²⁶ who reported the longest yield plateau for the D12 bars and the shortest for the D16 and D25 bars.

All tested bars showed considerable strain hardening, with the highest strain hardening observed for the D12 bars. No clear pattern emerged to show the effect of bar diameter on this aspect of behavior, which contrasts with the findings of Rafi et al.,²⁶ who reported the highest strain hardening for D25 bars. Finally, identical rupture strains (ϵ_f) were observed for all the bars, as shown in Fig. 5. Rafi et al.²⁶ also found that ϵ_f was not influenced by bar diameter.

A summary of all important points on the stress-strain curves of the control reinforcing bars is given in Table 2. These include proportional limit (f_p), f_y , tensile strength (f_u), and elastic modulus (E_s). The bar f_y was calculated using the offset method (0.2% offset stress) from ASTM E8/E8M.²⁷ The reduction in the bar area at its failure due to necking was determined as a percentage of the initial area, while elongation (EL) at failure was measured within the gauge length and is expressed as a percentage of this length. It is noted in Table 2 that the small values of standard deviation (SD) for the recorded strengths of the bars signify consistency and uniformity in the material strengths. An exception is the f_p value of the D19 bars, where a slightly higher SD was observed.

It is noted in Table 2 that all bars (regardless of diameter) met the yield strength requirements of ASTM A615/A615M-22,⁴⁷ which specifies a minimum f_y of 420 MPa (60×10^3 psi). However, this strength for the D19 bars

Table 2—Mechanical properties of control bars

Reinforcing bar ID	f_p , MPa	f_y , MPa	f_u , MPa	E_s , GPa (ksi)	Reduction in reinforcing bar area, %	EL , %
MC-D12DL0	507 ± 3.88	509 ± 4.46	761 ± 1.68	207 (30 × 10 ³)	64	23.30
MC-D16ML0	483 ± 1.96	485 ± 1.94	692 ± 3.52	201 (29.2 × 10 ³)	62	24.83
MC-D19ML0	522 ± 17.45	524 ± 6.26	722 ± 8.41	206 (29.9 × 10 ³)	64	23.98
MC-D25ML0	478 ± 8.72	481 ± 2.76	700 ± 9.42	203 (29.4 × 10 ³)	60	23.43
MC-D32ML0	493 ± 4.32	495 ± 6.84	728 ± 3.48	204 (29.6 × 10 ³)	59	24.96

Note: 1 MPa = 145 psi.

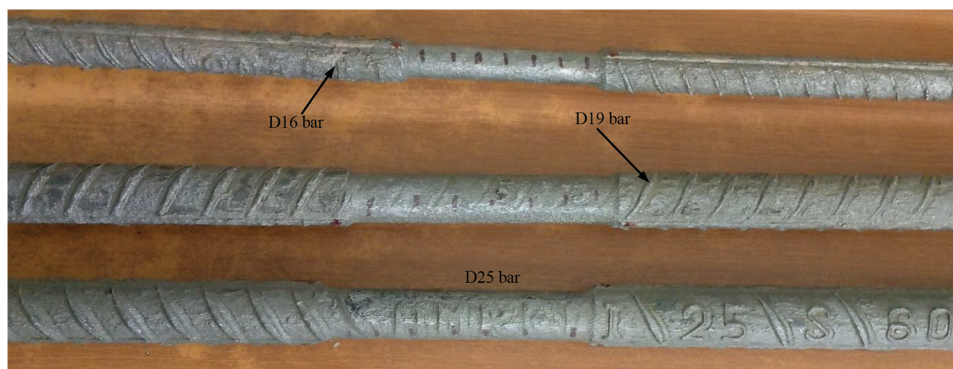


Fig. 6—View of corroded bars after cleaning of corrosion product.

was significantly (24%) higher. The smallest strength of 481 MPa (70×10^3 psi) observed for the D25 bars is also 14.5% higher than the ASTM A615/A615M⁴⁷ recommendation of 420 MPa (60×10^3 psi). This applies to the D16 bar as well, with $f_y = 485$ MPa (70×10^3 psi).

Table 2 also shows that f_u of the bars varied randomly with bar diameter and exceeded 620 MPa (90×10^3 psi), the value recommended by ASTM A615/A615M.⁴⁷ The D12 bar exhibited the highest ultimate strength, which was 23% above the recommended value. In contrast, the D16 bar had the lowest ultimate strength but was still 12% above the ASTM recommendation. The aforementioned variations suggest inconsistent chemical properties among the bars, a finding highlighted by Rafi et al.⁴⁸

The data for E_s were obtained as the tangent modulus of the initial linear-elastic portion of the stress-strain curve. Table 2 shows that all bars had similar E_s values, indicating consistency. This consistency is also reflected in the reduction in the bar area at failure and the elongation of the bars. The elongation requirements specified by ASTM A615/A615M⁴⁷ are as follows: 9% for diameters up to 19 mm (0.75 in.), 8% for 25 mm (1 in.), and 7% for 32 mm (1.26 in.) diameter bars. It is noted in Table 2 that all tested bars met the elongation requirements set by ASTM A615/A615M.⁴⁷

Behavior of corroded bars

The variations in the average diameters of the corroded bars were insignificant, suggesting uniform corrosion with negligible pitting effects in the machined regions of the bars exposed to corrosion. The observed values of d_f for the bars and corresponding CLs are given in Table 1. It is noted that the observed CLs closely matched the target CLs. Monitoring the current during the test indicated that the ohmic drop was minimal and negligible, as the current remained

stable during frequent checks. Consequently, no further adjustments to the voltage from the DC power supply were necessary, allowing the observed CLs to align well with those predicted by Faraday's law. These findings are consistent with results reported by Haris and Gadve,²⁴ who found an agreement within 10%. Additionally, El Maaddawy and Soudki³² reported a measured-to-predicted mass loss ratio ranging from 0.82 to 1.15. It is important to note that the ohmic drop and observed CLs are generally higher and lower, respectively, for bars embedded in concrete, due to the material's high resistance compared to bare bars.⁴ While Faraday's law assumes that mass loss begins immediately upon the application of current, there is often a delay in corrosion initiation. This delay occurs because time and energy are required to establish conditions conducive to corrosion for steel bars embedded in concrete. Figure 6 illustrates a view of a few corroded bars from Series 1.

Effect of corrosion technique—Figure 7 illustrates the average residual stress-strain curves of D16 bars subjected to accelerated corrosion and simulated corrosion techniques. The stress was calculated using the reduced area of the bar obtained from both techniques for a meaningful comparison. As shown in Fig. 7, the mechanical behaviors of the bars are similar for equivalent DL across both corrosion methods. This implies that the corrosion methods yield consistent effects on the mechanical properties of the bars, suggesting that either technique can be reliably used to assess the corrosion impact in practical applications.

Furthermore, it is seen in Fig. 7 that the machining of the bars did not significantly influence the corrosion morphology or mechanical behavior, as evidenced by the similar stress-strain responses obtained across corrosion techniques. This suggests that the surface preparation method did not introduce any bias in evaluating the effects of corrosion.

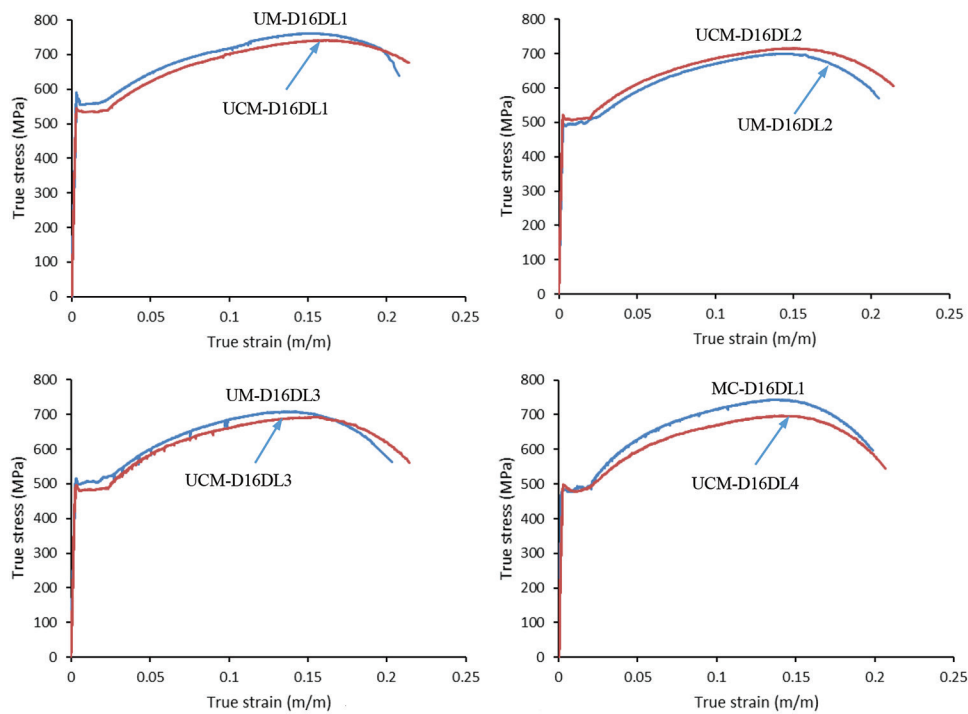


Fig. 7—Study of effects of corrosion technique on bar behavior. (Note: 1 MPa = 145 psi.)

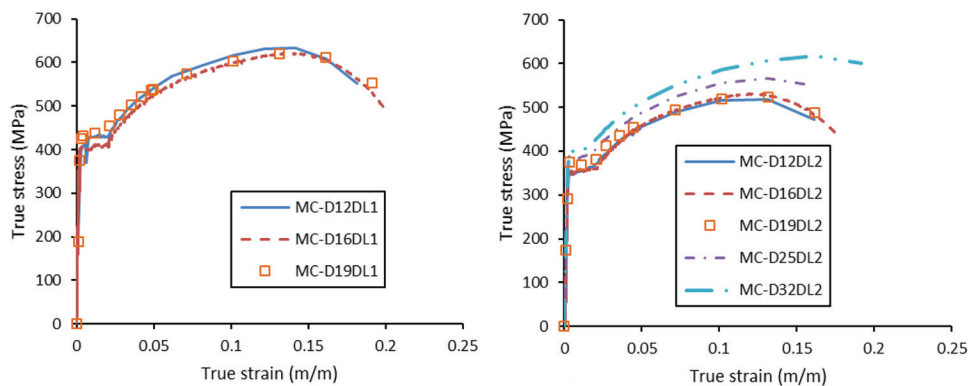


Fig. 8—Comparison of residual stress-strain curves based on equal diameter loss. (Note: 1 MPa = 145 psi.)

Effect of reinforcing bar diameter—Two types of comparisons were made to study the influence of corroded bar diameter on the residual stress-strain curve. Figure 8 compares the average stress-strain curves of corroded bars of different diameters subjected to the same DL. Conversely, Fig. 9 presents a comparison based on similar observed CL of the bars. The stress values in Fig. 8 and 9 were determined using the nominal cross-sectional area of the uncorroded bars (Table 1), as suggested by Imperatore et al.⁴⁹ The capacity of structural members in RC structures is also based on the nominal cross-sectional area of the bar in the provisions of design codes such as ACI 318-19⁵⁰ and Eurocode.⁵¹

It is seen in Fig. 8 and 9 that the stress-strain curves show minimal sensitivity to bar diameter across both comparison types. The D12, D16, and D19 bars exhibited similar behaviors at DLs of 1 and 2 mm (0.04 and 0.079 in.) (Fig. 8). The maximum differences in residual f_y (f_{yR}) and residual f_u (f_{uR}) for the MC-D25DL2 bars compared to these bars were 12.5% and 10.5%, respectively. Interestingly, the tested MC-D32DL2 specimens displayed an unexpected response,

deviating from the overall trends observed in the other bars. This anomaly may be attributed to the uncertain mechanical properties of the reinforcing bars commonly available in the construction industry in Pakistan.^{48,52}

It is evident from Fig. 9 that the data for the D12, D16, D19, and D25 bars are closely aligned and remain largely consistent with each other for similar CLs. The maximum variations in f_{yR} and f_{uR} were 11% and 13.5%, respectively. This suggests that the observed deviations between the corroded bars of different diameters are consistent with those seen between their corresponding control specimens, indicating that the behaviors of the corroded bars are not influenced by bar diameter. While Haris and Gadve²⁴ noted variations in f_{yR} and f_{uR} to be dependent on bar diameter, Du et al.¹⁶ found that the ductility of the bar is not influenced by the bar diameter. Finally, the residual ϵ_f (ϵ_{fR}) of the bars was found to be similar at the same DLs and similar CLs.

Effect of corrosion level—Figure 10 illustrates the average stress-strain curves of the D16 corroded bars in Series 1 subjected to varying CLs. The stress-strain curve of the control

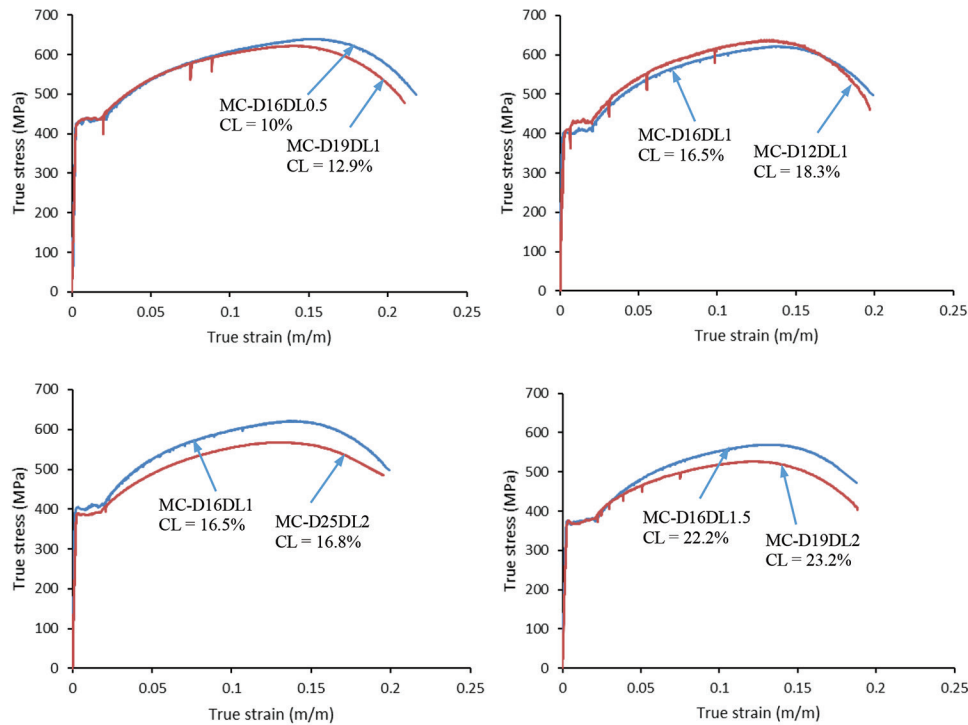


Fig. 9—Comparison of residual stress-strain curves based on similar corrosion level. (Note: 1 MPa = 145 psi.)

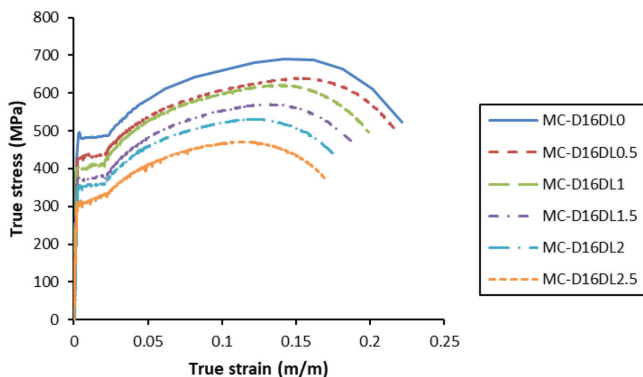


Fig. 10—Stress-strain curves of D16 bars at varying corrosion levels. (Note: 1 MPa = 145 psi.)

D16 bars (MC-D16DL0) is also included in Fig. 10 for comparison. As before, the stress values were determined based on the nominal cross-sectional area of the uncorroded bars.

As shown in Fig. 10, the bar behaviors are similar in many respects. First, the yield plateau for the bars remained constant across different CLs and closely resembled that of the control specimens. All bars exhibited similar strain-hardening behavior, although this was inconsistent with the observed CLs. Further, the slope of the initial linear portion of the stress-strain curves remained consistent for both corroded and uncorroded bars. Finally, f_{yR} , f_{uR} , and ϵ_{fR} decreased with increasing CLs.

A detailed summary of the residual tensile properties of the bars in Fig. 10 is provided in Table 3. It is noted that the reduction in f_{yR} and f_{uR} varies randomly at different CLs. This variability may partly stem from the fact that the strength of the corroded reinforcement is influenced by the extent of localized attack penetration for a specified degree of

corrosion. Increased severity of local penetration leads to a greater reduction in the strength of the corroded reinforcing bar.^{53–55} The maximum decrease in f_{yR} and f_{uR} at a 35% CL was 55% and 47%, respectively, which are similar. These findings align qualitatively with those reported by Du et al.¹⁶

Table 3 indicates that all other residual strains reduced with increasing CLs, except residual proportional strain (ϵ_{pR}). Nevertheless, the elastic modulus of the reinforcing bars remained unaffected by corrosion, which aligns with the findings of Haris and Gadve.²⁴ Finally, the reduction in ϵ_{uR} and ϵ_{fR} values was higher with increasing CLs as compared to other strains, signifying reduced bar elongation and ductility.

Several studies^{2,4,13,15,16,24,56} have investigated the behavior of bare bars under varying CLs. These studies consistently demonstrate that corrosion significantly reduces the ductility of reinforcing bars. For example, Du et al.¹⁶ reported a 29% reduction in ϵ_{uR} , reinforcing bar area, and elongation at 10% corrosion. Similarly, Haris and Gadve²⁴ documented a nearly 10% reduction in elongation at the same CL. Conversely, this study observed a nearly 6% reduction in ϵ_{fR} at approximately 10% corrosion (Table 3), indicating a comparatively lesser reduction in ductility at higher CLs. This discrepancy could be attributed to the lesser effect of localized penetration in the case of machined bars and corrosion over a small length, specifically the reduced section of $4d_b$. Nevertheless, these findings underscore the significant detrimental effects of corrosion on the ductility of reinforcing bars, highlighting the need for careful consideration of corrosion in the design and assessment of RC structures. In addition, this also indicates the need to assess the applicability of available analytical models for these bars.

Effects of reinforcing bar ductility—The ductility of reinforcement is characterized by the criteria of the strength

Table 3—Mechanical properties of D16 bars at varying corrosion levels

Reinforcing bar ID	Proportional limit	ϵ_{pR} , %	Yield point	ϵ_{yR} , %	Ultimate stress point	ϵ_{uR} , %	Fracture point	ϵ_{fR} , %	E_{sR} , GPa (ksi)
	f_{pR} , MPa		f_{yR} , MPa		f_{uR} , MPa		f_{fR} , MPa		
MC-D16DL0	483 ± 1.96	0.240	485 ± 1.94	0.458	692 ± 3.52	15.6	523 ± 3.80	22.2	201 (29.2 × 10 ³)
MC-D16DL0.5	424 ± 5.88	0.234	426 ± 6.78	0.452	636 ± 31.44	14.8	496 ± 36.60	21.2	202 (29.3 × 10 ³)
MC-D16DL1	396 ± 1.43	0.235	397 ± 1.44	0.491	617 ± 5.18	14.1	494 ± 0.60	19.9	203 (29.4 × 10 ³)
MC-D16DL1.5	370 ± 4.77	0.231	372 ± 4.89	0.442	575 ± 3.41	13.3	476 ± 5.77	18.8	204 (29.6 × 10 ³)
MC-D16DL2	351 ± 7.84	0.238	352 ± 7.19	0.485	532 ± 4.30	12.2	426 ± 2.39	17.8	204 (29.6 × 10 ³)
MC-D16DL2.5	313 ± 5.05	0.238	314 ± 5.31	0.446	472 ± 5.79	11.6	369 ± 7.79	17.1	202 (29.3 × 10 ³)

Note: 1 MPa = 145 psi.

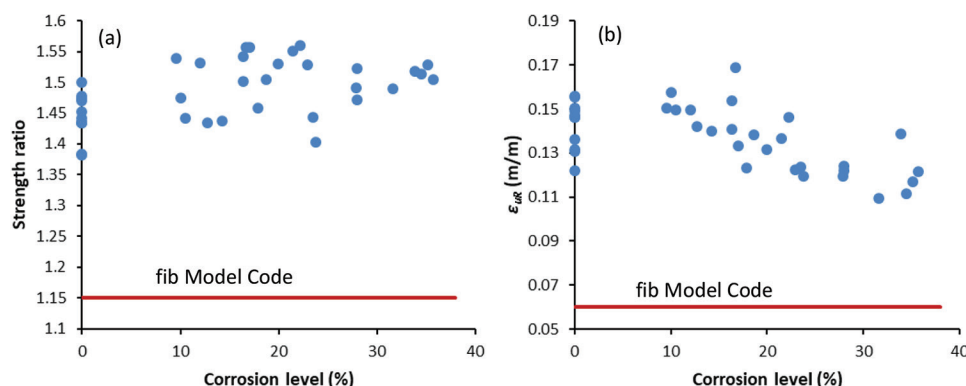


Fig. 11—Effect of corrosion on reinforcing bar ductility: (a) variations in strength ratio; and (b) variations in ultimate strain.

ratio and the ϵ_u value, as defined by the *fib* Model Code.⁵⁷ The strength ratio is defined as f_{uR}/f_{yR} for the steel bars. Data from all tested bars in Series 1 were employed to investigate the ductility characteristics of the corroded bars. For the high-ductility Class S reinforcing bars, the *fib* Model Code⁵⁷ recommends a strength ratio of 1.15 and ϵ_u value of 6%. Figure 11 illustrates the effects of corrosion on the residual strength ratio and ϵ_{uR} . It is seen that all tested bars met both ductility criteria at all CLs, with a sufficient margin. Although Du et al.¹⁶ reported failures of reinforcing bars in meeting the aforementioned ductility criteria at nearly 10% CL, the findings in this study differ.

Statistical evaluation and decay models

Different researchers^{24,49} conducted statistical analyses on the data for the corroded bars and proposed analytical methods for the estimation of residual mechanical properties corresponding to a given CL. These methods predict material stress and strain from engineering stress and strain measurements, which are used in the forthcoming discussion.

Haris and Gadve²⁴ proposed Eq. (3) to (5) for predicting f_{yR} , f_{uR} , and elongation (EL_R) of corroded bars, respectively.

$$f_{yR} = -7.14749CL - 2.65793d_b + 575.26183 \quad (3)$$

$$f_{uR} = -8.32148CL - 0.05245d_b + 638.79706 \quad (4)$$

$$EL_R = -0.23362CL - 0.0431d_b + 22.50831 \quad (5)$$

Imperatore et al.⁴⁹ suggested Eq. (6) to (8) for f_{yR} , f_{uR} , and ϵ_{uR} , respectively.

$$f_{yR} = f_y \times (1 - 0.0150957CL) \quad (6)$$

$$f_{uR} = f_u \times (1 - 0.0138222CL) \quad (7)$$

$$\epsilon_{uR} = \epsilon_u e^{-0.027713CL} \quad (8)$$

Figure 12 compares the predicted data with the experimental results of the reinforcing bars tested in this study. Data points below the diagonal line indicate a conservative estimate of the given quantity relative to the observed values, while points above the line represent overestimations. It is seen that although the analytical models provided reasonable estimates of f_{uR} , predictions for f_{yR} varied. The model by Haris and Gadve²⁴ slightly underestimated f_{yR} at intermediate CLs, while the model by Imperatore et al.⁴⁹ tended to overestimate f_{yR} at higher CLs.

Moreover, it is seen in Fig. 12 that both ϵ_{uR} and ϵ_{fR} are significantly overestimated by the available analytical methods. These findings suggest that there is room for improvement in existing analytical models to enhance their predictive accuracy, particularly in capturing the residual

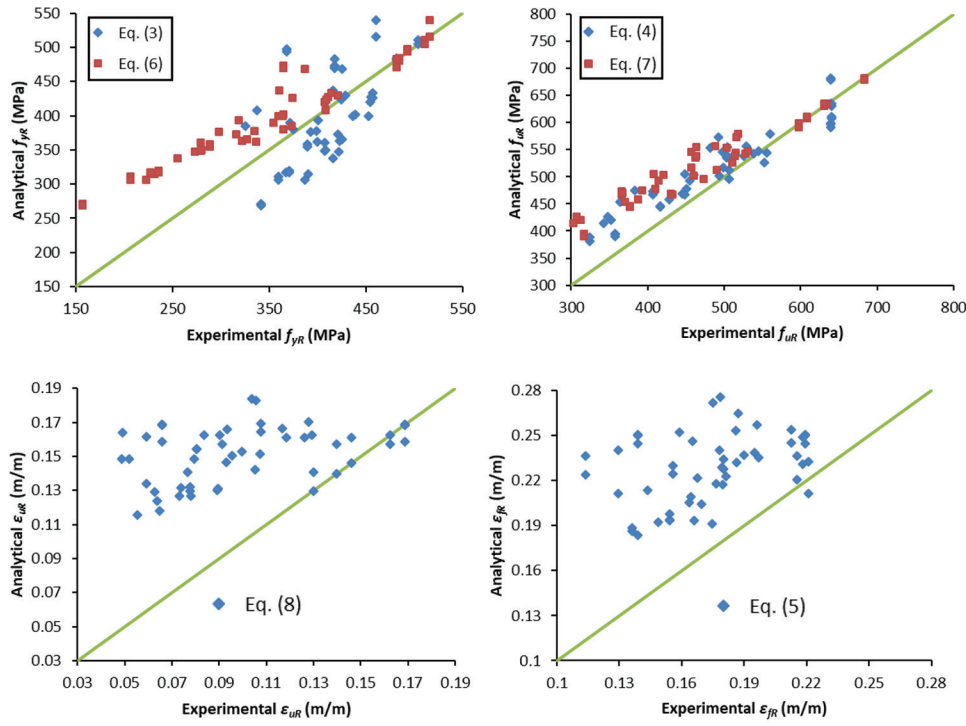


Fig. 12—Comparison of experimental and predicted tensile properties. (Note: 1 MPa = 145 psi.)

stress-strain behavior of corroded reinforcement. While several models have been proposed in the literature, limitations remain in accurately estimating strains at key limiting points, which are critical for developing reliable constitutive models. Continued refinement of these models could contribute to more comprehensive and robust predictions under corrosion-damaged conditions.

Given the previous information, the data of residual properties of all 50 bars tested in this study were employed to develop analytical methods for estimating the tensile properties of reinforcing bars at a given CL. Figure 13 illustrates variations in residual stress at various limiting strength levels with CLs. A linear regression of the data allowed the development of the relation between stress and CL, which is summarized in Eq. (9) to (12) for f_{pR} , f_{yR} , f_{uR} , and f_{fR} , respectively. As shown in Fig. 13, the correlation coefficients are between 0.87 and 0.93, indicating a strong correlation between the respective residual strengths of the bars.

$$f_{pR} = -5.05CL + 493.58 \quad (9)$$

$$f_{yR} = -5.07CL + 495.13 \quad (10)$$

$$f_{uR} = -6.24CL + 628.23 \quad (11)$$

$$f_{fR} = -4.51CL + 461.4 \quad (12)$$

Further, the residual strength of corroded reinforcing bars can be correlated with uncorroded bars by Eq. (13)²

$$f_R = f \times (1 - \beta \times CL) \quad (13)$$

where β is the strength factor.

The values of the yield (β_y) and ultimate (β_u) strengths for the corroded bars were in the range of 0.0013 to 0.015 and 0.0032 to 0.012, respectively. Haris and Gadve²⁴ reported the strength factors β_y and β_u in the range of 0.0115 to 0.0279 and 0.0107 to 0.0179, respectively. Similarly, the factors β_y and β_u were found to be in the range of 0.0076 to 0.0333 and 0.0061 to 0.0259, respectively, by Imperatore et al.⁴⁹ It is noted that the observed range of variation in these factors in this study is much wider compared to that reported by other researchers. This signifies better corrosion resistance of the TMT bars tested in this study. Various researchers^{21,22,58} have noted that TMT bars typically exhibit better mechanical properties and corrosion resistance than conventional carbon steel reinforcing bars.

Figure 14 illustrates the correlation between residual strains at various limiting points and CLs. A significant dispersion of the data is seen in Fig. 14, indicating that the residual strains exhibit greater variability in corroded reinforcing bars. This variability can be attributed to several interrelated factors, including differences in the reinforcing bar surface conditions,^{59,60} the heterogeneous nature of corrosion, and the localized nature of corrosion attacks. These factors can lead to irregular strain distributions along the gauge length, especially in the post-yield phase, where strain localization is more pronounced.

Given this inherent variability, some data points exhibit behavior that deviates substantially from the general trend and are considered statistical outliers. A regression analysis was performed using only the primary band of data in Fig. 14 (excluding these outliers) to establish practical and reliable predictive relationships. This approach enabled the derivation of empirical correlations (Eq. (14) to (17)) with strong correlation coefficients ($R^2 = 0.81$ to 0.85), capturing the dominant behavior without being overly influenced by

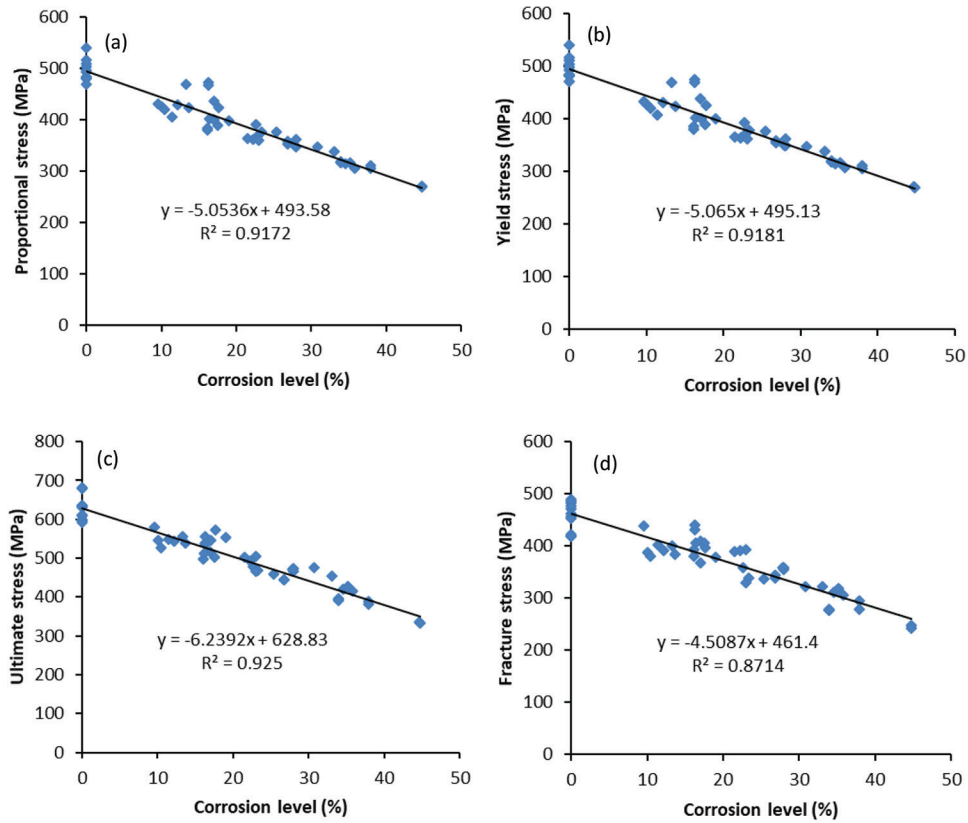


Fig. 13—Variations in residual strength: (a) proportional strength; (b) yield strength; (c) ultimate strength; and (d) fracture strength. (Note: 1 MPa = 145 psi.)

extreme values. This method is consistent with common engineering practice, where outliers (often due to experimental anomalies or localized effects) are excluded to improve model robustness and applicability.

$$\varepsilon_{pR} = 2 \times 10^{-8} \text{CL}^2 - 9 \times 10^{-6} \text{CL} + 0.0027 \quad (14)$$

$$\varepsilon_{yR} = 1 \times 10^{-6} \text{CL}^2 - 6 \times 10^{-5} \text{CL} + 0.0051 \quad (15)$$

$$\varepsilon_{uR} = -1 \times 10^{-5} \text{CL}^2 - 9 \times 10^{-4} \text{CL} + 0.1673 \quad (16)$$

$$\varepsilon_{fR} = -2 \times 10^{-5} \text{CL}^2 - 8 \times 10^{-4} \text{CL} + 0.2445 \quad (17)$$

CONSTITUTIVE STRESS-STRAIN MODEL

Rafi et al.²⁶ reported that the constitutive relation suggested by Eurocode 2⁶¹ (Eq. (18)) can adequately provide a stress-strain curve for the TMT bars available in Pakistan up to the yield point.

$$\sigma_{sR} = \varepsilon_{sR} E_{sR} \text{ for } \varepsilon_{sR} \leq \varepsilon_{pR}$$

$$\sigma_{sR} = c + \left(\frac{b}{a}\right) \sqrt{a^2 - (\varepsilon_{yT} - \varepsilon_{sT})^2} \text{ for } \varepsilon_{pR} \leq \varepsilon_{sR} \leq \varepsilon_{yR} \quad (18)$$

$$\sigma_{sR} = f_{yR} \text{ for } \varepsilon_{sR} \geq \varepsilon_{yR}$$

where

$$a^2 = (\varepsilon_{yR} - \varepsilon_{pR})(\varepsilon_{yR} - \varepsilon_{pR} + [c/E_{sR}])$$

$$b^2 = c(\varepsilon_{yR} - \varepsilon_{pR})E_{sR} + c^2$$

$$c = \frac{(f_{yR} - f_{pR})^2}{(\varepsilon_{yR} - \varepsilon_{pR})E_{sR} - 2(f_{yR} - f_{pR})}$$

Considering that Eq. (18) does not consider strain hardening of the bar, Rafi et al.¹⁴ proposed Eq. (19) for the strain-hardening region of these bars to be used in combination with Eq. (18)

$$\sigma_{sR} = a_1 f_{yR} \varepsilon^2 + a_2 f_{yR} \varepsilon + a_3 f_{yR} \text{ for } \varepsilon_R \geq \varepsilon_{yR} \quad (19)$$

The values of constants a_1 , a_2 , and a_3 were suggested as -44 , 11.5 , and 0.93 , respectively, for determining the strain-hardening region of the bar using true stress and true strain values. Considering that engineering properties are routinely required in the analysis and design of RC structures⁴⁹ and also reported by the material testing labs, the data and the methodology reported by Rafi et al.¹⁴ were used to calibrate these constants for determining the strain hardening of steel bars based on engineering properties. The resulting values of the constants a_1 , a_2 , and a_3 were -36 , 8.0 , and 0.9 , respectively. This allows practical application of the constitutive laws given in Eq. (18) and (19) for determining residual constitutive relations, which are often required in nonlinear analysis.

Figure 15 compares the experimental stress-strain curves with those predicted by Eq. (18) and (19), using modified constant values in Eq. (19). The residual stress and strain

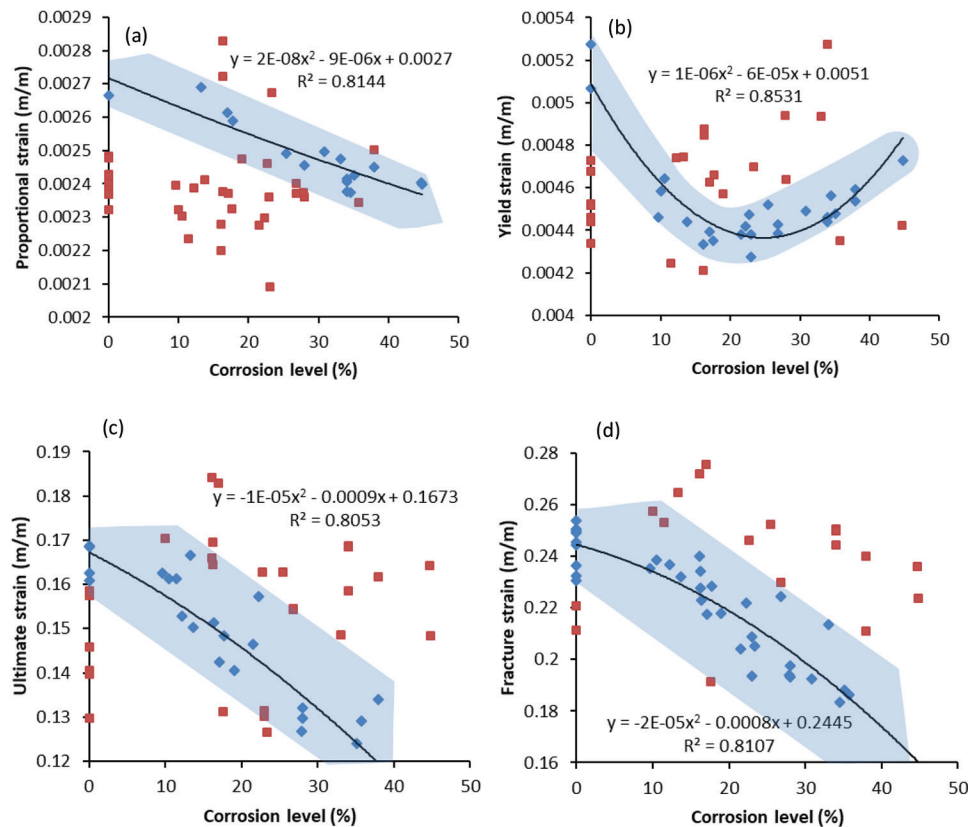


Fig. 14—Variations in residual strain: (a) proportional strain; (b) yield strain; (c) ultimate strain; and (d) fracture strain. (Note: Data within light blue band were used for regression analysis. Red markers indicate outlier points excluded from regression. Full-color PDF can be accessed at www.concrete.org.)

values were determined from Eq. (9) to (12) and (14) to (17), respectively. It is seen that the analytical models provide a good and consistent match with the experimental data. Therefore, it can be inferred that Eq. (18) and (19) are capable of estimating the residual constitutive stress-strain curves for retrofitting corrosion- and fire-damaged structures based on their respective residual properties.

CONCLUSIONS

This paper presents the results of an investigation into the effects of both electrochemically and mechanically induced corrosion on the mechanical properties of thermomechanically treated (TMT) steel bars. The corroded bars were tested under uniaxial tension to determine their residual properties, which were then compared with those of uncorroded bars. The study examined the effects of reinforcing bar diameter, corrosion technique, and corrosion levels (CLs) on the residual mechanical properties. The main findings of the study are listed as follows.

1. The decrease in the nominal yield strength and ultimate strength of the corroded bars was nearly proportional to the CL, indicating a linear relationship.
2. The tensile behavior of the corroded bars was not significantly influenced by the bar diameter or corrosion technique.
3. Although the elongation of the bars decreased with increasing CLs, they still met the ductility criteria based on both the ultimate strain and strength ratio, with a sufficient margin.

4. The strength factors for the yield strength and ultimate strength for the corroded bars ranged from 0.0013 to 0.015 and 0.0032 to 0.012, respectively, which were wider than the ranges reported in the literature. This suggests that the corroded bars in this study retained more of their original strength than those in previous studies. These findings imply that TMT steel bars may exhibit better resistance to corrosion-induced strength loss under similar conditions than was previously anticipated.

5. Analytical methods for estimating residual strength (Eq. (9) to (12)) and residual strain (Eq. (14) to (17)) at key points on the stress-strain curve have been proposed. A constitutive model (Eq. (18) and (19)) for predicting the residual stress-strain relationship of the bars has also been suggested, showing a good correlation with the observed experimental data.

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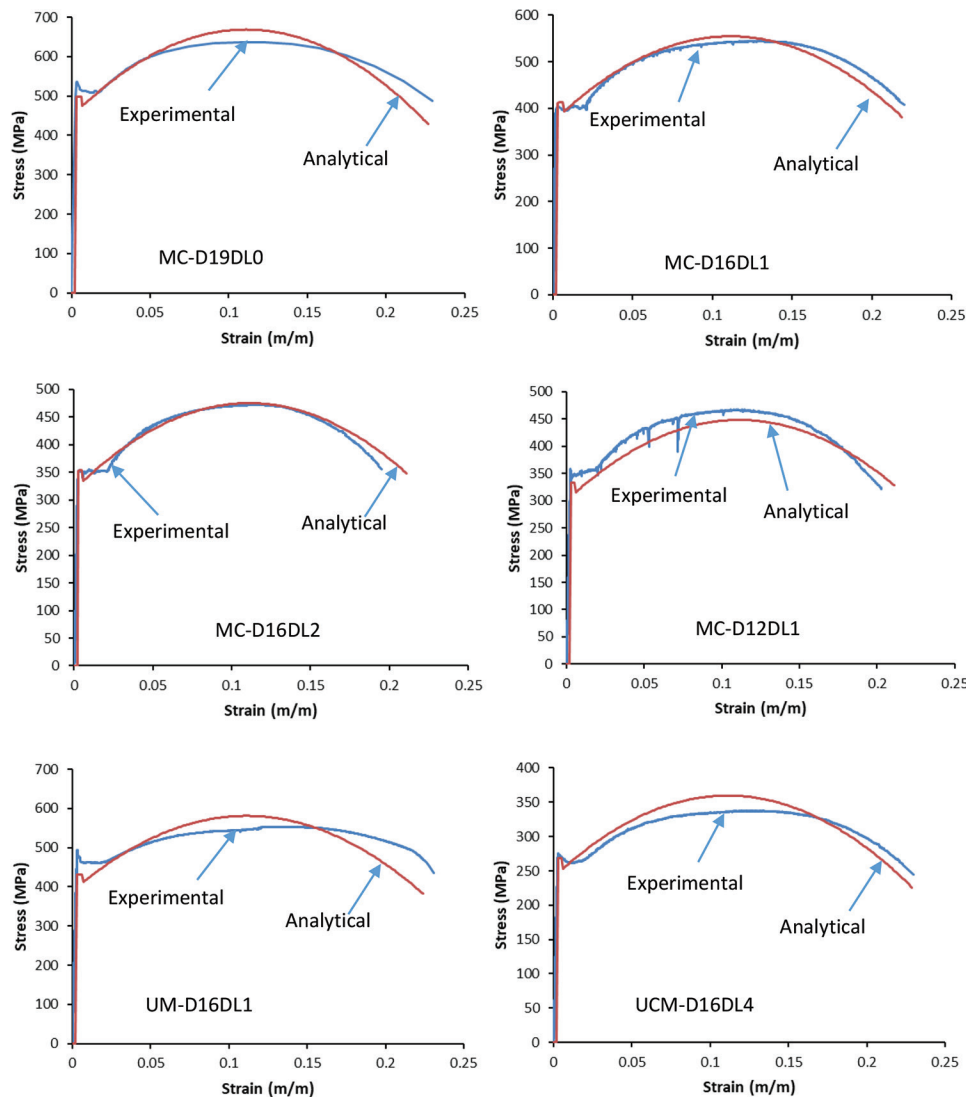


Fig. 15—Comparison of experimental and analytical stress-strain curves. (Note: 1 MPa = 145 psi.)

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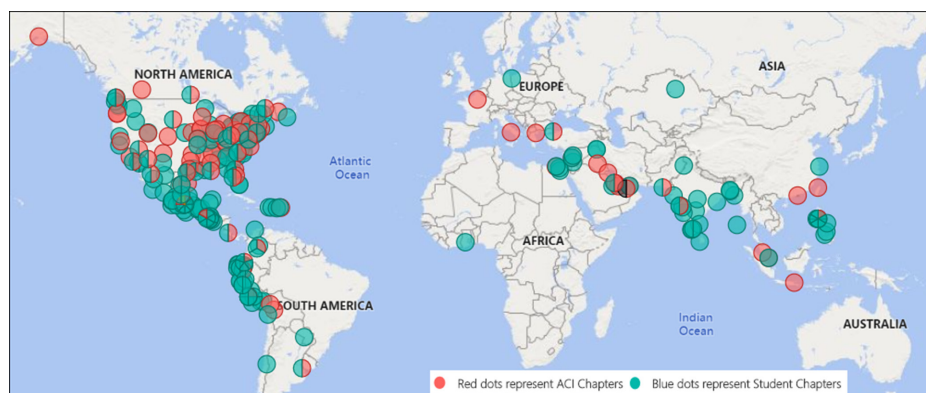
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Application of Low-Cement, High-Dosage High-Range Water-Reducing Admixture Concrete

by S. J. S. Bukhari, L. Bouchelil, A. Al Fahim, and M. Khanzadeh Moradllo

The production of ordinary portland cement (OPC) is a major contributor to carbon emissions. One immediate and viable solution is the use of optimized concrete mixtures that employ a decreased quantity of cement and increased dosage of high-range water-reducing (HRWR) admixtures. This study investigates five different concrete mixtures with varying water-cement ratios (w/c) (0.37 to 0.42) and reduced cement contents. The mixtures with “low-cement + high-dosage HRWR admixture” content had over 30% increase in mechanical strength and presented 40% lower water absorption, as well as 68 to 97% higher formation factor, indicating enhanced durability. The optimized concrete mixtures with reduced cement and lower w/c have a service life increase of up to 117% and a life-cycle cost reduction of 29%. The application of low-cement + high-dosage HRWR admixture mixtures can improve the sustainability of concrete mixtures by reducing cement and water contents and increasing the service life of concrete in severe environments.

Keywords: aggregate optimization; durability; formation factor; high-range water reducer (HRWR); life-cycle costs; packing density; service life; sustainability.

INTRODUCTION

The production of ordinary portland cement (OPC) is an energy-intensive (an average thermal energy intensity of 3.5 GJ/ton of clinker) and carbon-emitting process (≈ 1 ton CO_2 /ton cement) and accounts for approximately 5 to 8% of anthropogenic CO_2 emissions.¹ It is crucial to find practical and economically feasible solutions to reduce CO_2 emissions associated with cement and concrete production. Replacing conventional concrete mixtures (with a water-cement ratio $[w/c] \geq 0.40$) with a low- w/c (≤ 0.37) and low-cement-content (low w/c + low cement) concrete can be a viable immediate solution. This can address the sustainability challenge from two different perspectives: reducing CO_2 emissions from OPC production (≈ 200 million tons annual reduction) and reducing the amount of water consumed during the production of concrete. Increased uncertainty in the supply and availability of supplementary cementitious materials (SCMs) further highlights this need. However, this change would require the use of a higher dosage of high-range water-reducing (HRWR) admixtures (for example, higher than the dosage recommended by the manufacturer) as well as implementing the concept of optimized graded aggregate in low- w/c systems.

HRWRs can be employed to improve the performance of concrete (by reducing w/c), lower the cost, and improve sustainability (by reducing the cement content). Water-reducing admixtures can present various levels of effectiveness depending on the dosage used, the chemical

composition of the admixture itself, temperature, cement chemistry, fineness, and other mixture characteristics.^{2,3} HRWRs can reduce mixing water requirements by 12 to 30%.⁴ For instance, a water reducer with a 20% “effectiveness” would enable a 20% reduction in water content with no loss in slump. Water-reducing admixtures achieve these levels of effectiveness primarily through deflocculation of cement grains by modification of electrical charges on the cement surfaces.³ It should be noted that mixtures with a high dosage of HRWR generally can display a high rate of slump loss and excessive retardation.² However, systematic research is needed to quantify the impact of the application of higher dosages of HRWR on fresh and hardened properties of low- w/c + low-cement concrete mixtures, as well as initial and life-cycle costs.

Additionally, optimizing aggregate gradation (that is, minimizing the voids between aggregates that must be filled with cement paste) can enhance concrete strength, workability, durability, and sustainability.⁵ Research indicates that the application of particle packing models (PPMs) such as the modified Toufar model (MTM) and the compressible packing model (CPM) can notably decrease the amount of cement (that is, decrease in paste content) in concrete by enhancing the structure of its aggregates.^{6–8} These models focus on optimizing the particle-size distribution (PSD) or grading within the aggregate framework in concrete.^{9–11} Additionally, graphical combined gradation techniques based on practical experience have been introduced and implemented.^{12–16} For example, in the aggregate suspension mixture proportioning method (used in ACI PRC-211.1-22), the optimal combination of aggregates is selected based on grading, size, shape, angularity, and texture.¹⁶ Obla et al.¹⁷ proposed optimized mixture proportioning based on a paste-to-void ratio to achieve the required strength, workability, and durability. Previous studies also implemented other concrete proportioning approaches to determine the volume of the cement paste in excess of the amount required to fill the voids of the compacted combined aggregate to improve the consistency of concrete mixtures.^{18,19} However, a limited number of studies investigated the minimum possible cement content to satisfy the required fresh and hardened properties. Additionally, previous studies on concrete

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mixtures with reduced/low cement content primarily concentrated on substituting cement with SCMs such as fly ash and ground-granulated blast-furnace slag. However, as mentioned, due to uncertainty in the availability and quality of frequently used SCMs,²⁰ the application of optimized low-cement OPC mixtures without SCMs seems crucial.

This study aims to address the aforementioned needs for the application of low-cement + high-dosage HRWR admixture plain concrete mixtures by determining the minimum possible cement content and the optimum dosage of HRWR admixtures. The impact of a higher dosage of HRWR and low cement content on the fresh and hardened (mechanical and durability) properties of concrete mixtures was examined. Additionally, life-cycle cost analysis (LCCA) is performed to quantify the impact of the application of the recommended sustainable concrete mixture on the life-cycle costs when compared to the control conventional concrete mixtures. This includes the impact of the reduction in cement and water contents and the increase in HRWR dosage, as well as the impact of the predicted service life of concrete. Incorporation of concrete service life is significant because durability can be a limiting factor for low-cement concretes. This study assesses the durability performance of the developed concrete mixtures in a reinforced concrete bridge deck exposed to chlorides. The findings of this study can lead to more sustainable concrete production for different applications. This study can benefit the concrete industry by establishing new performance-based mixture proportions for the potential use of “low- w/c + low-cement + high-dosage HRWR admixture” concrete.

RESEARCH SIGNIFICANCE

The application of low-cement + high-dosage HRWR admixture concrete mixtures can be an immediate solution to address the sustainability challenge in the concrete industry. This study investigates the minimum possible cement content and the optimum dosage of HRWR admixtures in optimized concrete mixtures, considering the required fresh and hardened properties of concrete. It also quantifies the impact of the application of the recommended low-cement + high-dosage HRWR admixture concrete mixture on life-cycle costs. The outcomes of this study provide a pathway for greener, cost-effective concrete production and advancing sustainability in the construction industry.

EXPERIMENTAL PROGRAM

Materials

A Type I OPC conforming to ASTM C150-07²¹ with a specific gravity of 3.15 was used. The chemical composition of the cement was 62.54% CaO, 19.92% SiO₂, 5.48% Al₂O₃, 2.99% Fe₂O₃, 3.37% SO₃, 3.20% MgO, 1.17% K₂O, 0.27% Na₂O, and a loss on ignition (LOI) of 2.17%. Natural river sand was used as fine aggregate, with a specific gravity of 2.63, an absorption rate of 0.58%, and a fineness modulus (FM) of 2.23. Three coarse/intermediate aggregates were used: 1) No. 57 crushed limestone with a maximum size of 20.0 mm, a specific gravity of 2.69, and an absorption capacity of 0.61%; 2) No. 8 crushed limestone with a maximum size of 9.5 mm, a specific gravity of 2.79, and an absorption

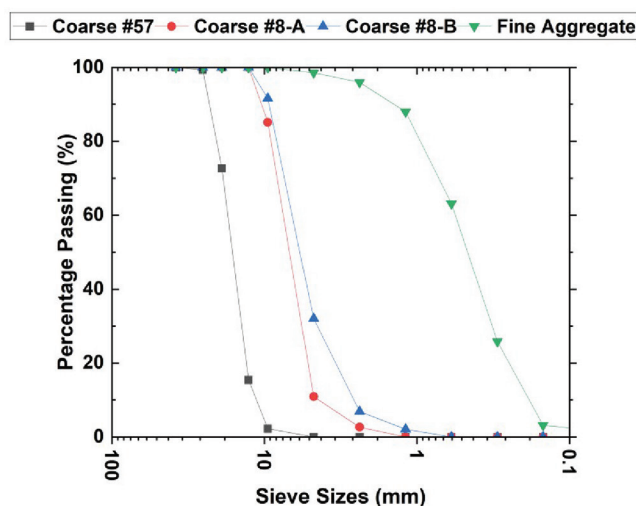


Fig. 1—Sieve analysis of fine and coarse aggregates.

capacity of 0.30% (No. 8-A); and 3) No. 8 crushed limestone with a maximum size of 9.5 mm, a specific gravity of 2.69, and an absorption capacity of 0.76% (No. 8-B). Figure 1 represents the sieve analysis for the fine and coarse aggregates. In addition, finely ground limestone with a specific gravity of 2.7 and a median particle size of 22 μ m was used as a filler to replace 5% of fine aggregate (sand) in one of the concrete mixtures. The chemical composition of the ground limestone was 94.74% CaO, 2.09% SiO₂, and 2.16% MgO. Polycarboxylate (PC)-based HRWR admixture (Types F and G according to ASTM C494/C494M-05) with a density of 1.0763 g/cm³ was used to modify the workability of the mixtures and reduce the w/c and cement content.

Mixture design

Figure 2 presents a path to achieving the sustainability target of this study (that is, a mixture with a w/c of 0.37 and a 10% reduction in cement content compared to conventional concrete with a w/c of 0.42). In Fig. 2, the water content in reference mixtures (0% HRWR) was calculated according to ACI PRC-211.1-22.⁴ As seen in Fig. 2, a 20 to 30% admixture effectiveness is required to reduce w/c and cement content by 5% and 10% (0.37 instead of 0.42), respectively, and ensure a 100 mm slump. Achieving such effectiveness would require a higher dosage of HRWR. It is worth noting that the gradation of aggregates should be optimized to ensure high packing density (that is, minimum pore space) and minimum admixture use.

Five concrete mixtures were developed with varying w/c (0.37 to 0.42) and cement content. The mixtures were designed according to ACI PRC-211.1-22⁴ requirements to achieve a target slump of 100 mm. Two concrete mixtures were used as control/reference mixtures, one of which was not optimized for aggregates (control), while the second one (optimized control mixture) had aggregates optimization using the Tarantula Curve method,^{22,23} which is a combined gradation method with four basic requirements: 1) the aggregate gradation should adhere to the specified limits for each sieve dimension; 2) a minimum of 15% of the aggregate composition should consist of coarse sand, ranging in size from No. 8 to 30; 3) the proportion of fine sand, with sizes

ranging from No. 30 to 200, should be maintained between 24 and 34% of the total volume; and 4) the quantity of flat or elongated particles in the coarse aggregate should not exceed 15%, as per the 1:3 ratio stipulated by ASTM D4791-19.²⁴ For the third mixture, the w/c was reduced from 0.42 to 0.37 while keeping the cement content constant. For the fourth and fifth mixtures, the cement content of the 0.37 w/c mixture was reduced by 5% and 10%, respectively. The 10% low-cement mixture required a 5% addition of ground limestone filler instead of fine aggregate to sustain workability.

The volumetric mixture proportions per cubic meter for the developed concrete mixtures are listed in Table 1. The quantity of materials used is given in kg/m^3 , except for the HRWR, which is stated in grams per 100 kg of cement. As stated, only the control mixture has no optimized aggregate gradation, while the rest of the four concrete mixtures have optimized aggregate gradation using the Tarantula Curve method, as shown in Fig. 3.

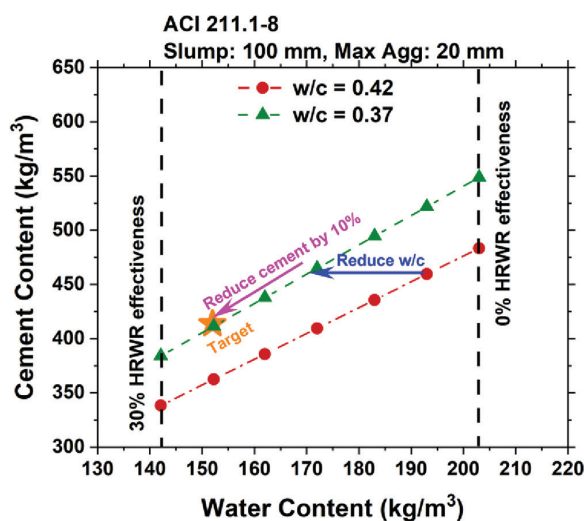


Fig. 2—Cement and water contents as function of HRWR effectiveness for concrete mixtures incorporating 20 mm coarse aggregate, and slump of 100 mm with different w/c . (Note: Water content in reference mixtures [0% HRWR] was calculated.)

Mixing procedure

For mixing, the aggregates were oven-dried and then cooled to room temperature. The mixture proportions were adjusted for moisture absorption by aggregates. The aggregates were placed into a planetary mixer and mixed for 2 minutes to achieve uniform distribution. Subsequently, the cementitious materials, water, and HRWR admixture were introduced and mixed for an additional 3 minutes. The mixture was then allowed to rest for 2 minutes, during which the sides of the mixing drum were scraped. Finally, mixing resumed for an additional 3 minutes to ensure homogeneity. A minimum volume of 0.016 m^3 of concrete was prepared for each trial batch to ensure even mixing, which was sufficient to carry out two to three workability tests, with varying dosages of HRWR, and the test for the time of setting. Cylindrical and prismatic samples were prepared for different tests.

Workability and time of setting

The workability of the developed mixtures was evaluated using the slump test method according to ASTM C143/C143M-20.²⁵ The target workability was a minimum of $100 \pm 12.5 \text{ mm}$ without segregation or bleeding.

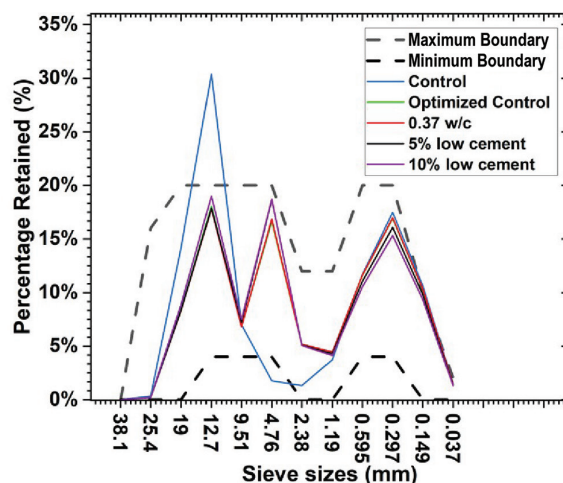


Fig. 3—Combined gradation of aggregates for different mixtures.

Table 1—Concrete mixture proportions (saturated surface-dry condition)

Material	Unit	Mixture				
		Control	Optimized control	0.37 w/c	5% low cement	10% low cement
Coarse aggregate No. 57	kg/m^3	978.9	581.4	590.3	604.6	640.7
Coarse aggregate No. 8-A		0	213.6	225.5	297.8	297.8
Coarse aggregate No. 8-B		0	219.5	225.5	207.7	207.6
Coarse aggregate combined		978.9	1014.5	1041.2	1110.0	1146.2
Fine aggregate		847.2	820.5	847.0	818.7	778.4
Cement		397.8	397.8	397.8	377.9	358.0
Limestone		0	0	0	0	40.9
Water	kg	167.1	167.1	147.2	139.8	132.5
HRWR (per 100 kg of cement)		0.35	0.35	0.63	1.03	1.09
w/c		0.42	0.42	0.37	0.37	0.37
Coarse-to-fine aggregates ratio	—	1.16	1.24	1.23	1.36	1.47

The initial and final setting times were measured for different mixtures by penetration resistance according to ASTM C403/C403M-08.²⁶ The initial and final setting times were determined as the times when the penetration resistance equals 3.44 and 27.58 MPa, respectively.

Compressive strength test

A compressive strength test was conducted for cylindrical samples (100 x 200 mm) after curing periods of 3, 7, 28, and 90 days for all concrete mixtures as per ASTM C39/C39M-23.²⁷ The samples were cured in saturated lime solution at 23°C. A compression test system was used to test samples. The samples were tested at a rate of 680 kg/min until the sample failed (that is, reached a peak load). A minimum of three samples were tested for each curing time to ensure consistency and reliability of the results, with the criterion of standard deviation less than 5%.

Flexural strength test

The modulus of rupture test, commonly known as the flexural strength test for concrete, is an established method for assessing the bending strength of a concrete specimen. This procedure entails positioning a concrete beam on two supports and applying force at the midpoint of a beam sample according to ASTM C293/C293M-16. The test measures the capacity to bear a maximal load prior to failure. ASTM C293/C293M outlines the procedural framework for executing this test. Experiments measuring flexural strength were performed on beam specimens measuring 406 x 100 x 100 mm after periods of 28 and 90 days, during which they were cured in a saturated limewater solution at 23°C. A minimum of three samples were tested for each period to ensure consistency and reliability of the results with the criterion of standard deviation less than 5%. The compression testing system was used to conduct these tests.

Length change test

The concrete length change test is a widely recognized method for assessing the dimensional stability of concrete when exposed to varying environmental conditions. This procedure is crucial for determining the tendency of concrete to shrink or expand in response to temperature fluctuations and moisture variations. It involves exposing a concrete sample to specified temperature and moisture conditions, followed by regular measurements of length change over a designated duration. ASTM C157/C157M-17²⁸ delineates the protocol for executing the length change test for concrete, and two methods are given for sample storage: 1) water storage, in which the sample is stored in a saturated lime solution; and 2) air storage, in which the sample is stored in an environmental chamber with a relative humidity (RH) of 50% while the specimens are at a temperature of 23°C.

In this study, a combination of the two sample storage methods was used by following the procedure listed in CP-L 4103-15 by the Colorado Department of Transportation (CDOT),²⁹ in which concrete prism specimens measuring 276 x 100 x 100 mm were used. Initially, these specimens underwent wet curing in limewater for a period of 7 days. Subsequently, they were placed in an environmental chamber

(RH of 50% and a temperature of 23°C) for 32 weeks. The length change measurements of the specimens were periodically taken and documented to track any variations. To ensure the reliability and consistency of the results, three replicates were prepared for each mixture to conduct the length change measurements.

Bulk electrical resistivity test and formation factor

The bulk electrical resistivity of concrete represents its capacity to resist the flow of electrical current through its pores, serving as an indirect indicator of mass transport within the concrete matrix. This property is particularly critical in scenarios where the concrete is subjected to severe conditions, such as marine environments or areas where deicing salts are prevalent. Generally, higher resistivity is indicative of enhanced resistance of the concrete against the penetration of water, chloride ions, and other deleterious substances.

The electrical resistance of cylindrical concrete samples (100 x 200 mm) was assessed after a curing period of 28 days in a saturated lime solution. This measurement was performed at a temperature of $23 \pm 2^\circ\text{C}$ using a resistivity meter operating at a frequency of 1 kHz. For this process, two stainless steel plate electrodes, each 102 mm in diameter, were employed, along with two 3 mm thick sponge pieces. Throughout the test, the sponges, saturated in lime water, were positioned between the concrete specimens and the plate electrodes to ensure consistent electrical connectivity. The electrical resistivity was then calculated by subtracting the resistance of the sponge pieces from the measured electrical resistance, following AASHTO TP 119-15.³⁰ Three replicates were tested, and the average value was reported.

The formation factor was determined using bulk electrical resistivity measurements. This parameter serves as an essential indicator of concrete transport properties and is defined as the ratio between concrete resistivity and pore solution resistivity. Additionally, the formation factor corresponds to the inverse of the product of porosity and pore connectivity. The pore solution resistivity for the mixtures with w/c of 0.37 and 0.42 was assumed to be 0.115 and 0.128 $\Omega \cdot \text{m}$, respectively, according to Qiao et al.³¹

Porosity and water absorption

After 28 days of wet curing, the cylindrical concrete specimens, each measuring 100 x 200 mm, underwent permeable pore testing, adhering to ASTM C642-21.³² However, a deviation from the standard method was employed: the specimens were saturated using a vacuum saturation chamber instead of immersion in boiling water. Additionally, the rate of water absorption was calculated in accordance with ASTM C1585-20 with two exceptions. After vacuum saturation of the samples, they were dried at 60°C until they reached a 50% degree of saturation (DOS), following which the samples were sealed for 24 hours to ensure that the absorbed solution was evenly distributed throughout the entire volume of the sample.

Packing density

The packing density of aggregates was calculated both experimentally and using theoretical modeling. The packing

Table 2—Individual aggregate properties

Aggregates	Specific gravity	Bulk density, kg/m ³	Characteristic diameter, mm	Packing density, %
Sand	2.63	1555.91	11.95	54.89
Coarse No. 8-A	2.79	1581.57	8.08	56.80
Coarse No. 8-B	2.69	1463.39	7.20	54.47
Coarse No. 57	2.69	1473.51	0.60	59.28

density was measured for the five developed concrete mixtures following the procedure listed in ASTM C29/C29M-97³³ and was also calculated using the MTM.³⁴ One advantage of the MTM is that it can be used to design aggregate blends from multiple sources by optimizing volume proportions to achieve the highest possible packing density of the combined gradation. Key aggregate properties required as inputs for the MTM include PSD, relative density, and loose bulk density, as shown in Table 2 and Fig. 1. Instead of assuming uniform spherical particles, the MTM first calculates the characteristic diameter using linear interpolation (the point at which 36.8% of particles are retained³⁵), and then the packing density for each individual aggregate is calculated using Eq. (1).^{36,37}

$$\emptyset = \frac{\rho_i}{\gamma_i \times \rho_w} \quad (1)$$

where $\emptyset$ is the packing degree of an individual material; ρ_i is the bulk density of an individual material; γ_i is the relative density of an individual material; and ρ_w is the density of water (1000 kg/m³).

After determining the packing degree and characteristic diameter of each type of aggregate, the binary blended packing density is determined using Eq. (2).^{36,37} This process is repeated until each group is taken into consideration, and the final packing density of the polysized mixture is determined.

$$\emptyset_c = \frac{1}{\left\{ \left(\frac{\gamma_1}{\emptyset_1} \right) + \left(\frac{\gamma_2}{\emptyset_2} \right) - \gamma_2 \left(\frac{1}{\emptyset_2} - 1 \right) \times K_d \times K_s \right\}} \quad (2)$$

where K_d is the diameter factor ratio = $(d_2 - d_1)/(d_1 + d_2)$; d is the combined/binary characteristic diameter = $\sqrt{d_1 \times d_2}$; d_1 and d_2 are the characteristic diameter of the fine and coarse aggregates, respectively; γ_1 and γ_2 are the volume fractions of the fine and coarse aggregates, respectively; $\emptyset_1$ and $\emptyset_2$ are the packing degree of fine and coarse aggregates, respectively; K_s is the statistical factor

$$K_s = \begin{cases} \frac{0.3881x}{0.4753}, & x < 0.4753 \\ 1 - \frac{1+4x}{(1+x)^4}, & x \geq 0.4753 \end{cases}$$

and x is the (bulk volume of the fine particles)/(void volume between the coarse particles), calculated from the following

$$x = \frac{\left(\frac{\gamma_1}{\gamma_2} \right) \times \left(\frac{\emptyset_2}{\emptyset_1} \right)}{(1 - \emptyset_2)} \quad (3)$$

LIFE-CYCLE COST ANALYSIS (LCCA)

LCCA is a method to evaluate the total cost of ownership of a project or product over its entire life cycle. In the context of concrete, LCCA involves considering costs associated with planning, design, construction, operation, maintenance, and disposal. In this research, the focus is on the LCCA components that vary among the developed concrete mixtures, such as initial costs, service life, and number of repairs during the analysis period. By considering these factors, LCCA provides a comprehensive view of the economic implications of using the developed concrete mixtures for a specific project. It helps decision-makers choose materials and construction methods that offer the best balance between initial costs and long-term benefits.

Life-cycle cost represents the total of the initial construction expenses plus the discounted costs of future repairs throughout the structure's service life (100 years in this study). Future repair costs are calculated on a "present worth" (PW) basis using the inflation rate i and the real discount rate r , according to Eq. (4).³⁸

$$PW = c \left(\frac{1+i}{1+r} \right)^t \quad (4)$$

where c is a future cost in year t .

The initial construction costs were calculated as the sum of concrete and reinforcement (1.9% volume of concrete) costs. Table 3³⁹⁻⁴² lists the cost per unit of raw material, based on which the initial cost per cubic meter of concrete was calculated for each mixture, as summarized in Table 4. A 930 m² bridge deck section having a thickness of 300 mm and reinforcement depth of 56.30 mm was taken as the example structure for performing the LCCA. The United States' long-term inflation rate of 3.28% and the discount rate of 3.00% were taken for cost calculations over a design life of 100 years, starting from the year 2021. The repair cost was assumed to be \$400.0 per m², with 10% of the total area requiring maintenance/repair.^{43,44} It was assumed that

the first repairs would be completed after corrosion initiation and a 10-year repair interval would be implemented after the first repair.⁴⁵

To estimate the service life of concrete structures, Life-365 software was used, which estimates the time to chloride-induced corrosion initiation using Fick's second law of diffusion.³⁸ The service life prediction involves assessing the rate of chloride penetration (that is, chloride diffusion), the critical chloride threshold for corrosion initiation, and the rate of corrosion propagation. Surface chloride buildup and temperature profiles were selected from the database of Life-365 for an urban highway bridge in Philadelphia, PA. The concentration of chlorides on the surface of the internal reinforcing steel that is necessary to initiate corrosion, c_b , is taken as 0.05% by weight of concrete.^{46,47} The propagation period was taken as 6 years for all concrete mixtures, which is the period from corrosion initiation (t_i) to the end of the concrete's serviceable life (that is, the point at which the first repair needs to take place). Life-365 uses Eq. (5) to predict time-dependent diffusion³⁸

$$D_t = D_{ref} \left(\frac{t_{ref}}{t} \right)^m \tag{5}$$

where D_t is the diffusion coefficient at time t ; D_{ref} is the diffusion coefficient at time t_{ref} (for example, 28 days); and m is the diffusion decay index. The m value was assumed to be

Table 3—Cost per unit for raw materials³⁹⁻⁴²

Material	Unit	Cost, \$
Coarse aggregate	Cost per kg	0.0220
Fine aggregate	Cost per kg	0.0150
OPC	Cost per kg	0.3045
Tap water	Cost per kg	0
Limestone	Cost per kg	0.1500
HRWR	Cost per kg	0.0176
Black steel	Cost per kg	0.98

Table 4—Initial cost per m³ of concrete mixtures

Concrete mixtures	Cost of concrete, \$
Control	110.5
Optimized control	111.0
0.37 w/c	117.0
5% low cement	121.6
10% low cement	122.4

Table 5—Workability and setting time of concrete mixtures

Mixture	w/c	HRWR, wt. % of cement	Slump, mm	Initial setting time, minutes	Final setting time, minutes
Control	0.42	0.35	127	290	365
Optimized control	0.42	0.35	90	260	345
0.37 w/c	0.37	0.63	102	275	345
5% low cement	0.37	1.03	114	315	400
10% low cement	0.37	1.08	130	280	370

0.27 and 0.32 for the mixtures with w/c of 0.42 and 0.37, respectively, according to Park et al.⁴⁸

In this study, D_{ref} was estimated using the calculated formation factor values from bulk electrical resistivity measurements for different mixtures. Equation (6) shows the relationship between the formation factor and the diffusion coefficient^{49,50}

$$F_{ref} = \frac{D_0}{D_{ref}} \tag{6}$$

where F_{ref} is the formation factor at time t_{ref} , and D_0 is the self-diffusion coefficient for a chloride ion (2.03×10^{-9} m²/s at 25°C).

RESULTS AND DISCUSSION

Fresh concrete properties

Workability and time of setting were the fresh properties determined for the concrete mixtures. A summary of slump test results with the required dosage of HRWR for different mixtures is listed in Table 5. The control mixture with the addition of 0.35% of HRWR (by cement weight) had a slump of 127 mm. However, the mixture was susceptible to separation due to the non-optimized aggregate gradation (Fig. 4(a)). The slump value was 90 mm in the optimized control mixture with the addition of 0.35% of HRWR. The optimized control mixture was more cohesive compared to the control mixture (Fig. 4(b)). To develop the low-w/c mixture with the required workability, the w/c was reduced from 0.42 to 0.37, and varying dosages of HRWR were examined (0.49 to 0.77% of cement weight). The mixture with the addition of 0.63% of HRWR had a slump of approximately 102 mm without any segregation issue (Fig. 4(c)). For the 5% low-cement mixture, the HRWR content was increased to 1.03% to attain a slump of 114 mm with a good consistency (Fig. 4(d)). For the 10% low-cement mixture, the HRWR content was slightly increased to 1.08% after a few trial batches to attain a slump of 130 mm with a good consistency and viscous paste (Fig. 4(e)). As discussed, the 10% low-cement mixture also required a 5% addition of ground limestone filler instead of fine aggregate to sustain workability. Therefore, the required HRWR dosage in the 10% low-cement mixture was three times higher than that of control mixtures to obtain the target slump value.

The slump loss over time was also calculated for the concrete mixtures. The slump loss was measured for 60 minutes duration with an interval of 15 minutes between each reading; so, a total of five slump readings were taken at 0, 15, 30, 45, and 60 minutes after mixing. The concrete



Fig. 4—Images captured from different mixtures after completion of mixing process: (a) control mixture; (b) optimized control mixture; (c) 0.37 w/c mixture; (d) 5% low-cement mixture; and (e) 10% low-cement mixture.

mixtures with w/c of 0.37 had an 8% slump loss at the 30-minute mark. After 60 minutes, in high-dosage HRWR mixtures, the slump was reduced by 35%, which was still in the workable range. However, the addition of workability-retaining admixture compensated for the slump loss by reducing the slump loss by 40% at the 60-minute duration.

As per ASTM C403/C403M,²⁶ the initial and final setting times are the time elapsed until the mixture has a penetration resistance of 3.44 and 27.58 MPa, respectively. A summary of the results of the time of setting is listed in Table 5. There is a slight delay in setting time with the increase in HRWR dosage when compared to the control mixture. The 10% low-cement mixture showed 30 minutes less setting time than the 5% low-cement mixture despite having a slightly higher quantity of HRWR; this reduction in setting time can be attributed to the addition of 5% limestone filler instead of sand.

Mechanical properties

A compressive strength test was performed after the specified curing period. The compressive strength of different mixtures as a function of curing time is shown in Fig. 5. As expected, the compressive strength increased for all mixtures with the increase in the curing period. At early ages, the low-w/c mixtures, particularly the 10% low-cement mixture, exhibited 40 to 60% higher compressive strength compared to the control and optimized control mixtures. The 3-day strength of concrete mixtures with reduced w/c is comparable to the 28- and 90-day compressive strengths of the control mixtures. Additionally, the 10% low-cement mixture showed approximately 10 to 20% greater compressive strength compared to the 0.37 w/c and 5% low-cement mixtures. This increase in compressive strength

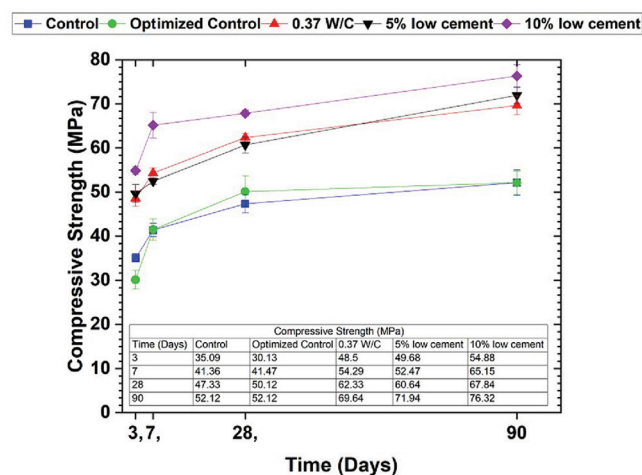


Fig. 5—Compressive strength test of concrete mixtures.

can be attributed to the higher packing density of the 10% low-cement samples due to the addition of limestone filler. At 90 days, the 10% low-cement mixture exhibited the highest compressive strength (76.32 MPa), indicating its superior long-term performance, followed by the low-w/c mixture and the 5% low-cement mixture.

The flexural strength (that is, the modulus of rupture) of the concrete mixtures was measured after 28 and 90 days of wet curing. Figure 6 shows that the low-w/c mixtures generally exhibited higher flexural strengths compared to the control mixtures. The 10% low-cement mixture showed the greatest strength at both intervals, indicating its potential for higher performance in structural applications. As mentioned, this increase can be explained by the higher packing density of the 10% low-cement samples.

Shrinkage properties

As discussed, the length change test had two phases, the wet-curing phase (for the initial 7 days) and the dry/air-curing phase (for 32 weeks). The results for the wet and dry phases are shown in Fig. 7(a) and (b), respectively. During the wet-curing phase, the control mixtures experienced higher expansion within the first few days. However, the mixtures with low w/c and low cement content displayed very limited expansion. During the dry phase, the data indicate that all concrete mixtures tend to shrink over time, with the most substantial shrinkage occurring within the first 40 days. After this period, the rate of shrinkage diminishes, and the concrete begins to stabilize, regardless of the mixture type. The control mixture experienced slightly higher initial shrinkage, while the optimized and low- w/c mixtures showed reduced shrinkage, potentially reflecting their improved composition for long-term dimensional stability. By day 240, the length changes were minimal, and the low- w/c and low-cement systems showed comparable results to the control samples.

Bulk electrical resistivity and formation factor

The bulk electrical resistivity was measured for all the concrete mixtures after 28 days of curing following the method prescribed in AASHTO TP 119.³⁰ The bulk electrical resistivity of the concrete mixtures is shown in Fig. 8. After 28 days of curing, the concrete mixtures with lower w/c and reduced cement content demonstrated higher bulk

electrical resistivity. Particularly, the mixture with 10% low cement had approximately 20% greater electrical resistivity than the 0.37 w/c mixture. This suggests that these mixtures have lower porosity and permeability and are more durable against the intrusion of deleterious substances such as chloride ions. More data on porosity and permeability of the concrete mixtures are demonstrated in the succeeding sections.

The electrical resistivity measurement is highly susceptible to pore solution resistivity. Therefore, the formation factor of the samples was determined from the bulk electrical resistivity measurements, as shown in Fig. 9. It is noticeable that the low- w/c + low-cement mixtures have a significantly higher formation factor (≈ 68 to 97% higher) compared to the control mixtures, which is in agreement with the bulk electrical resistivity measurements. As previously discussed, the formation factor has an inverse relationship with the product of porosity and pore connectivity. Thus, the observed increase in formation factor for the low- w/c + low-cement mixtures is likely due to their lower porosity and the enhanced tortuosity of their pore network.

Porosity and water absorption

Figure 10 shows the porosity of various concrete mixtures after 28 days of wet curing. The optimized control mixture had a slightly lower porosity (11.63%) compared to the control mixture. This suggests that even though the aggregate gradation was optimized, this mixture still maintained a significant level of void spaces due to a high w/c . The concrete mixtures designed with a lower w/c and reductions in cement content result in decreased porosity. The 10% low-cement mixture exhibited the lowest porosity (13 to 26% lower porosity compared to other mixtures) after 28 days. This low porosity can be attributed to the higher packing density of the 10% low-cement samples. The porosity data are in agreement with the formation factor measurements. Lower porosity in concrete is generally associated with improved durability and strength, indicating that the optimized mixtures with reduced w/c and cement content translate to enhanced material properties.

The water absorption for concrete mixtures is depicted in Fig. 11, with initial absorption readings taken after 1, 5, 10, 20, 30, and 60 minutes, followed by hourly readings until the eighth hour, and the secondary absorption readings taken every 24 hours for 9 consecutive days. The initial rate of

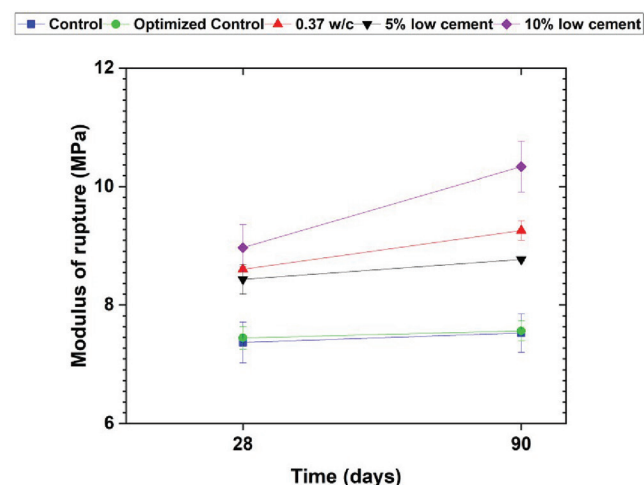


Fig. 6—Flexural strength test of concrete mixtures.

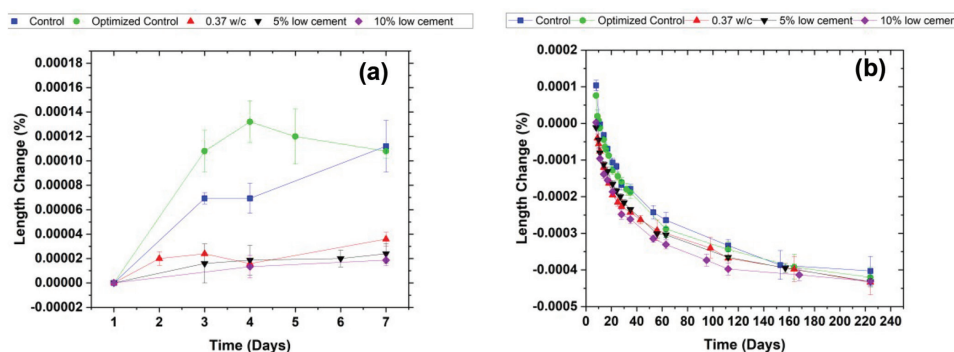


Fig. 7—(a) Wet-curing phase; and (b) dry/air-curing phase of length change measurements.

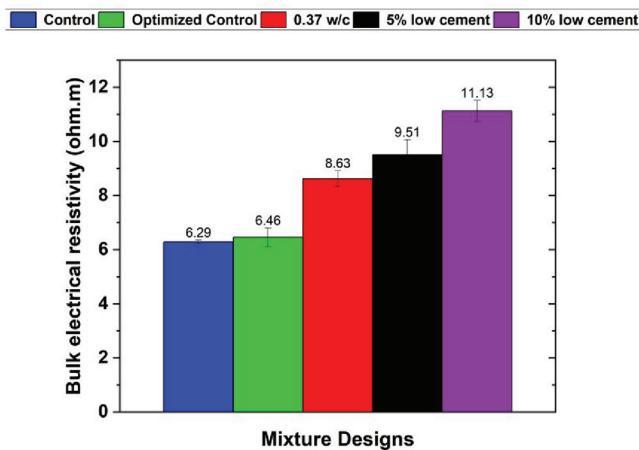


Fig. 8—Bulk electrical resistivity of concrete mixtures after 28 days of curing.

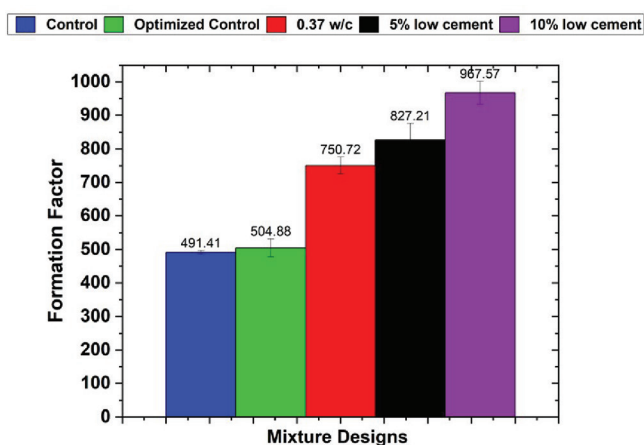


Fig. 9—Formation factor of concrete mixtures after 28 days of curing.

absorption is the slope of the line joining the initial readings until 8 hours, while the secondary absorption rate is represented by the slope of the line joining the readings from day 1 until day 9. Table 6 lists the initial and secondary rates of absorption for different concrete mixtures.

Figure 11 indicates that the low- w/c + low-cement systems show lower permeability due to a dense matrix. This can be attributed to the higher packing density of the low-cement mixtures. Additionally, in the 10% low-cement mixture, limestone particles can act as nucleation points, accelerating the hydration process (that is, refined pores). Further, an increase in HRWR dosage generally leads to an improvement in cement hydration by enhancing the dispersion of cement particles. The absorption results align closely with the findings from the formation factor and porosity tests. As indicated in Table 6, the mixture with 10% reduced cement exhibited an initial absorption rate approximately 25 to 45% lower than the other mixtures. Conversely, the control mixtures demonstrated lower secondary absorption rates. This is because water reached the top surface of the control samples after 24 hours, and the absorption curve plateaued (that is, the samples were mostly saturated), while the low- w/c + low-cement samples continued to absorb moisture due to lower initial absorption rates. The lower

Table 6—Initial and final rates of absorption of concrete

Mixtures	Initial rate of absorption, mm/ $\sqrt{s}$	Secondary rate of absorption, mm/ $\sqrt{s}$
Control	0.0138	2.166×10^{-4}
Optimized control	0.0127	2.113×10^{-4}
0.37 w/c	0.0105	5.033×10^{-4}
5% low cement	0.0099	4.414×10^{-4}
10% low cement	0.0077	4.739×10^{-4}

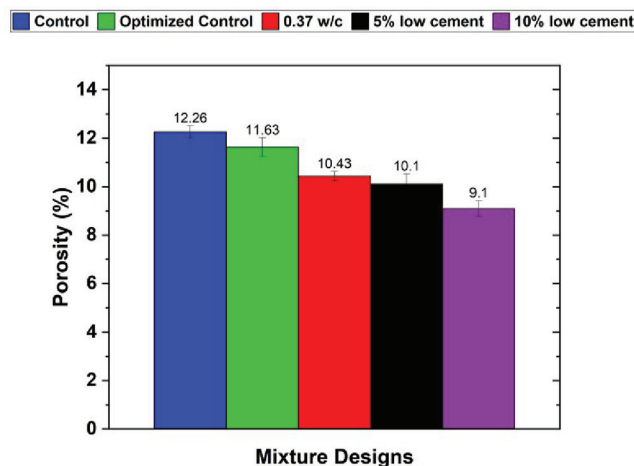


Fig. 10—Porosity of concrete mixtures after 28 days of curing.

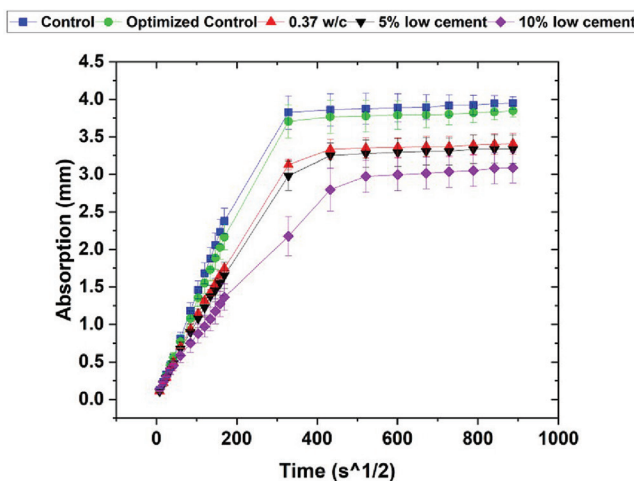


Fig. 11—Water absorption of concrete mixtures after 28 days of curing.

absorption can lead to more durable concrete in severe exposure conditions such as cold environments.

Packing density

A comparison between the measured and calculated packing densities of the concrete mixtures is given in Fig. 12. The experimental packing density, calculated based on ASTM C29/C29M,³³ is in line with the packing density calculated using the MTM. For each mixture, the experimental results closely match the values predicted by the model, which suggests that the MTM is a reliable method for

estimating the packing density of these concrete mixtures. The optimized control mixture showed a slight increase in packing density compared to the control mixture, indicating a better arrangement of particles within the mixture to minimize the void space. The 10% low-cement mixture exhibited the highest packing density among the mixtures, exceeding 74% in both the model and experimental results, which implies an optimal mixture design with minimal void space, potentially leading to higher strength and durability properties. Besides the aggregate optimization, the higher packing density of the 10% low-cement mixture can also be attributed to the replacement of 5% fine aggregates with limestone filler.

Service life prediction and LCCA

The chloride diffusion inputs are listed in Table 7 along with the predicted service life for different mixtures using Life-365. The concrete mixture with the lowest cement content (10% low-cement mixture) has the highest service life of 66.5 years, which is increased by 117% with respect to the control mixture, and by 25% compared to the 0.37 *w/c* mixture.

Based on LCCA, the life-cycle cost was estimated for each concrete mixture, and the cumulative present value of all concrete mixtures is shown in Figure 13. The concrete mixture with a 10% reduction in cement was estimated to be 29% more economical when compared to the control mixture. A summary of the LCCA inputs and outputs has

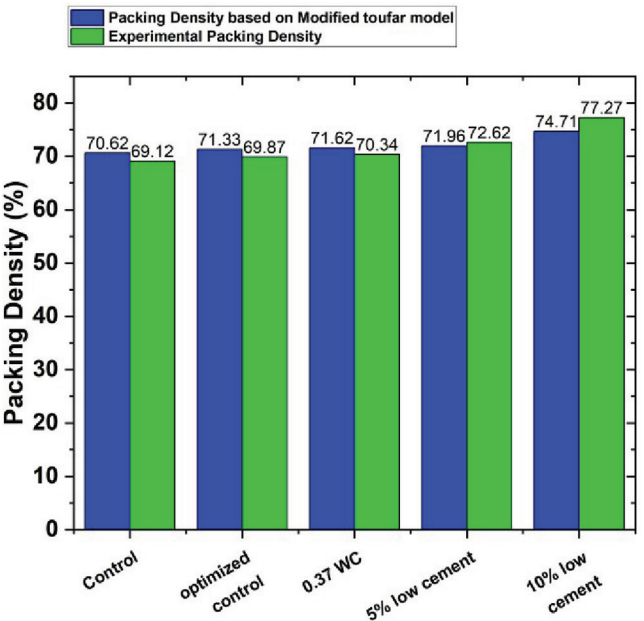


Fig. 12—Packing density of concrete mixtures.

Table 7—Service life prediction of concrete mixtures

Concrete mixtures	$D_{28} \times 10^{-6}, \text{mm}^2/\text{s}$	m	Corrosion initiation, years	Propagation, years	Service life, years	% increase in service life
Control	4.13	0.27	24.6	6	30.6	0%
Optimized control	4.02	0.27	25.2	6	31.2	2%
0.37 <i>w/c</i>	2.70	0.32	47.2	6	53.2	74%
5% low cement	2.45	0.32	51.8	6	57.8	89%
10% low cement	2.10	0.32	60.5	6	66.5	117%

been given in Table 8 for the costs of a 930 m² section of a bridge deck in Philadelphia. The concrete mixture with a 10% reduction in cement has the lowest life-cycle cost of \$286,541, which is approximately 30% lower than the control mixtures. This analysis shows that while the application of low-cement + high-dosage HRWR admixture mixtures slightly increases the initial costs, it considerably reduces the life-cycle costs by improving the mechanical and durability properties of concrete. The application of low-cement + high-dosage HRWR admixture mixtures can improve the sustainability of concrete mixtures by reducing cement and water contents and increasing the service life of concrete in severe environments.

CONCLUSIONS

This study investigated the impact of applying “low-cement + high-dosage HRWR admixture” mixtures on fresh and hardened properties and life-cycle costs of concrete. Five concrete mixtures with varying water-cement ratio (*w/c*), aggregate gradation, cement content, and high-range water-reducing (HRWR) admixture dosage were developed. A suite of experimental methods was applied to determine the fresh, mechanical, and durability properties of the mixtures, and to predict the service life of concrete in a corrosive environment. Life-cycle cost analysis (LCCA) was conducted to estimate the life-cycle costs of low-cement + high-dosage HRWR admixture mixtures. The following conclusions can be drawn:

- The optimized mixture with 10% reduced cement and low *w/c* required three times greater HRWR dosage to satisfy the required workability. In high-dosage HRWR

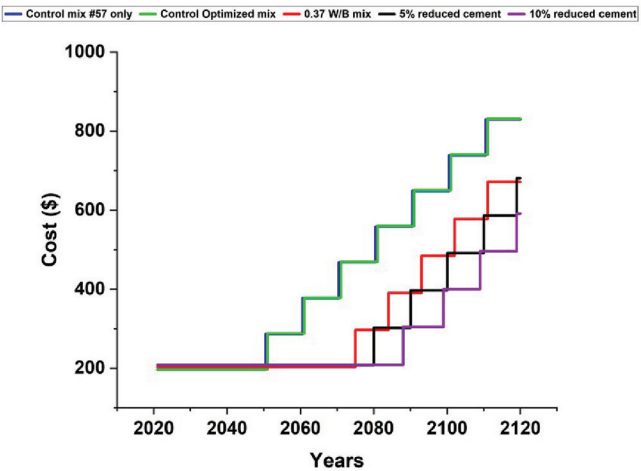


Fig. 13—Cost per cubic meter of concrete mixture over life of structure.

Table 8—LCCA inputs and outputs of 930 m² section of bridge deck in Philadelphia

Mixture	Control	Optimized control	0.37 w/c	5% low cement	10% low cement
w/c	0.42	0.42	0.37	0.37	0.37
Steel type	Black steel				
Steel %	1.9				
Service life, years	30.6	31.2	53.2	57.8	66.5
Initial mixture cost, \$/m ³	110.5	111.0	117.0	121.6	122.4
Repair interval, years	10				
Initial cost, \$	95,249	95,578	98,549	100,719	101,252
Life-cycle cost, \$	401,846	403,009	325,246	329,892	286,541

mixtures, the slump was reduced by 35% 1 hour after mixing. However, the addition of workability-retaining admixture compensated for this slump loss.

- Optimizing the aggregate gradation in the concrete mixtures resulted in a more cohesive mixture, reducing the susceptibility to segregation.
- The time of setting of sustainable low-cement systems was comparable to that of the control mixture.
- The developed sustainable low-cement systems show superior early-age and long-term mechanical performance (30 to 50% increase in compressive and flexural strength), indicating the efficacy of mixture design optimization for structural applications.
- The amount of shrinkage in the low-w/c + low-cement mixture was comparable to that of the control samples.
- The developed sustainable low-cement mixtures showed a dense matrix (13 to 26% lower porosity) and low permeability (≈ 68 to 97% higher formation factor and 25 to 45% lower initial water absorption rate), which can improve the durability of concrete structures in harsh environments. This can be attributed to the higher packing density of the low-cement mixtures. Additionally, in the 10% low-cement mixture, limestone particles can act as nucleation points, accelerating the hydration process (that is, refined pores). Further, an increase in HRWR dosage generally leads to an improvement in cement hydration by enhancing the dispersion of cement particles.
- The optimized mixture with 10% reduced cement exhibited the highest packing density among the mixtures, exceeding 74% using both the modified Toufar model (MTM) and experiments, which implies an optimal mixture design with minimal void space. This high packing density is partially attributed to the addition of limestone filler to replace 5% of fine aggregates.
- The estimated service life due to chloride-induced corrosion for the optimized low-cement system was 117% greater than that of the control mixture.
- The concrete mixture with a 10% reduction in cement content and three times greater HRWR dosage had the lowest life-cycle costs (29% lower than the control mixtures).

Based on this study, the developed optimized low-cement concrete mixtures can be a viable immediate solution to reduce CO₂ emissions from cement and concrete production. These conclusions highlight the successful application

of low-cement + high-dosage HRWR admixture concrete mixtures to improve the strength and durability of concrete in different environments. The application of low-cement + high-dosage HRWR admixture mixtures can improve the sustainability of concrete mixtures by reducing cement and water contents and increasing the service life of concrete in severe environments.

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Effect of Silica Fume with Crystallizing Admixture on Self-Healing Engineered Cementitious Composite

by Hinoel Ehrenbring, Fernanda Pacheco, Roberto Christ, and Bernardo Tutikian

This study aimed to evaluate the effect of isolated silica fume (SF) and SF combined with three contents of crystallizing admixture (CA) in the self-healing of engineered cementitious composites (ECCs) with different polymeric fibers. Self-healing was evaluated in coupon specimens subjected to bending to produce cracking. Healing products were evaluated in the cracks within 84 days. Exposure conditions for self-healing were water-saturated (SAT) and wetting-and-drying (WD) cycles. The results showed that the composites with isolated SF presented a continuous layer of healing product, covering widths of up to 100 μm . The final widths for these composites were 40 μm for different conditions. In composites with CA, the volume of product generated (gel) was considerably greater, causing it to leak out of the microcracks existing in the ECC, impairing healing. Thus, the results showed that the use of SF + CA reduced the ECC healing potential. Healing from the CA was spot-wise only, decreasing its healing potential. The performance of the crystallizing additive was impaired under WD conditions. Leaching was observed both under SAT and WD exposure conditions. More leaching was observed from WD, while SAT formed a more uniform product layer.

Keywords: crystallizing admixture (CA); engineered cementitious composite (ECC); polymer fibers; self-healing; silica fume (SF).

INTRODUCTION

An engineered cementitious composite (ECC) is a ductile composite with cement, high pozzolan content, and non-continuous polymer fibers. It has a high threshold of strain, usually above 3%, and tensile strength close to 15 MPa.¹ The mechanical behavior of an ECC is characterized by high strain and deflection rates, which produce several microcracks with widths of less than 100 μm .² Compared to reinforced concrete, ECCs can increase strength and deformability as microcracks form in a controllable fashion with no loss to the structure's durability.³

The ductile behavior of an ECC is a consequence of the interaction between the matrix and polymer microfibers. The most common fiber used in ECCs is polyvinyl alcohol (PVA), but other polymers such as polypropylene (PP), polyethylene (PE), and polyester (POL) can also produce ductile behavior. However, fiber content must be adjusted for the reinforcements to produce the desired level of ductility in ECCs, mainly due to the properties of the reinforcement and the number of fibers present in the element cross section.²

A limited number of studies were conducted concerning the degradation of steel in different cracked cementitious composites.⁴ In ECCs, cracks more than 100 μm wide substantially increased the degradation of longitudinal reinforcements.⁵ However, Djerbi et al.⁶ determined that the diffusion of chlorides in water-filled cracks had a linear

behavior with widths up to 100 μm , while degradation was negligible in cracks less than 50 μm wide.⁷ Nonetheless, crack widths between 50 and 100 μm substantially increased the probability of ingress of aggressive agents with negative impacts on the structure's durability.⁸

An ECC is a multipurpose cementitious composite because, besides its mechanical properties, it is also able to self-heal.⁹ Healing can occur with partial or complete closure of microcracks, which extends the useful service life of the structure.¹⁰ In general, cementitious composites can fill pores and cracks.¹¹ However, without fibers, crack formation is unstable, with diverse geometric shapes and variations in width, which decreases healing potential. In addition to decreased healing capacity, such irregular cracks become preferential pathways to the ingress of aggressive agents.¹²

Two methods can characterize self-healing: autogenous and autonomous.¹³ Autogenous self-healing occurs through agents with multiple functions in the cementitious matrix, such as pozzolans,¹⁴ while autonomous self-healing relies on agents specific to healing.^{15,16}

In ECCs, restricted crack widths (<100 μm) and elevated content of cement and pozzolans are appropriate conditions for autogenous self-healing. An ECC matrix could contain up to 70% of the total volume in cement and pozzolans,¹⁰ while the water-agglomerate ratio could remain between 0.22 and 0.30. Consequently, a substantial amount of anhydrous cement and pozzolan particles do not react initially within the composite.¹⁷ To the ECC production, the pozzolanic materials most frequently applied are bottom ash and furnace slag. Muller et al.¹⁸ noted that as the pozzolanic activity improved, cracks up to 100 μm in width could be healed.

As an alternative to conventional coproducts, one possibility is to apply cementitious complementary materials, such as silica fume (SF). SF performance when applied in ultra-high-performance concrete has been under investigation.¹⁹ However, a few studies evaluate the development of ECC with SF. Due to its high pozzolanic activity, SF can reduce cement consumption and accelerate the ECC self-healing process.

Once ECC can be applied to structures in contact with water, it is needed to make the system watertight. Thus, crystallizing admixtures (CAs) are used in the matrix.²⁰ In

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addition to protecting the system from water, those admixtures provide the ability to seal cracks, which is considered autonomous healing.

Self-healing properties can be further enhanced with crystalline catalysts with autonomous properties.²¹ This would make it possible to heal cracks and pores in the matrix.²² Because ECC matrixes concentrate large amounts of pozzolans, as SF, it is essential to understand the interaction and behavior of both agents (SF and CA) when used in conjunction.

Li and Herbert²³ defined the criteria for sturdy healing in a cementitious composite as preservation of precipitated product, dispersion in the matrix, quality, versatility, and repeatability of occurrences. The quality of healing could be evaluated visually (with microscopes), chemically (energy-dispersive X-ray spectroscopy [EDS]), physically (permeability or ultrasonography), and mechanically (tensile strength and modulus of elasticity).

Environmental conditions of the composite could also affect the healing results. Few results were available on the sturdiness of healing products in an ECC, especially under realistic conditions such as wetting-and-drying (WD) cycles.²⁴ As noted by Li,⁷ WD cycles better represent the real structure's conditions.

To validate the healing potential of ECCs, cracks must be induced in mechanical tests. Li⁷ noted that, in addition to cracking through tensile stress, coupon tests on plates could be performed to evaluate the healing potential of ECCs better, recommending a two-step validation procedure for healing with visual and chemical analyses.

Thus, the objective of this study was to evaluate the efficiency of SF and CA (varied contents) in the healing potential of ECCs reinforced with PVA, POL, and PP fibers

(coupon specimens), when curing conditions were 77 days water-saturated (SAT) or exposure to WD.

RESEARCH SIGNIFICANCE

In this paper, the authors evaluated the effect of isolated SF combined with a CA in the self-healing of ECCs. The results of this research provide valuable confirmations, indicating that the healing of cracks in ECCs may be impaired when there is a combination of supplementary cementitious materials and crystallizing additives. Also, the exposure conditions of these composites directly affect the results, and in the WD cycles the healing potential of ECCs decreased.

EXPERIMENTAL PROCEDURE

The testing methodology was organized in two stages, as shown in the flowchart of Fig. 1. Stage 1 consisted of inducing cracks after 7 days of curing. Stage 2 determined the healing potential of the cracks under two curing conditions: SAT and WD. These analyses started on the cracking age (seventh day) and lasted until the 77th day (thus, specimens were aged 7 + 77 days in total). The cracking age was defined considering the days after curing when cementitious composites are more susceptible to cracking, in accordance with Cuenca et al.²⁵ and Gupta et al.²⁶ Table 1 presents the experimental plan with the specimens, compositions, and corresponding specimen number.

The mixture ratios and fiber content were selected based on the results of Ehrenbring et al.² Thus, the fiber contents used were chosen due to the similar tensile strength values, as indicated in the research by Ehrenbring et al.² Therefore, the variables inherent to the ECC are reduced, and the evaluation was focused on the width of the cracks, healing conditions, and healing agents.

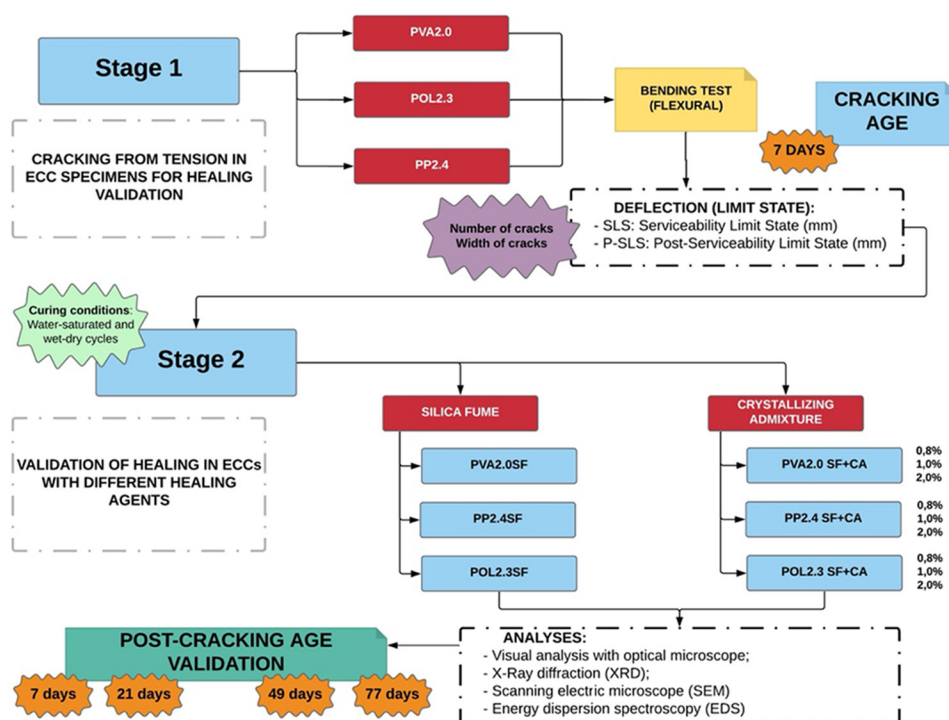


Fig. 1—Flowchart of stages of study.

Table 1—ECC composites and contents used in this study

Composite	Parameters						No. of specimens	Properties evaluated
	Fiber	V_f , %	SH_A	M_{SHA} , %				
ECC	PVA	2.0	SF	—	—	—	8	w_{lf} $w_{ave,initial}$ $w_{ave,final}$ AVE_{heal}
			SF+CA__	<u>0.8</u>	<u>1.0</u>	<u>2.0</u>	24	
	POL	2.3	SF	—	—	—	8	
			SF+CA__	<u>0.8</u>	<u>1.0</u>	<u>2.0</u>	24	
	PP	2.4	SF	—	—	—	8	
			SF+CA__	<u>0.8</u>	<u>1.0</u>	<u>2.0</u>	24	

Note: V_f is fiber content by volume; SH_A is self-healing agent; M_{SHA} is self-healing agent content by mass of cement; T1, T2, and T3 are CA content of 0.8%, 1.0%, and 2.0%, respectively; w_{lf} is crack width in the coupon specimen; $w_{ave,initial}$ is initial average crack width at formation; $w_{ave,final}$ is final average crack width (at 77 days); AVE_{heal} is average healing factor (at 77 days).

Table 2—Properties and characteristics of polymer reinforcements

Property	Reinforcement		
	POL	PVA	PP
Diameter, μm	36	40	15
Length, mm	12	12	12
Aspect ratio	315	300	800
Tensile strength, MPa	700	1600	400
Modulus of elasticity, GPa	8.0	41	5.0
Elongation, %	11.5	6.0	25.0
Density, g/cm^3	1.40	1.30	0.91

Materials

The portland cement Type III (high early strength) was used without pozzolanic materials. The cement was classified by the standard ASTM C150/C150M-21²⁷ and had a specific mass of 3.005 g/cm^3 and specific surface area obtained from the BET method of 2.0474 m^2/g . The SF used in this study was sourced from metallic or ferrous silicon manufacturing. It was densified to a specific mass of 2.220 g/cm^3 and a specific surface area of 27.5332 m^2/g . Fine aggregate was natural fine river sand with maximum dimension of 0.6 mm and a fineness modulus of 1.18. The apparent and specific mass of the aggregate were 1.58 g/cm^3 and 2.63 g/cm^3 , respectively. The chemical high-range water-reducing admixture was polycarboxylate-based with a 42% solid suspension content and density of 1.12 g/cm^3 .

Different healing agents were selected for the study: autogenous and autonomous, the latter added with varying contents. SF was used as the autogenous agent, while the powdered CA was selected as the autonomous agent in the mixing process. The SF content was 80%, concerning the mass of cement. The CA contents were 0.8, 1.0, and 2.0% concerning the mass of cement.

Three types of polymer fibers were used: PVA, POL, and PP. The properties and characteristics of each reinforcement fiber are shown in Table 2. The mechanical properties were sourced from the suppliers.

Material proportions

The dosage used in this study was based on the recommendations of Ehrenbring et al.² For a standard ECC matrix, the

Table 3—Materials proportion for ECC matrixes

Material	Proportion	Quantity, kg/m^3
Cement	1.0	618
Silica fume	0.8	495
Fine sand	1.2	742
Water	0.29	323
Fibers*	—	—
High-range water-reducing admixture	0.03	33.4
Crystallizing admixture	0.008	4.95
	0.01	6.19
	0.02	12.38

*PVA = 2.0% by volume; POL = 2.3% by volume; PP = 2.4% by volume.

cement, SF, and fine sand mixture ratio was 1.00:0.80:1.20 by mass. The water-agglomerate ratio was set at 0.29 and high-range water-reducing admixture additive content was 3% with respect to the mass of agglomerates. Table 3 shows the mixture ratio and quantity of materials of the matrix. The contents of each polymer fiber are also shown in Table 3.

Tensile strength

Tensile strength was performed considering the four-point bending test according to JSCE 82,²⁸ which defines the load per prescribed displacement equivalent to 0.5 mm/min. The samples applied were 70 x 20 x 400 mm (width, thickness, and length), with a span of 300 mm. In the center of the theoretical span of the plates, a linear variable differential transformer (LVDT) was instrumented to measure the vertical displacement (deflection).

Cracking states

Cracking of the ECC coupon specimens was performed with a four-point bending test. Two states of cracking were used in the self-healing analysis: the serviceability limit state (SLS) and the post-serviceability limit state (P-SLS). The SLS was related to the deflection (δ_{SLS}) of structural elements that still maintained user comfort and functionality in the system. This was defined as 0.4% of the span length, or 1.2 mm. The P-SLS was related to rupture deflection. This was defined as a deflection (δ_{P-SLS}) equivalent to 80% of the maximum deflection limit of the composites (rupture).

Cracking age and exposure conditions for healing

The age selected to induce cracking was after 7 days of curing at a temperature of $23 \pm 2^\circ\text{C}$ and relative humidity of $98 \pm 2\%$. After cracking, the width (w_{ij}) and the number of cracks (n_{ij}) were determined for each specimen. Two healing exposure conditions were applied for curing: SAT and WD cycles. Under SAT conditions, specimens were submerged in water at $23 \pm 2^\circ\text{C}$ and kept for 77 days after cracking with replacement of water every 7 days. The WD cycles were used to simulate the real material's conditions of rain and dry weather. Precipitation data from the city of Porto Alegre, Brazil, were used to determine a WD cycle of 4 days, consisting of 1 day of wetting followed by 3 days of drying. The temperature during the WD cycles was kept at $23 \pm 2^\circ\text{C}$ with $55 \pm 5\%$ relative humidity. At each cycle, the water in the reservoir was changed to avoid cross-contamination between the test specimens.

Average healing factor (AVE_{heal}) and mineral analysis

The average healing factor (AVE_{heal}) was determined from images taken from a $20\times$ optical microscope. Digital images were taken, and crack widths were measured with computer software (ImageJ). Crack widths at the crack formation and after 77 days of exposure were measured at 20 points along the length of all cracks in the coupon specimens to calculate the average values of initial average crack width (w_{ave}) and final average crack width ($w_{ave,final}$). The average healing factor (AVE_{heal}) was then calculated from Eq. (1) from Wiktor and Jonkers.²⁹

$$AVE_{heal} = \frac{w_{ave} - w_{ave,final}}{w_{ave}} \times 100 \quad (1)$$

The average residual crack width ($w_{ave,res}$) was then calculated from Eq. (2).

$$w_{ave,res} = w_{ave} - w_{ave,final} \quad (2)$$

Healing products formed within the cracks were evaluated with EDS, scanning electron microscopy (SEM), and X-ray diffraction (XRD). Evaluations were conducted 77 days after cracking and XRD specimens were collected from within the cracks with a sterile metallic scraper.

For mineralogical analysis using XRD, healing products were extracted from within the fissures using a sterilized metal needle. Care was taken not to remove material from the fissure walls, avoiding contamination of the cementitious matrix compounds. The amount of material removed per sample was 5 g. The powdery material was ground to the required fineness using an agate grinder.

For EDS and SEM tests, cylindrical cores were extracted without the use of water from the coupon specimens in the fissure region. The cores were 10 mm in diameter and 15 mm in height. The cores had their surfaces treated with gold, as required for the technique.

EXPERIMENTAL RESULTS AND DISCUSSION

Tensile strength

Figure 2 shows the tensile strength results from the composites. It can be noticed that all of them showed ductile behavior, increasing deformation and cracking (stress drops along the curve) as there is a gain in stress. The results refer to the age of 7 days because the stresses served to support the cracking rate.

It is possible to notice that the addition of the CAs did not affect the ductile behavior of the composites, maintaining a cracking index, tensile strength, and deflection like composites with only SF. This behavior may be related to the inactivity of the additive until the test date, acting as a filler. As the percentage added is low, it did not change the granular structure of the composite and showed no impact on the mechanical properties.

The maximum tensile strength was 7.8 MPa for ECCPOL, followed by ECCPVA with 6.9 MPa. ECCPP, on the other hand, reached a resistance of 6.5 MPa. The stress in the first crack, for all samples, remained between 4 and 5 MPa. The results presented were already expected, agreeing with Ehrenbring et al.² Both POL and PP fibers have the potential for the development of ECCs to increase the number of options in relation to PVA fibers.

Sample cracking induction

Table 4 presents the results obtained for each sample after the bending test (to induce the cracks).

It was possible to observe that the total number of cracks formed in the samples of each composite was similar in the SLS stage due to the low stress rate generated by the limited deflection. Authors such as Alyousif,³⁰ Lu et al.,³¹ and Zhou et al.³² have already presented results similar to those for low levels of strain. The contribution of reinforcements is more evident in the P-SLS stage, with an increase in deflection levels that generated a substantial increase in the number of cracks.

In the P-SLS, the crack average thickness also increased because the polymeric fibers underwent elongation inside the opening, as they act as tension transfer bridges, mainly those of PP and POL. With this elongation, the reinforcements presented residual deformations, making it impossible to return to the initial elongation. The more elongated

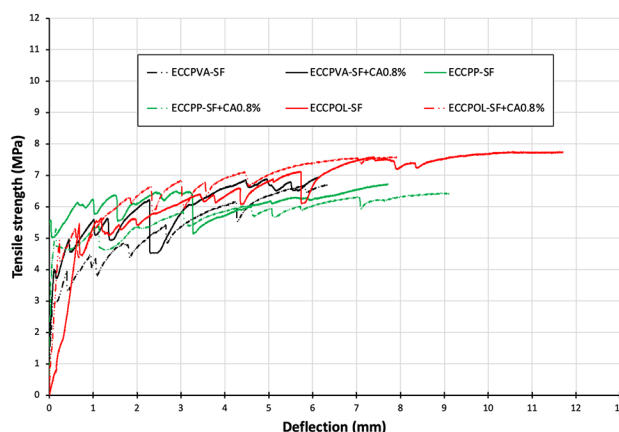


Fig. 2—Behavior curves of ECCs in four-point bending test.

the fiber is, as in composites containing PP, the smaller the number of cracks formed and the greater the average thickness of these gaps.

According to Tosun-Felekoğlu et al.,³³ Kewalramani et al.,³⁴ and Lepech and Li,³⁵ the average thickness of cracks in ECCs should remain between 30 and 100 μm . However, variations of up to 20% above the limit are allowed. Given these considerations, it appears that practically all samples remained with thickness within the limits recommended in the literature. It is important to emphasize the similarity between the composites containing PVA fibers, which are more rigid, and recycled POL fibers, which are more elongated. Both the number of cracks and the average thickness were similar, showing the potential of the POL fibers, as presented by Ehrenbring et al.²

Healing with isolated SF

Curing immersed in SAT—Figure 3 presents the results of AVE_{heal} and w_{ave} for composites with isolated SF curing from immersion in SAT.

Results show that AVE_{heal} varied between SLS and P-SLS and a similar trend followed for w_{ave} . The higher values of AVE_{heal} for each composite occurred for SLS and smaller

w_{ave} . Under SLS conditions, w_{ave} varied between 60 μm and 90 μm for corresponding AVE_{heal} between 100% and 84%, respectively. Additionally, Sahmaran et al.³⁶ verified that AVE_{heal} for ECCs immersed in water was 85% for crack widths of up to 100 μm .

In the case of P-SLS, healing was affected by the increase in w_{ave} to a range between 97 and 122 μm , which resulted in AVE_{heal} between 88% and 58%, respectively. Similar results were reported by Yıldırım et al.³⁷ with a reduction in healing factor to 60% for crack widths between 100 and 200 μm , which resembled the results of composites ECCPOL-SF and ECCPP-SF of this study. More stringent results were reported by Yang et al.³⁸ with ECC healing factors between 20 and 50% in cracks larger than 100 μm .

For the values of AVE_{heal} obtained on ECCPVA-SF, it may be caused by the chemical reactions from the PVA fibers as mechanisms to achieve the crack healing, besides the low w_{ave} . Fan and Li³⁹ showed the start of the healing process around PVA fibers, besides the crack walls. According to the authors, the hydrophilic surface from the fibers allowed the formation and precipitation of $\text{Ca}(\text{OH})_2$ around it. Thus, the products were not only on the crack wall and surface, but also around the fibers. In this research, the differences in AVE_{heal} were up to 25% between the composites, considering similar values of w_{ave} .

Figure 4 shows the indication of the formation of healing products on the PVA fiber. The presence of precipitate around the fiber can be noticed, in addition to there being points with product development, which corroborates the hypothesis based on Fan and Li.³⁹ Thus, on PVA2.0SF, the healing process through the pozzolanic reaction happened in different areas (walls, bottom, and PVA fiber), which resulted in an elevated AVE_{heal} .

The visual differences in healing for composite ECCPOL-SF under P-SLS conditions are shown in the optical microscope images of Fig. 5.

Results show distinct AVE_{heal} for Fig. 5(b) and (c) despite being from the same specimen. Figure 5(b) contained two cracks: the left crack had a width of 25 μm and achieved an AVE_{heal} of 100%, while the right crack had a width of 96 μm and achieved an AVE_{heal} of 79%. Similar results could be seen in Fig. 5(c) in that crack width directly affected the level of healing that could be achieved. The volume of healing products produced in each crack was relatively constant but,

Table 4—Number of cracking (η_{tf}) and cracking thickness (e_{tf}) after bending test

Composition	SLS		P-SLS	
	η_{tf}	$e_{\text{tf,meds}}$ mm	η_{tf}	$e_{\text{tf,meds}}$ mm
ECCPVA-SF	4	59	12	65
ECCPOL-SF	4	71	10	80
ECCPP-SF	3	93	15	129
ECCPVA-SF+CA0.8%	3	45	23	82
ECCPOL-SF+CA0.8%	4	51	19	114
ECCPP-SF+CA0.8%	3	114	11	160
ECCPVA-SF+CA1.0%	3	52	28	120
ECCPOL-SF+CA1.0%	4	62	27	112
ECCPP-SF+CA1.0%	4	115	10	193
ECCPVA-SF+CA2.0%	5	81	26	114
ECCPOL-SF+CA2.0%	3	67	20	120
ECCPP-SF+CA2.0%	3	116	12	178

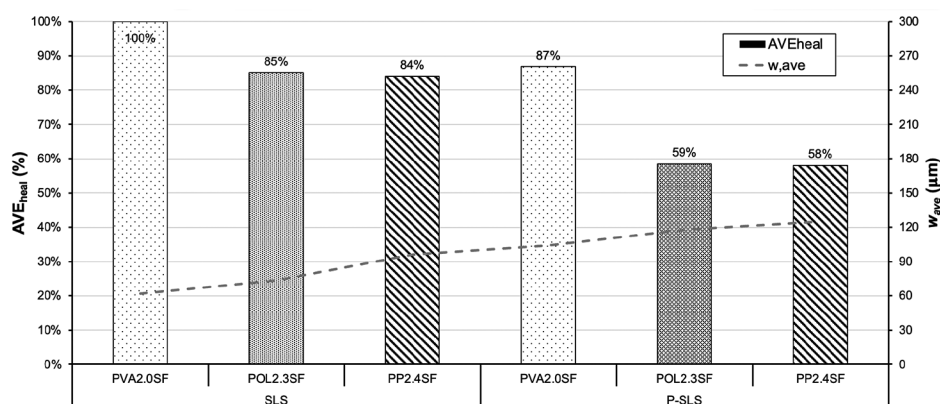


Fig. 3—Healing results for composites with SF as healing agent and curing immersed in water (SAT).

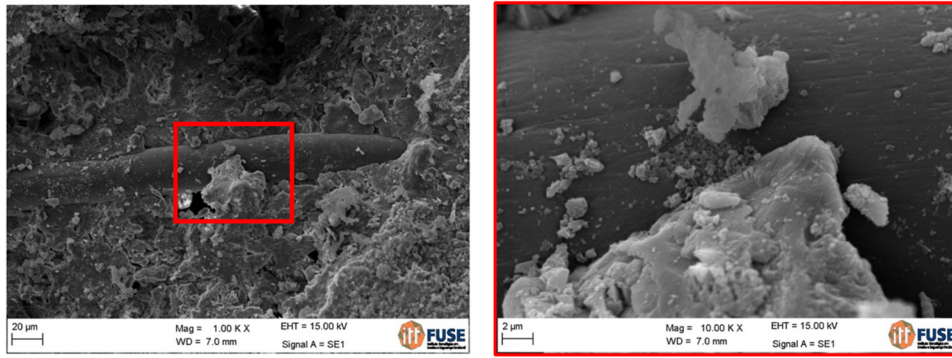


Fig. 4—Healing product on surface of PVA fiber.

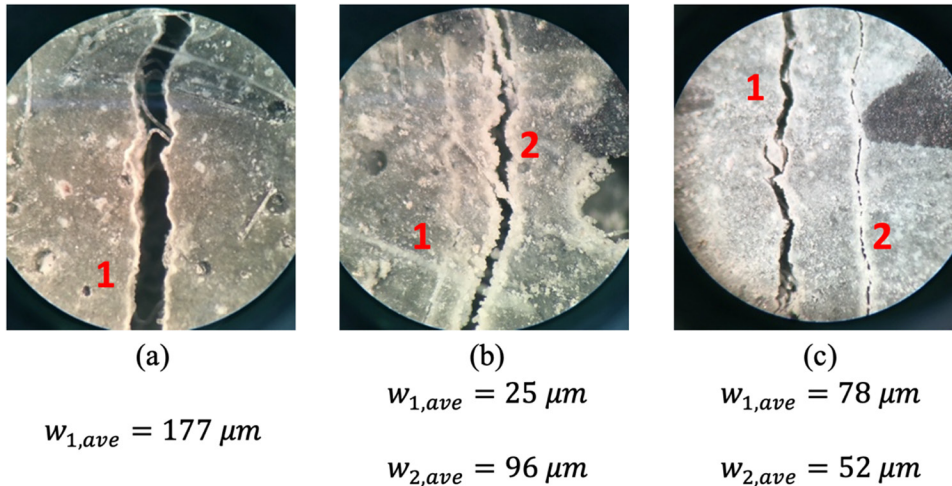


Fig. 5—Variations in healing for composite ECCPOL-SF under P-SLS condition (50× magnification).

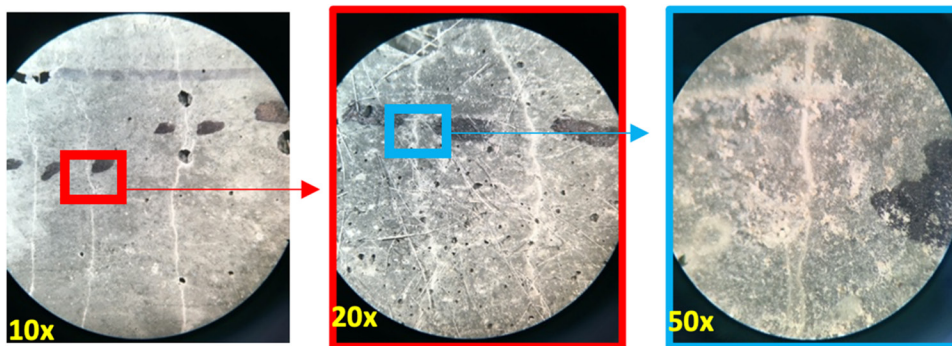


Fig. 6—Visual aspect of completely healed cracks of ECCPOL-SF.

because crack widths varied, it was sufficient to complete healing only for some openings.

The speed of healing and precipitate formation was similar for both widths but the volume necessary to heal the wider crack was approximately six times larger (Fig. 5(b) and (c)). Thus, the effects of crack width on average healing factor were mainly related to physical parameters, rather than chemical parameters related to product formation. Indeed, Roig-Flores et al.⁴⁰ observed that wider cracks required a much larger volume of precipitate to fill the opening. An additional issue would be that, as more material was needed to fill in the crack, the increase in volume could also increase the probability of detachment throughout the process.

Figure 6 shows the visual aspect of completely healed cracks of ECCPOL-SF. The product formed on the inner surface of the cracks was white in color and crystalline in shape. Similar results were observed by Zhang et al.¹⁰ Healing also occurred horizontally, originating from the walls and growing toward the center of the crack. This was also observed by Khaliq and Ehsan,⁴¹ who also determined that the products on the inner surface of the cracks were CaCO_3 crystals from the carbonation of $\text{Ca}(\text{OH})_2$, secondary calcium-silicate-hydrate (C-S-H), and calcium-alumino-silicate-hydrate (C-A-S-H). Additional studies, such as Alyousif,³⁰ noted that as healing progressed, water access to the crack became restricted and the formation of products slowed down.

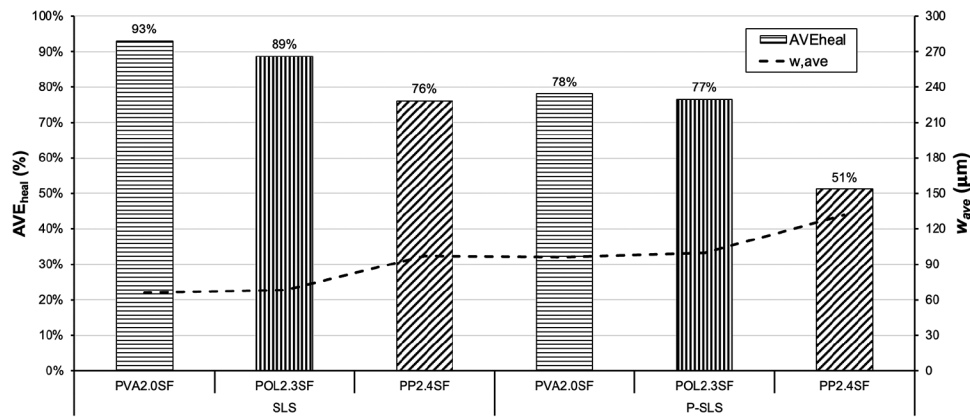


Fig. 7—Healing results for composites with SF as healing agent and curing with WD cycles.

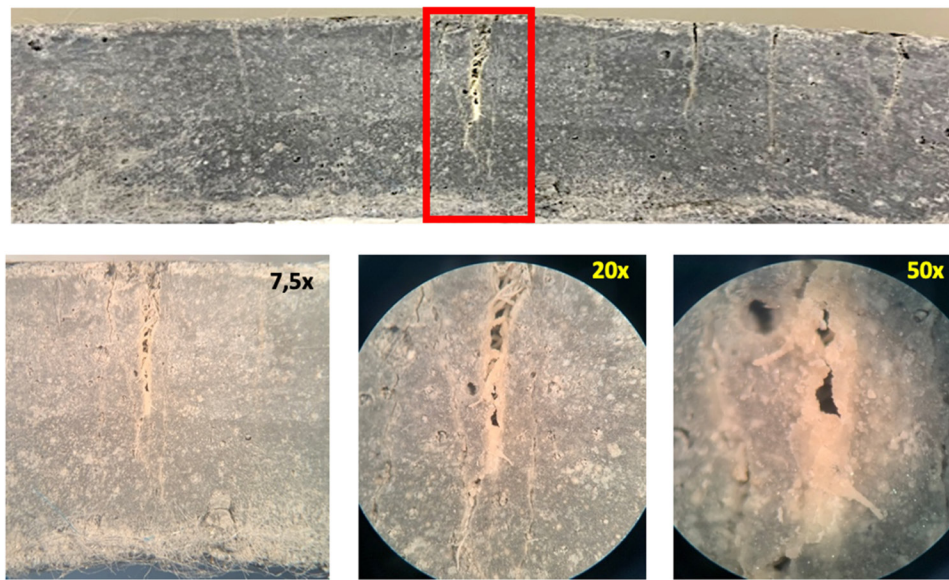


Fig. 8—Precipitation and formation of healing products in composites with SF as healing agent and exposed to WD cycles (ECCPVA-SF).

Curing by exposure to WD cycles—Figure 7 shows healing results for the composites containing SF as the only healing agent and curing by exposure to WD cycles. Similar to the results from SAT curing, AVE_{heal} was affected by crack width (w_{ave}). For example, composite ECCPVA-SF had the same crack width as SAT curing but decreased AVE_{heal} by 8% down to 92%. Still, ECCPVA-SF healing performance was the best among the composites tested.

Results for ECCPVA-SF with SLS condition showed 93% AVE_{heal} for a crack width of approximately 60 μm. These results were in line with Fan and Li³⁹ in that 100% healing was obtained for cracks up to 50 μm wide and between 90 and 95% healing for cracks wider than 50 μm. The other composites of this study under SLS condition achieved AVE_{heal} between 70 and 90%, which was similar to the results of Qiu et al.⁴² for cracks up to 90 μm wide. Results for P-SLS condition were similar to Yıldırım et al.³⁷ Nonetheless, it was possible to verify healed cracks with predominantly white color healing products being formed and precipitated. The form and visual aspect of the healed crack surface were similar to the results of Pacheco.⁴³

In both SLS and P-SLS conditions, the highest values of AVE_{heal} were related to narrower w_{ave} . There were notable similarities, such as ECCPP-SF under SLS condition, and ECCPVA-SF and ECCPOL-SF under P-SLS condition. All these presented w_{ave} near 100 μm and AVE_{heal} between 78% and 75%, respectively. This pointed to a commonality of healing behavior in different composites due to the equivalent crack width.

Overall, composites exposed to WD cycles had lower AVE_{heal} than the ones with SAT curing. This could be related to the changes to the healing mechanisms. In SAT curing, $Ca(OH)_2$ precipitated on the surface of reinforcements and allowed product formation at several points in the crack. In the case of WD cycles, $Ca(OH)_2$ cannot remain adhered to the walls of the crack because it tended to be leached throughout the cycles.

Figure 8 shows the healing product formation in the cracks throughout the WD cycles, which were deposited at the bottom of the cracks as the products precipitated. It should be stressed that the mechanism was precipitation and not formation. Fan and Li³⁹ noted that products formed at the bottom of the crack from autogenous agents relied on direct

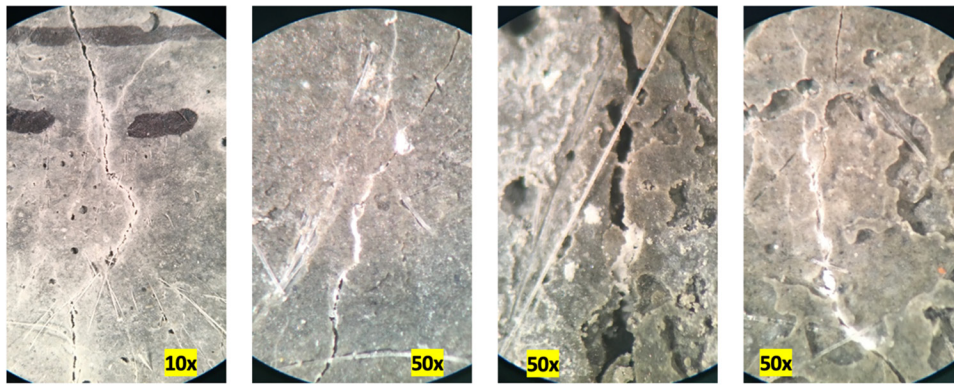


Fig. 9—Retention of healing products and precipitate in sinuous parts of cracks (PP 2.4 SF).

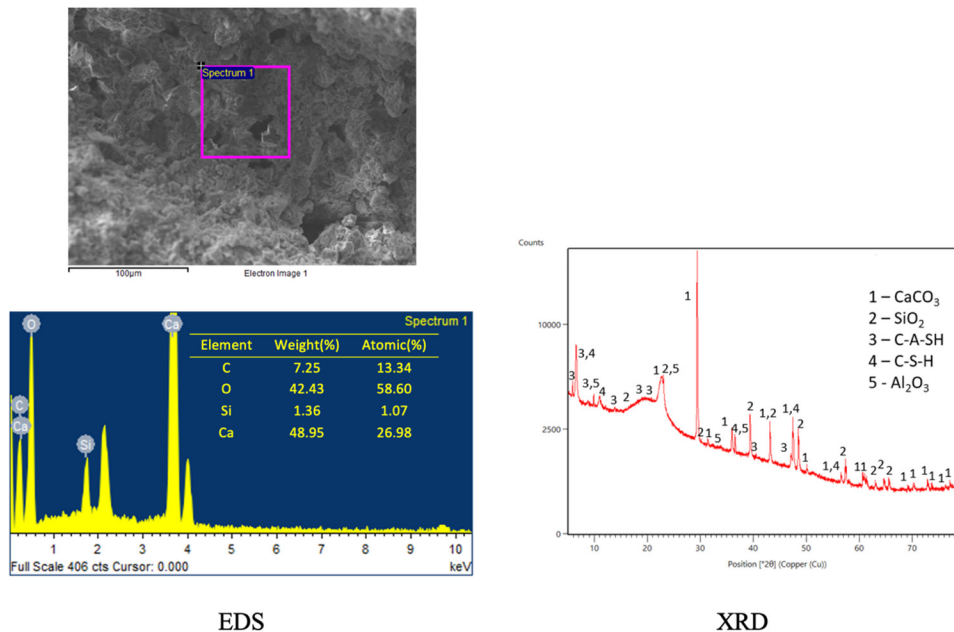


Fig. 10—Chemical elements and compounds identified on healing products—samples with isolated SF.

contact with water through the opening over a prolonged interval, longer than 180 days.

Zhang et al.⁴⁴ stressed that healing decreased as the WD cycles progressed, which indicates products being transported out of the crack. Healing within the crack was as continuous as in the SAT curing. However, Fig. 9 shows that leached material was deposited in sinuous or narrow parts of the crack, which matched the retention of precipitates along the crack observed by Pacheco.⁴³

Mineralogical analysis of healing products—The results of the mineralogical analysis of the healing products are shown in Fig. 10. The significant presence of Ca, O, C, and Si was noted, which can relate to the presence of C-S-H, CA-S-H, and CaCO_3 . The existence of these compounds in healing products was also reported by Fan and Li³⁹ and Kan et al.⁴⁵ The formation of C-S-H and C-A-S-H crystals inside the cracks of these composites may be related to the chemical reactions involving binders and pozzolanic materials used in the reference matrix. In other words, the C-S-H is related to cement hydration reactions and the C-A-S-H to the pozzolanic activities of SF.

The presence of CaCO_3 is due to the carbonation of $\text{Ca}(\text{OH})_2$. Initially, inside the cracks, crystals of C-S-H were

generated, next to $\text{Ca}(\text{OH})_2$, and after C-A-S-H. However, during the curing process, both SAT and in the WD cycles, the CH was exposed to air and humidity variations, which favored the formation of calcite. Thus, the presence of CaCO_3 is related to the carbonation of portlandite, proving the occurrence of the second healing mechanism in these composites.

Because the specimens investigated in the EDS were submerged, it is noteworthy that, chemically, there is no tendency to the natural production of CaCO_3 . Thus, this is attributed to the exposure of the specimens over the periods in which they were removed from the curing process to capture images. The amount of SiO_2 may be related to the SF grains that have not yet been activated.

Healing from SF in combination with CA

Cured immersed in SAT—Figure 11 presents the result of average healing factor AVE_{heal} and w_{ave} from composites with SF in combination with different contents of CA.

Results show a substantial variation in AVE_{heal} both within SLS and P-SLS and between them. For the composites tested with SLS condition, AVE_{heal} varied between 40 and 90%, a variation of a factor of more than two times, despite

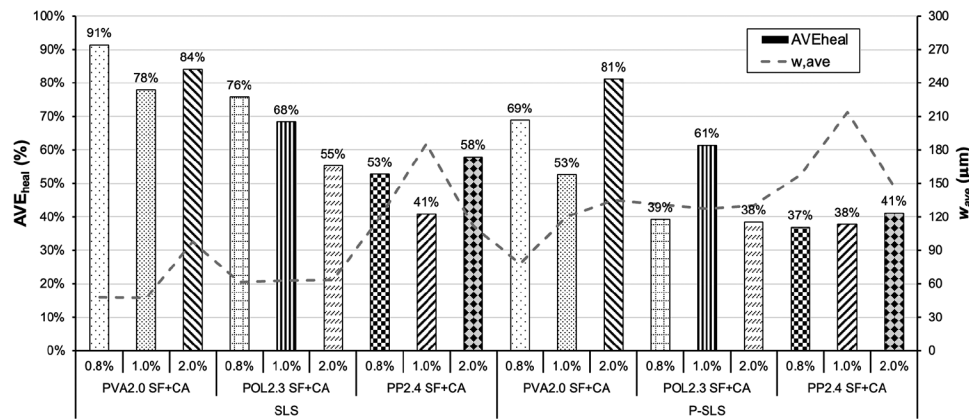


Fig. 11—Healing results for composites with SF combined with CA and cured immersed in water (SAT).

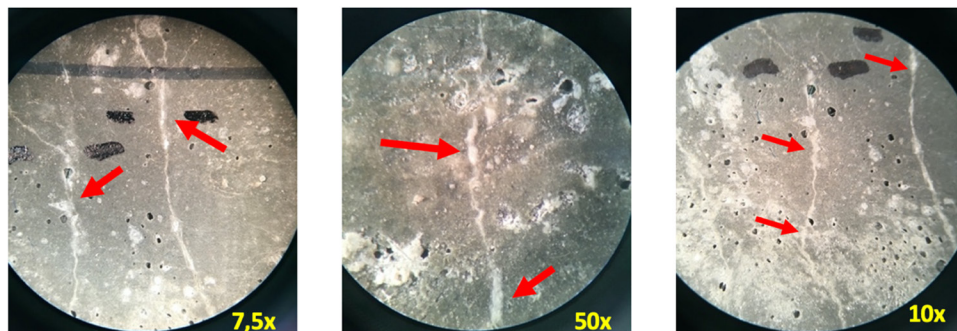


Fig. 12—Visual confirmation of continuous autogenic healing from CA with material runoff on edges of crack (ECCPVA-SF+CA0.8%).

the similarity of the composites. Similar variations between 54 and 78% were also reported by Cuenca et al.²⁵ in specimens with 0.8% CA content and cured immersed in water for 3 months. In addition, w_{ave} varied for the composites of Cuenca et al.²⁵ between 80 and 180 μm, which directly impacted the results. Based on previous results, it was expected the combination of SF and CA would be more effective on crack widths larger than 100 μm. However, Fig. 11 presents AVE_{heal} in the range between 37 and 81% for such opening crack. In comparison, Guzlena and Sakale⁴⁶ reported that CAs filled cracks up to 400 μm wide but only at some locations so that only partial healing was achieved.

It was believed that most healing was provided from the autogenic SF, which acted continuously along the entire crack, unlike the crystalline admixture. Escoffres et al.⁴⁷ also commented that deviations in healing behavior tended to be more noticeable when considering the entire length of the crack due to variations in the healing process itself. Nonetheless, Fig. 12 shows that parts of the cracks were completely healed with product runoff.

The below-expectation performance of the combination of SF and CA might be related to both agents depending on the same cement hydration compound (CH). Thus, competition between the agents ensued during healing, as noted by Oliveira et al.⁴⁸ The limited amount of CH available limited the individual response of each agent within the crack and caused deleterious effects in AVE_{heal} . This linked effect between the agents in the ECC was noticeable in the composite with PVA fibers. Under SAT curing and SF only, total healing of w_{ave} up to 60 μm was possible. But the same

SAT curing applied to the composite with a combination of SF and CA achieved 91% AVE_{heal} , even though w_{ave} were at most 50 μm.

One alternative to enlarge CA efficiency would be to increase its content and dispersion in the matrix, because a greater dispersion would increase the probability of inactive grains being available once a crack formed. Ravitheja et al.⁴⁹ also verified that CA dispersion in the matrix led to healing pointwise or on short stretches along the crack.

As noted by Roig-Flores et al.,⁴⁰ because the product formed from the CA is expansive in nature, this agent had a greater capacity to heal wide cracks that required a greater volume of precipitation. Thus, similar to composites with SF only, healing from crystalline admixtures also depended on w_{ave} . Both factors can be seen at work in Fig. 13, with runoff and external concentration of crystallizing products at several points along the crack, especially at narrower widths. The runoff of crystallizing product from narrow cracks denoted the need to vary the content of healing agent in the composite because it led to waste and inefficient healing. On the other hand, in wider crack widths produced from P-SLS condition, crystallizing product expanded horizontally across the crack and remained mostly within the opening.

Cured by exposure to WD cycles—Figure 14 presents result of the composites with SF in combination with CA cured from exposure to WD cycles.

Figure 14 shows that average healing factor decreased to a minimum of 34% under P-SLS condition, denoting the detrimental effect of WD exposure in the performance of the crystallizing agent. This low value of AVE_{heal} occurred

for composites ECCPP-SF+CA, which also presented the larger w_{ave} among the composites tested and was consequently more susceptible to the same physical limitations in healing observed in composites with SF only. In addition to requiring a substantial amount of healing product to fill the opening, the larger crack widths also affected the flow of water through the crack and healing speed. Leached precipitates were detected in the polymer fibers but the cyclical flow of water in WD cycles led to dissolution and washing away of products. Indeed, the w_{ave} for composites ECCPP-SF+CA reached as high as 210 μm (ECCPP-SF+CA2.0%).

The highest AVE_{heal} values were measured in composite ECCPVA-SF+CA both under SLS and P-SLS conditions. In this case, leached products contained in the fibers were retained within the cracks. Analogous behavior in AVE_{heal} was noted for composites ECCPVA-SF+CA and ECCPOL-SF+CA, with differences between 5 and 11% in average healing factor, while average crack widths were mostly the same.

The crystalline admixture was expected to contribute to the full healing of the cracks but instead AVE_{heal} varied considerably due to the variation in crack width and healing agent dispersion in the matrix. Also, there were negative effects of the flow of water during the WD cycles and the limited amount of CH available both to SF and crystalline admixture. Precipitated healing product was also observed to detach from the walls of the crack throughout the tests, which impacted healing results.

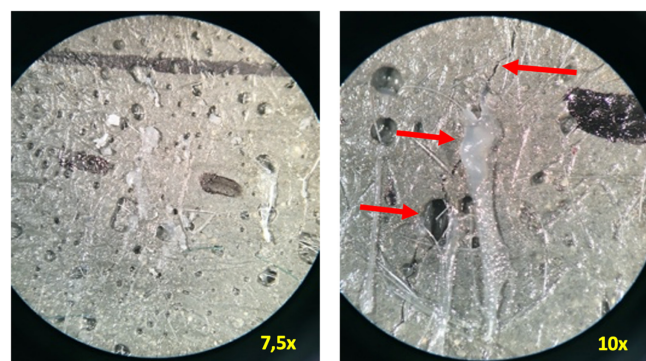


Fig. 13—Runoff evidence of crystallizing product (ECCPOL-SF-CA1.0%).

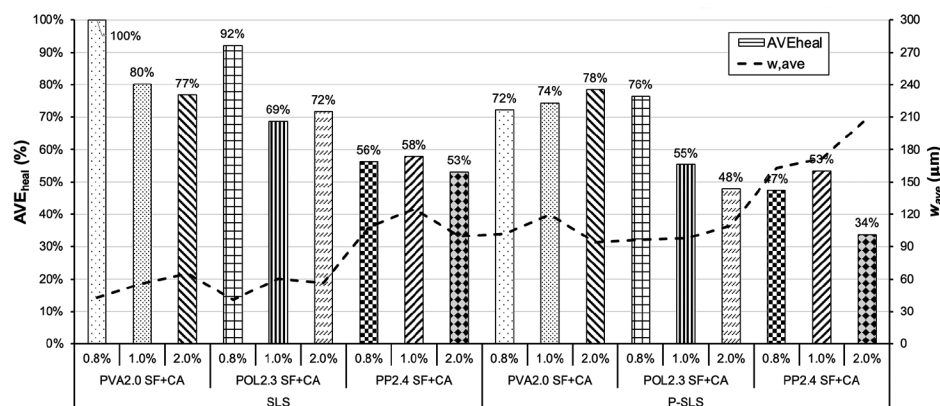


Fig. 14—Healing results for composites with SF combined with CA and exposed to cycles of WD.

The healing performance of composites exposed to WD cycles was, for most cases, better than SAT curing. However, this performance was not with respect to the formation of healing products but for the leaching of soluble crystals into the opening. If only chemical reactions were to be considered, SAT curing was more favorable to healing, as noted by Roig-Flores et al.⁴⁰ In the first WD cycle, both CA and pozzolanic activity from SF were activated from contact with water. But, due to the variations in humidity as the WD cycles progressed, crystallization products did not develop as much as under SAT curing and produced no runoff from the crack. The chemical process tended to slow down in the dry phases and generate smaller crystals. There was a further possibility that anhydrous crystallizing grains remained in the crack but were encapsulated by hydrated and solidified gel, which prevented contact with water and any further activation. The solidification of healing products from the CA is shown in Fig. 15.

The phase change of healing products was also noted by Nasim et al.⁵⁰ Healing products had a high expansion potential in gel form but only in parts of the crack with inert grains. Healing was noted to be vertical, starting from the bottom and growing toward the surface of the opening (inside out). However, healing products presented cracks and detached from the walls of the cracks.

Mineralogical analysis of healing products—The chemical compounds observed in the healing products of the cracks of the specimens are shown in Fig. 16.

The concentration of compounds and chemical elements in the EDS is similar to those presented by Cuenca et al.²⁵ and Sisomphon et al.⁵¹ The crystals and chemical compounds identified were the same highlighted in the composites containing SF (autogenic agent), mainly calcium carbonate and C-S-H.

According to Park and Choi⁵² and Takagi et al.,⁵³ there is indirect formation of CaCO_3 in the chemical reactions of the crystallizer, because there is presence of CaO in the crystallizer which, when in contact with water, is hydrated and forms more expansive crystals of Ca(OH)_2 . With this product, the process of formation of CaCO_3 begins by carbonation, and more compounds for the pozzolans react to form C-A-S-H. The presence of calcite from direct and indirect chemical reactions related to the hydration of crystallizing additives

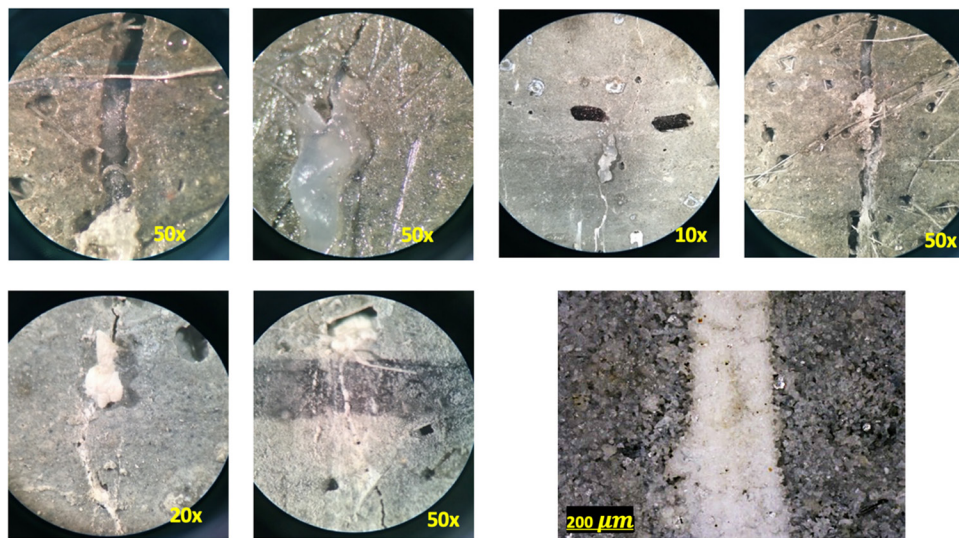


Fig. 15—Solidification process of healing products from chemical reactions of CA (ECCPOL-SF+CA0.8%).

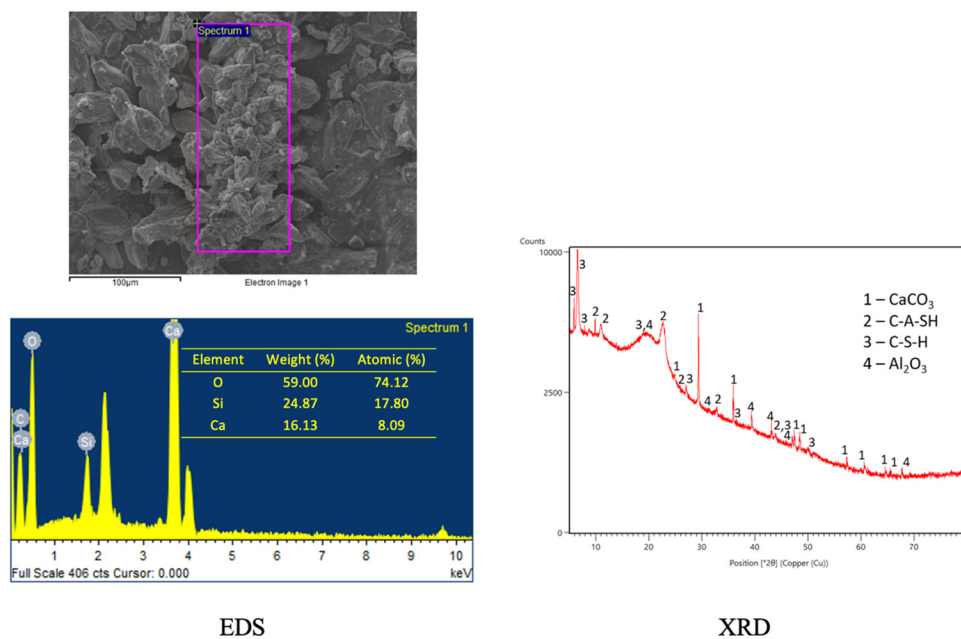


Fig. 16—Chemical compounds identified in healing product formed in composites containing CA (autonomous agent).

was evidenced. Because crystallizer reactions can produce calcite, this formation was considered direct.⁴⁸

Due to the composition of the crystallizing additive used in this research, it was considered that some of its chemical components, such as C_3S , reacted with the soluble crystals $\text{Ca}(\text{OH})_2$ to form crystalline products that disconnect the pores and precipitated in the cracks of each ECC as C-S-H, as indicated by Oliveira et al.⁴⁸

Healing discussion

Comparing the results from specimens cured by immersion in SAT or exposed to WD cycles, a trend of decreasing in AVE_{heal} when subjected to variations in humidity was noted. This decrease was not necessarily significant as AVE_{heal} variations were less than 10% for all composites except for ECCPOL-SF with P-SLS condition. Qiu et al.⁴² also examined the effect of exposure conditions and noted a variation of up to 20% in AVE_{heal} in composites with pozzolans as

autogenous healing agent. The healing potential from SAT curing was superior to WD cycles. However, this result was related to the washout of products during the WD cycles and the stabilization of the crystallizing agent when no longer exposed to humidity.

After the curing period, the post-healing average residual crack width was calculated ($w_{\text{ave,res}}$), and the results are shown in Table 5.

Table 5 shows that composites based on ECCPVA under SLS condition and SAT curing presented the best results for $w_{\text{ave,res}}$ regardless of the added healing agents. Compared to composites based on ECCPOL and ECCPP, the largest $w_{\text{ave,res}}$ of ECCPVA was 16 μm , which was quite small compared to Yildirim et al.³⁷ and Zhang et al.⁵⁴

Under SLS conditions, results from WD cycles were similar to SAT curing in that $w_{\text{ave,res}}$ was still smaller for ECCPVA composites compared to the others, albeit with larger overall values due to the alterations in healing caused

Table 5—Post-healing average residual crack width ($w_{ave,res}$) in all composites of this study exposed to SAT curing or WD cycles

Composites	$w_{ave,res}$, μm			
	SLS		P-SLS	
	SAT	WD	SAT	WD
ECCPVA-SF	0	7	14	14
ECCPOL-SF	11	8	49	23
ECCPP-SF	40	23	53	64
ECCPVA-SF+CA0.8%	4	0	17	28
ECCPOL-SF+CA0.8%	15	3	80	23
ECCPP-SF+CA0.8%	56	47	100	85
ECCPVA-SF+CA1.0%	10	11	57	31
ECCPOL-SF+CA1.0%	20	19	49	44
ECCPP-SF+CA1.0%	110	53	134	80
ECCPVA-SF+CA2.0%	16	15	25	20
ECCPOL-SF+CA2.0%	28	16	80	57
ECCPP-SF+CA2.0%	47	47	87	138

by the cyclical flow of water. Under SAT curing, the average difference in $w_{ave,res}$ between ECCPVA and ECCPOL composites was 11 μm , which reduced to 3 μm under WD cycles. This reduction in the differences in $w_{ave,res}$ between composites indicated a characteristic of WD cycles.

Results for testing under P-SLS conditions were similar to SLS. Overall, $w_{ave,res}$ were smaller for SAT curing than for WD cycles and PVA 2.0 composites outperformed the other composites regardless of healing agents. On the other hand, the differences in $w_{ave,res}$ between composites increased when compared to SLS. Comparing ECCPVA and ECCPOL composites, the average difference in $w_{ave,res}$ increased to 33 μm , which was three times larger than under SLS conditions.

In the case of P-SLS condition with exposure to WD cycles, the smallest $w_{ave,res}$ was 14 μm for ECCPVA-SF. This indicated that PVA fibers partially blocked the ingress of aggressive agents in the cracks compared to the others, which had larger $w_{ave,res}$. Comparing ECCPVA and ECCPOL, the average difference in $w_{ave,res}$ after WD cycles was 16 μm . Thus, it was clear that leaching from the WD cycles increased w_{ave} and produced discrepancies in $w_{ave,res}$ between composites.

Results showed that the most efficient healing agent was autogenous SF, which demonstrated continuous healing along the crack length and presented the lowest $w_{ave,res}$. The autonomous CA also contributed to healing pointwise, but did not contribute significantly to $w_{ave,res}$.

Analyzing the results from a design perspective, WD conditions are those in which structures are most exposed, and under these circumstances, the most appropriate values were obtained with ECCPVA using exclusively active silica as a healing agent. For structures and elements with ECC subject to WD cycles, the crystallizing additive is not an attractive solution due to the conditions and dimensions of the cracks produced.

CONCLUSIONS

The following conclusions were drawn from the results of this study:

1. The results showed that the combination of silica fume (SF) and crystallizing additive reduced the healing potential of engineered cementitious composites (ECCs) because both mechanisms involve the reaction with calcium hydroxide and water.

2. Comparing healing agents, there was a clear superiority of SF over crystallizing admixture (CA). SF was noted to produce average product widths in cracks up to 100 μm wide. Additionally, the smaller average residual widths ($w_{ave,res}$) were measured in composites with SF only. SF composites had a maximum $w_{ave,res}$ of 40 and 64 μm for serviceability limit state (SLS) and post-SLS (P-SLS) conditions, respectively.

3. Autonomous agents, in this case CA, made a limited contribution to healing with average product widths below 100 μm , regardless of its content. Their main effect was pointwise, localized healing along the length of the crack.

4. The width of the crack had a direct impact on AVE_{heal} for both healing agents. Leaching was observed for both water saturation (SAT) curing and wetting-and-drying (WD) cycle exposure. More leaching occurred in specimens exposed to WD cycles and resulted in products adhering to the crack inner surface, especially in more sinuous portions. In the case of SAT curing, a more uniform product formation was noted along the length of the crack, and no runoff was observed.

5. Cracks more than 150 μm in width had varied healing results from exposure conditions. Water flow through the crack removed part of the products and reduced AVE_{heal} . Consequently, wider cracks could present a considerable challenge to be filled because the cyclical and more intense flow of water from WD cycles would be detrimental to the deposition of products.

6. For structures and elements with ECC subject to WD cycles, the crystallizing additive is not an attractive solution due to the conditions and dimensions of the cracks produced.

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Title No. 123-M11

Optimizing Cementitious Materials for SiO₂ Aerogel-Gypsum Composites

by Yunan Wan and Hongping Zhang

To prepare SiO₂ aerogel gypsum-based lightweight thermal insulation wall materials with better water resistance, α -hemihydrate gypsum (HG) was used as the main cementitious material. By adding portland cement (PC), fly ash (FA), and hydrated lime (HL), HG was modified. Using these materials, the HG-PC system and HG-PC-FA-HL system were constructed. The effects of inorganic admixture content on the performance of both systems were analyzed. Results show that the mechanical properties and water resistance were improved after adding a certain proportion of mineral admixtures to HG. The mechanical properties and water resistance of the HG-PC-FA-HL system were better than those of the HG-PC system. At the content of 9 wt. % FA, 20 wt. % PC, and 4 wt. % HL, the 28-day strength reached 41.07 MPa (5955 psi), the water absorption after soaking for 48 hours was 12.7%, and the softening coefficient was 0.72.

Keywords: gypsum-based cementitious materials; SiO₂ aerogel inorganic composite materials; solid wastes; water resistance.

INTRODUCTION

SiO₂ aerogels are characterized by high porosity, high specific surface area, low density, low thermal conductivity, and strong fire resistance.^{1,2} Incorporating SiO₂ aerogel into inorganic cementitious materials can significantly improve thermal insulation performance and fire resistance, demonstrating great potential in building insulation applications. Current composite thermal insulation materials are mainly prepared by adding SiO₂ aerogel particles or powders as lightweight aggregates into inorganic cementitious materials, such as thermal insulation mortar,³⁻⁵ thermal insulation board,⁶ thermal insulation concrete,⁷⁻⁹ and so on. However, most existing inorganic cementitious materials are cement-based rather than gypsum-based, despite the latter's better thermal insulation properties.

Compared with cement, gypsum offers distinct advantages in lightweight applications, presenting substantial market potential for wall insulation materials.¹⁰ However, the water resistance of gypsum is poor, which significantly limits its construction applications.^{11,12} The incorporation of hydraulic cementitious materials (for example, cement) can fundamentally alter the structure of hardened gypsum. This method is commonly used to improve its water resistance.¹³⁻¹⁹ For instance, Jin et al.²⁰ used calcium sulfoaluminate cement (SAC) to improve the performance of β -hemihydrate phosphogypsum. With the addition of 20% SAC, the compressive strength and softening coefficient reached 21.5 MPa (3118 psi) and 0.66. Compared to the mixture without SAC, the values increased by 60.85% and 96.04%, respectively. Camarini and De Milito²¹ investigated

the effect of blast-furnace slag portland cement (PC) addition to β -hemihydrate. Results showed an improvement in gypsum performance when this kind of cement was added to the mixture, and a 3-year rendering exposed to the external environment had no changes.

The hydration product calcium-silicate-hydrate (C-S-H) gel enhances gypsum's water resistance by filling intergranular pores in CaSO₄·2H₂O and preventing dissolution, while ettringite (AFt) improves mechanical strength through pore-structure optimization and framework reinforcement. However, excessive or untimely AFt formation may cause strength loss and cracking, compromising long-term durability.^{22,23} Therefore, when using cement to improve the water resistance of gypsum, the volume stability issues caused by AFt must be addressed.^{24,25} Qi et al.²⁶ and Zhang et al.²⁷ proposed using ultrafine mineral admixtures to optimize the crystalline-amorphous ratio in gypsum-cement composite systems. This optimized mineral composition achieves a synergistic enhancement in both water resistance and volume stability of gypsum-based cementitious materials. Gou et al.²⁸ found that both binary and ternary systems consisting of gypsum, cement, and mineral powder showed good water resistance compared to pure gypsum. The softening coefficient at 28 days improved, reaching 0.71 at the optimal proportion.

As a representative solid waste resource, fly ash (FA) exhibits dual functionalities of pozzolanic activity and physical filling. Its vitreous components, activated by hydrated lime (HL), participate in secondary hydration reactions to form C-S-H gel. This effectively densifies the microstructure by occupying intergranular pores within gypsum crystals. Furthermore, the constructed HG-PC-FA-HL system not only achieves high-value use of FA but also enhances water resistance and mechanical properties. This is achieved through a synergistic mechanism involving controlled AFt and C-S-H gel reinforcement, aligning with the sustainability objectives for low-carbon green building materials.

Although extensive research has been conducted on cement-gypsum-FA combinations, most studies focus on the high-strength enhancement of cement-based cementitious materials. For gypsum-based cementitious materials, current research primarily concentrates on the strength optimization

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of β -hemihydrate gypsum (HG). The α -hemihydrate gypsum is a crystalline state of HG, which has excellent mechanical properties due to its very dense structure. In addition, α -hemihydrate gypsum also has the characteristics of lightweight thermal insulation and fire resistance. This study modifies α -hemihydrate gypsum by adding an inorganic admixture, thereby developing both a cement-gypsum binary system and a gypsum-cement-FA-lime quaternary system. The influence of mineral admixture dosage on the properties of the composite cementitious system was studied, and its reaction mechanism was discussed. Furthermore, the performance of SiO₂ aerogel gypsum-based composites was evaluated.

RESEARCH SIGNIFICANCE

By systematically investigating the HG-PC and HG-PC-FA-HL systems, this study elucidates the synergistic enhancement of mechanical strength and water resistance of gypsum-based cementitious materials. This approach not only enables the preparation of high-performance gypsum-based cementitious materials but also promotes the resource use of solid wastes. Furthermore, by integrating gypsum-based cementitious materials with silica aerogels, this research lays a foundation for developing advanced building insulation materials. These materials combine exceptional thermal insulation efficiency with flame retardancy.

EXPERIMENTAL INVESTIGATION

Raw materials

The α -hemihydrate gypsum was supplied by a gypsum product company in China, with physical properties detailed in Table 1. The chemical composition of all raw materials is shown in Table 2, and the particle-size analysis is shown in Fig. 1.

Mixture proportions

The HG-PC system was constructed by adding different proportions of cement, as shown in Table 3. To systematically study the influence of various factors on the performance of the HG-PC-FA-HL system, an orthogonal test design was employed. This test included three factors: cement content (Factor A), FA content (Factor B), and HL content (Factor C). Each factor was tested at five levels. Orthogonal testing was

Table 1—Physical properties of HG

Water requirement for normal consistency, %		37.5
Setting time	Initial setting time, minutes	7
	Final setting time, minutes	13
Strength	Compressive strength at drying, MPa (psi)	30.72 (4454)
	Flexural strength at 2 hours, MPa (psi)	4.17 (605)

Table 2—Chemical composition of raw materials, %

Raw materials	CaO	Al ₂ O ₃	SiO ₂	MgO	Fe ₂ O ₃	SO ₃
HG	37.53	1.05	2.18	0.93	0.31	48.77
PC	65.39	5.57	22.00	1.32	3.70	0.29
FA	2.55	40.5	46.3	0.56	1.68	1.81
HL	95.4	0.41	1.78	1.27	0.35	0.28

applied to evaluate the effects of these factors. Qi et al.²⁶ and Wang et al.²⁹ studied the effects of superfine mineral admixture and FA content on gypsum. They observed that the optimum addition was approximately 10 to 15%. James et al.³⁰ formulated a blended binder from FA and phosphogypsum incorporating lime and found that a 5% lime dosage achieved the peak compressive strength in cube specimens. In the authors' previous studies, the compressive strength of the cementitious material was investigated. It was found that the highest compressive strength of the cementitious material was achieved when the composition mass ratio of the inorganic cementitious material was 1:3:9:0.8 (FA: cement:HG:lime).³¹ To enhance testing efficiency, it is essential to set appropriate factor levels. The test factors and their respective levels are presented in Table 4, while the orthogonal test mixture proportions design is provided in Table 5.

Preparation and test methods

Preparation and mechanical testing—All raw materials were mixed in a pan mixer according to the designated proportions for 2 to 3 minutes. After water addition, the mixtures were stirred evenly for 2 minutes and then cast into steel molds. In this study, the compressive strength was measured according to the Chinese standard GB/T 17669.3-1999, reporting dry strength values.

Softening coefficient—Test specimens at designated ages were water-saturated to determine wet strength, then completely dried to measure dry strength. The softening coefficient was defined as the ratio of wet strength to dry strength of the test specimens and was calculated as follows

$$K = \frac{R_{cw}}{R_{cd}}$$

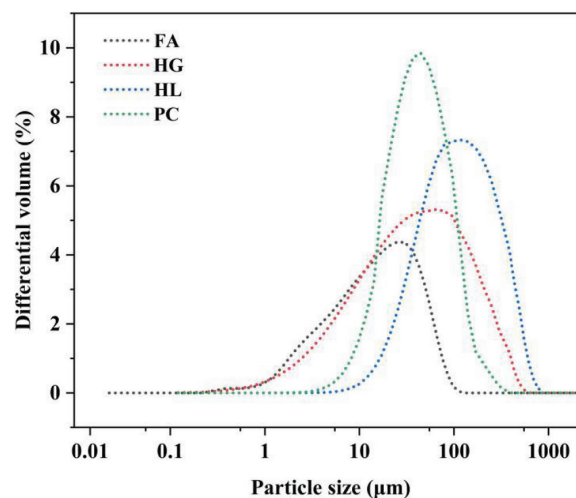


Fig. 1—Particle-size distribution of raw materials.

Table 3—Mixture ratio of PC-HG system

Sample	PC, wt. %	HG, wt. %
CG0	0	100
CG1	10	90
CG2	20	80
CG3	30	70
CG4	40	60
CG5	50	50

Table 4—Factors and levels of orthogonal tests

Level	Factor, wt. %		
	A (cement)	B (fly ash)	C (hydrated lime)
1	10	3	2
2	20	6	4
3	30	9	6
4	40	12	8
5	50	15	10

where K is the softening coefficient; R_{cw} is the compressive strength of the specimens submerged in water, MPa (psi); and R_{cd} is the compressive strength of specimens that have cured in dry air, MPa (psi).

Water absorption—The test specimens, having reached the expected age, were placed in an oven until achieving constant mass, which was recorded as dry mass. Then, the test specimens were completely immersed in water to absorb water. After reaching the specified immersion time, the test specimens were taken out. The surface moisture was removed using a wrung-dry wet towel, and the saturated mass was measured. The water absorption rate of the test specimens was calculated as follows

$$W = \frac{m_1 - m_0}{m_0} \times 100\%$$

where W is the water absorption; m_1 is the water-saturated mass of the specimens, g (oz.); and m_0 is the dry mass of the specimens, g (oz.).

EXPERIMENTAL RESULTS AND DISCUSSION

Performance of HG-PC system

Compressive strength and softening coefficient of HG-PC system—As shown in Fig. 2(a), the compressive strength of the HG-PC system at different ages first increased then decreased with an increase in PC content. When the PC content was 10 wt. %, the compressive strength at 3 and 7 days was higher than that of other specimens. As the curing age progressed, the hydration reaction of the HG-PC system continued. When PC content was 20 wt. %, the compressive strength at 28 days exceeded that of other specimens, reaching 37.83 MPa (5485 psi), an increase of 11.0% compared with HG. It can be seen from Fig. 2(b) that the softening coefficients of the HG-PC system at 3 and 7 days both followed similar trends. When PC content was 20 wt. %, the softening coefficient of the 3-day specimen

Table 5—Orthogonal test table of HG-PC-FA-HL system

Number	A	B	C	Combination mode
F1	1	3	2	$A_1B_3C_2$
F2	2	5	2	$A_2B_5C_2$
F3	3	3	5	$A_3B_3C_5$
F4	1	1	1	$A_1B_1C_1$
F5	4	5	5	$A_4B_5C_5$
F6	1	5	3	$A_1B_5C_3$
F7	2	4	4	$A_2B_4C_4$
F8	5	5	4	$A_5B_5C_4$
F9	2	3	1	$A_2B_3C_1$
F10	2	2	3	$A_2B_2C_3$
F11	5	1	2	$A_5B_1C_2$
F12	3	2	2	$A_3B_2C_2$
F13	5	4	1	$A_5B_4C_1$
F14	4	1	3	$A_4B_1C_3$
F15	3	5	1	$A_3B_5C_1$
F16	4	3	4	$A_4B_3C_4$
F17	5	2	5	$A_5B_2C_5$
F18	3	4	3	$A_3B_4C_3$
F19	2	1	5	$A_2B_1C_5$
F20	5	3	3	$A_5B_3C_3$
F21	3	1	4	$A_3B_1C_4$
F22	4	2	1	$A_4B_2C_1$
F23	4	4	2	$A_4B_4C_2$
F24	1	2	4	$A_1B_2C_4$
F25	1	4	5	$A_1B_4C_5$

was higher than that of the other specimens, reaching 0.63. When the PC content was 30 wt. %, the softening coefficient of the 7-day specimen was higher than that of the other specimens, reaching 0.65. Furthermore, the softening coefficient of the HG-PC system increased with the increase in curing age. At the curing age of 28 days, the softening coefficient increased with the increase in PC content.

According to previous literature reports,^{20,26} the formation of C-S-H gel and ettringite (AFt) by cement hydration may be the main reasons for the increase in compressive strength. The authors' subsequent tests also proved this point. During the early stage of hydration, the compressive strength of HG-PC composite cementitious materials was mainly provided by the interlocking of $CaSO_4 \cdot 2H_2O$ crystals generated from HG hydration. Upon the addition of partial PC, the dihydrate gypsum precipitated during the HG hydration process will react with tricalcium aluminate (C_3A) in the cement clinker. This reaction forms AFt. The C-S-H gel and AFt are generated from the hydration of cement. They cross-lap with each other, wrapping the gypsum dihydrate crystal and increasing the system's compressive strength and softening coefficient. When cement content is too high, a larger proportion remains as unhydrated particles in the

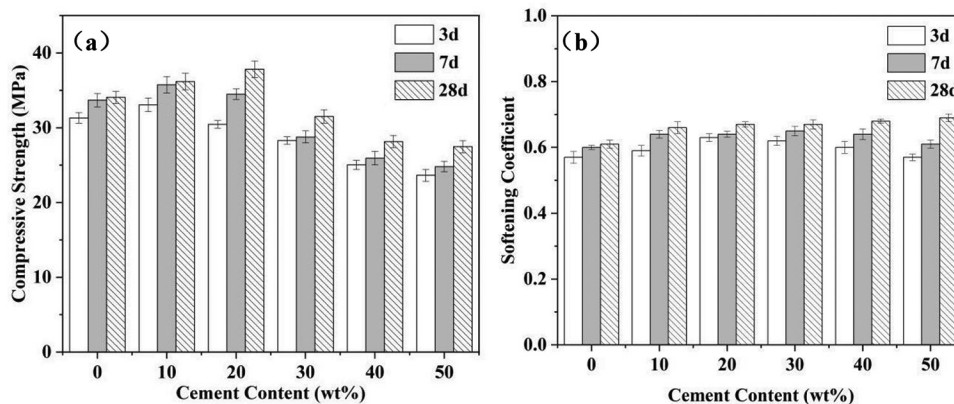


Fig. 2—Compressive strength and softening coefficient of HG-PC system: (a) compressive strength; and (b) softening coefficient. (Note: 1 MPa = 145 psi.)

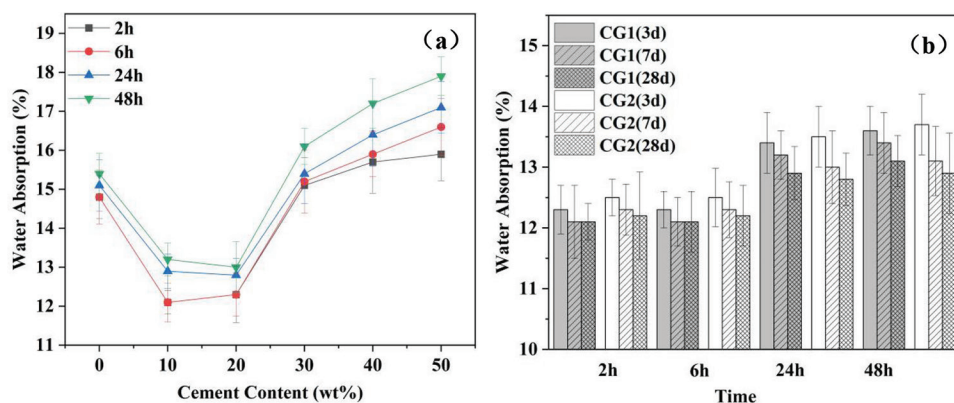


Fig. 3—Water absorption of HG-PC system: (a) water absorption of samples; and (b) water absorption of CG1 and CG2 at different curing ages.

system. The contact opportunity between dihydrate gypsum and water in the system increases, and the compressive strength and softening coefficient decrease.

More C-S-H gel and Aft are generated from the hydration of cement with the increase in age, which cross-lap with each other and wrap the gypsum dihydrate crystal. The scanning electron microscopy (SEM) images and X-ray diffraction (XRD) patterns that will appear subsequently also provide compelling evidence to confirm this speculation. This helps to reduce structural damage caused by water infiltration, thereby improving water resistance. With the continuous increase in PC content, the proportion of C-S-H gel and Aft in the hydration products increases, leading to enhanced water resistance and an increase in the softening coefficient. However, when PC content is too high, the hydration products formed by continued PC hydration will disrupt the framework built by gypsum hydration. This results in a decrease in the compressive strength of the system.

Water absorption of HG-PC system—The water absorption of the HG-PC system is related to its hydrated pore structure. As shown in Fig. 3, with the increase in PC content, the water absorption of the HG-PC system first decreased and then increased. As the absorption time extended, the water absorption rate gradually rose. When the PC content was 0 to 30 wt. %, the water absorption of HG-PC specimens soaked for 2 and 6 hours showed no significant change. After soaking for 24 and 48 hours, the water absorption rate

increased slightly, but the increase was not significant. When the PC content was 40 to 50 wt. %, the water absorption of the specimen increased significantly with longer soaking time. This is because, after adding an appropriate amount of PC to the HG, the C-S-H gel and Aft produced by PC hydration filled some of the pores in the HG-PC system. As a result, the material's density increased, and the water absorption decreased. However, when PC content is too high, C-S-H gel and Aft will disrupt the framework of the hydration products of HG due to the different hydration rates of HG and PC. This leads to cracks in the material, an increase in porosity, and consequently, higher water absorption. Figure 4 shows the corresponding microstructural changes.

Based on the aforementioned results, the water absorption of CG1 and CG2 was tested at different curing ages, which were significantly lower than those of other specimens. As shown in Fig. 3(b), the water absorption rate of the HG-PC system decreased with the increase in curing age. When the hydration age was 3 and 7 days, the water absorption of CG1 for different soaking times was lower than that of CG2. When the hydration age was 28 days, the water absorption of CG2 was lower than that of CG1. This is because in the early stage of hydration, the hydration rate of gypsum is very fast, while the hydration rate of cement is slow. When PC content was 10 wt. %, the HG content with a faster hydration rate in CG1 was higher. This leads to higher compactness in

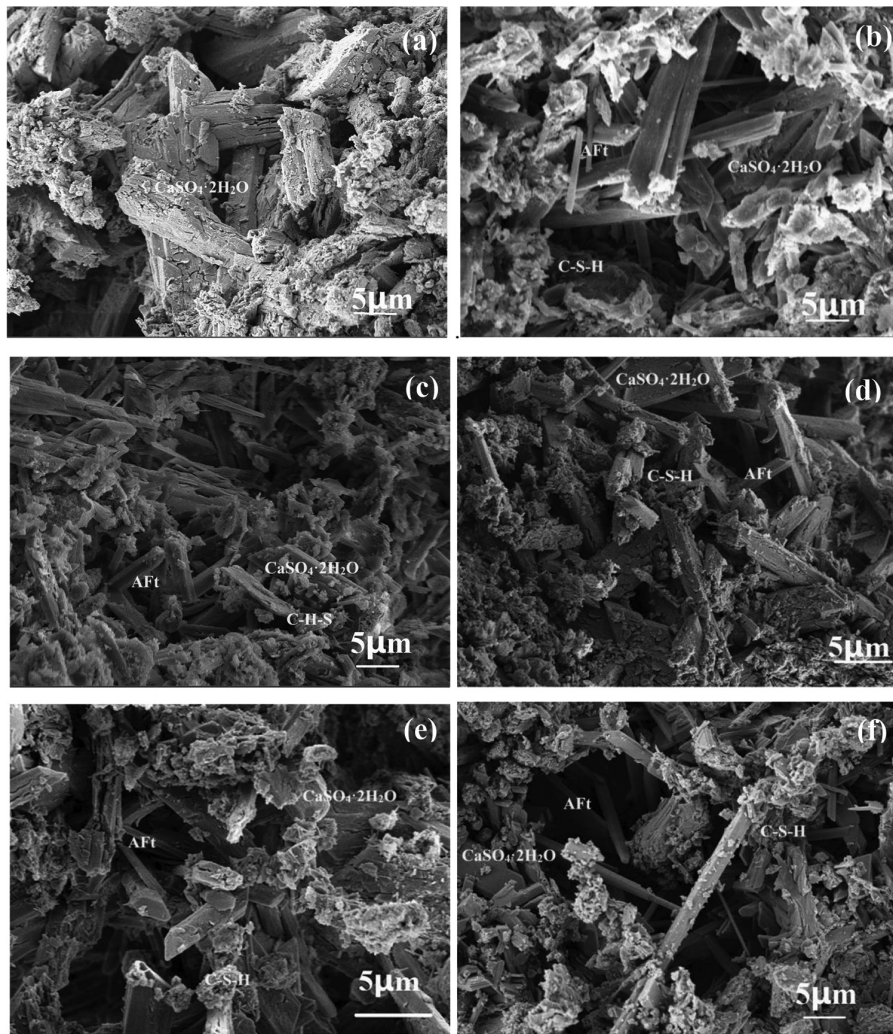


Fig. 4—SEM pictures of HG-PC system: (a) CG0; (b) CG1; (c) CG2; (d) CG3; (e) CG4; and (f) CG5.

the early stage of hydration compared to CG2 and lower water absorption. With the increase in hydration time, the cement in the composite cementitious material gradually hydrates, generating more C-S-H gel and AFt. At this time, the structure of CG2 is denser, and the water absorption is lower. This variation aligns well with the trend in compressive strength changes.

Microstructural and mineral composition analysis of HG-PC system—The microstructural characteristics and XRD patterns of the specimen at 28 days are shown in Fig. 4 and 5. According to Fig. 5, the main components of the hydration products of CG0 are $\text{CaSO}_4 \cdot 2\text{H}_2\text{O}$ crystals. When PC content was 10 wt. %, the diffraction peaks of C-S-H gel and AFt began to appear in CG1, while the diffraction peak intensity of $\text{CaSO}_4 \cdot 2\text{H}_2\text{O}$ decreased. When PC is added, the tricalcium silicate (C_3S) and dicalcium silicate (C_2S) in PC undergo a hydration reaction to generate C-S-H gel. The tricalcium aluminate (C_3A) will immediately react with HG to form AFt crystals. As a result, the amount of $\text{CaSO}_4 \cdot 2\text{H}_2\text{O}$ in the hydration products was reduced compared to HG.

As shown in Fig. 4(a), the main hydration product in CG0, without the addition of cement, was coarse, rod-shaped crystals that interlace with each other. These structures form a relatively compact structure, contributing to high compressive strength. It can be seen from Fig. 4(b) and (c) that when

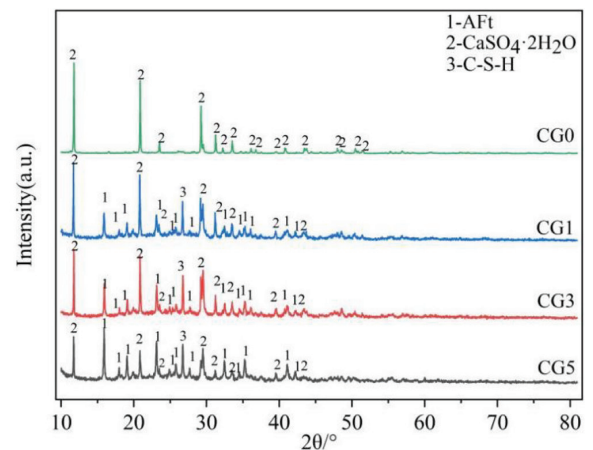


Fig. 5—XRD patterns of HG-PC system.

10 to 20 wt. % cement was added to gypsum, the hydration products of CG1 and CG2 contained long, rod-shaped AFt and reticular and fibrous C-S-H gel. These either overlapped with thick, rod-shaped $\text{CaSO}_4 \cdot 2\text{H}_2\text{O}$ or coated the surface of $\text{CaSO}_4 \cdot 2\text{H}_2\text{O}$. This makes the cementitious system structure more compact and reduces the exposure of $\text{CaSO}_4 \cdot 2\text{H}_2\text{O}$ to water. As a result, the system shows higher compressive

Table 6—Orthogonal test table of HG-PC-FA-HL system

Number	PC, wt. % A	FA, wt. % B	HL, wt. % C	Compressive strength, MPa			Softening coefficient 28 days	Water absorption, % 48 hours
				3 days	7 days	28 days		
F1	1 (10)	3 (9)	2 (4)	30.17	31.28	38.56	0.61	12.69
F2	2 (20)	5 (15)	2 (4)	18.17	29.42	40.93	0.73	12.34
F3	3 (30)	3 (9)	5 (10)	21.23	27.71	32.15	0.56	15.91
F4	1 (10)	1 (3)	1 (2)	32.54	34.60	40.57	0.61	12.51
F5	4 (40)	5 (15)	5 (10)	12.42	17.29	27.50	0.60	17.91
F6	1 (10)	5 (15)	3 (6)	17.80	23.70	31.07	0.58	15.93
F7	2 (20)	4 (12)	4 (8)	26.28	26.50	35.02	0.61	14.53
F8	5 (50)	5 (15)	4 (8)	12.61	15.80	28.00	0.73	17.28
F9	2 (20)	3 (9)	1 (2)	30.00	30.71	39.31	0.65	12.57
F10	2 (20)	2 (6)	3 (6)	24.37	35.48	41.71	0.64	12.31
F11	5 (50)	1 (3)	2 (4)	25.28	29.20	34.01	0.65	15.41
F12	3 (30)	2 (6)	2 (4)	26.71	30.47	35.46	0.67	14.10
F13	5 (50)	4 (12)	1 (2)	20.51	26.24	30.58	0.73	16.17
F14	4 (40)	1 (3)	3 (6)	23.39	26.45	29.90	0.68	16.37
F15	3 (30)	5 (15)	1 (2)	25.31	27.61	37.76	0.64	12.73
F16	4 (40)	3 (9)	4 (8)	19.52	25.30	30.22	0.60	16.11
F17	5 (50)	2 (6)	5 (10)	20.17	22.62	25.87	0.62	18.41
F18	3 (30)	4 (12)	3 (6)	20.59	27.76	32.27	0.68	15.77
F19	2 (20)	1 (3)	5 (10)	16.21	22.67	33.40	0.50	15.47
F20	5 (50)	3 (9)	3 (6)	28.14	31.75	34.84	0.71	13.83
F21	3 (30)	1 (3)	4 (8)	19.45	25.39	30.14	0.60	16.12
F22	4 (40)	2 (6)	1 (2)	28.13	30.20	33.58	0.64	15.39
F23	4 (40)	4 (12)	2 (4)	25.52	32.10	36.73	0.66	13.01
F24	1 (10)	2 (6)	4 (8)	22.24	27.78	33.40	0.56	15.45
F25	1 (10)	4 (12)	5 (10)	23.23	25.76	31.22	0.58	16.23

Note: Numbers in parentheses indicate the respective mass percentages corresponding to the three components; 1 MPa = 145 psi.

strength and softening coefficient, along with a lower water absorption rate.

With the increase in PC content, the diffraction peaks of $\text{CaSO}_4 \cdot 2\text{H}_2\text{O}$ in the hydration products of CG3 and CG5 gradually decreased. At the same time, the diffraction peaks of C-S-H gel and AFt increased. As shown in Fig. 4(d) to (f), when 30 to 50 wt. % cement is added to gypsum, the hydration products of CG3, CG4, and CG5 contain more network-like or fibrous C-S-H gel. They also show more long, rod-shaped AFt crystals surrounding the coarse rod $\text{CaSO}_4 \cdot 2\text{H}_2\text{O}$. This further reduces the structural damage caused by water infiltration, thereby improving the softening coefficient of the specimens. Moreover, due to the rapid hydration rate of gypsum and the slow hydration rate of cement, the C-S-H gel and AFt from cement hydration will disrupt the framework structure after gypsum hydration. This results in the formation of cracks, increased porosity, and consequently reduced compressive strength and increased water absorption.

Performance of HG-PC-FA-HL system

Test results of HG-PC-FA-HL system—To reflect the characteristics of the HG-PC-FA-HL system, the softening coefficient and compressive strength at 3, 7, and 28 days for 25 groups of specimens were measured in this test. The results are shown in Table 6.

Range analysis—As can be seen from Table 7, the order of influence on the compressive strength of the HG-PC-FA-HL system at 3 and 7 days was $C > B > A$. At 28 days, the order changed to $A > C > B$. From Fig. 6, it can be seen that the compressive strength of the system at 3 days decreased with the increase in PC and HL contents. However, it first increased and then decreased with the increase in FA content. The optimal value of FA content is 9 wt. %. At 7 and 28 days, the compressive strength of the system first increased and then decreased as the contents of PC, FA, and HL increased. The highest compressive strength was achieved when the contents of PC, FA, and HL were 20 wt. %, 9 wt. %, and 4 wt. %, respectively.

As seen in Table 8, the order of influence on the softening coefficient of the system was $A > C > B$. As shown in Fig. 7, the softening coefficient of the system increased with

Table 7—Test range results of compressive strength

Index	Extreme difference and average	A	B	C
Compressive strength at 3 days, MPa	k_1	25.2	23.37	27.3
	k_2	23.01	24.32	25.17
	k_3	22.66	25.81	22.86
	k_4	21.8	23.23	20.02
	k_5	21.34	17.26	18.65
	R	3.85	8.55	8.65
Compressive strength at 7 days, MPa	k_1	28.62	27.66	29.87
	k_2	28.96	29.31	30.49
	k_3	27.79	29.35	29.03
	k_4	26.27	27.67	24.15
	k_5	25.12	22.76	23.21
	R	3.83	6.59	7.28
Compressive strength at 28 days, MPa	k_1	34.96	33.6	36.36
	k_2	38.07	34	37.14
	k_3	33.56	35.02	33.96
	k_4	31.59	33.16	31.36
	k_5	30.66	33.05	30.03
	R	7.41	1.96	7.11

Note: 1 MPa = 145 psi.

Table 8—Test range results of softening coefficient

Index	Extreme difference and average	A	B	C
Softening coefficient	k_1	0.59	0.61	0.65
	k_2	0.63	0.63	0.66
	k_3	0.63	0.63	0.66
	k_4	0.64	0.65	0.62
	k_5	0.69	0.66	0.57
	R	0.1	0.05	0.09

increasing PC and FA contents. However, it increased first and then decreased as the HL content rose.

According to Table 9, the order of influence on the water absorption of the system was $C > A > B$. As shown in Fig. 8, the lowest water absorption occurred when the contents of PC, FA, and HL were 20 wt. %, 9 wt. %, and 4 wt. %, respectively.

The experimental findings reveal that the contents of HL and PC are the main factors affecting the performance of the system. The compressive strength, water absorption, and softening coefficient of the system are closely related to its porosity. Lower porosity enhances the system's compactness, resulting in higher compressive strength and reduced water absorption. Furthermore, the increased compactness mitigates structural damage caused by moisture penetration, thereby elevating the softening coefficient. In the early stages of hydration, the strength of the system mainly comes from HG, with the contribution of cement being limited. As hydration progresses, PC and FA undergo hydration, forming a denser microstructure with increasing PC and FA contents. However, when the amounts of PC and FA

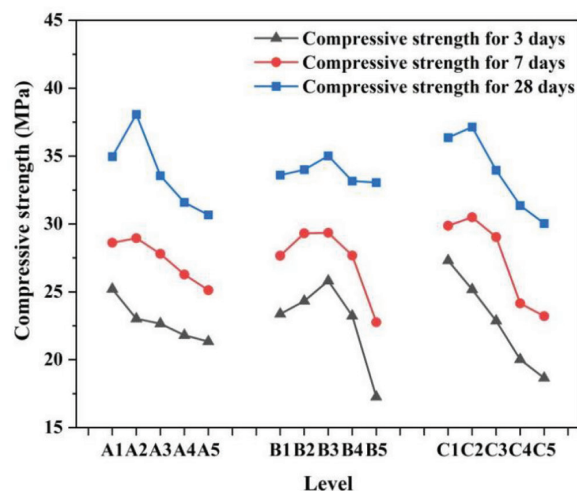


Fig. 6—Relationship between influence factors and average compressive strength. (Note: 1 MPa = 145 psi.)

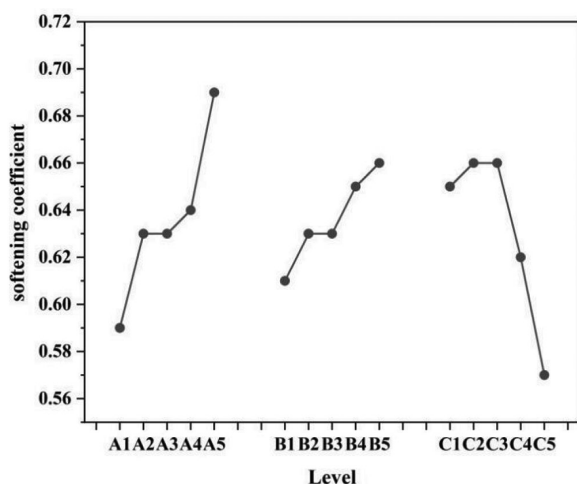


Fig. 7—Relationship between influence factors and average softening coefficient.

become excessive, cracks will occur. In addition, while an appropriate amount of HL promotes the formation of an Aft framework by facilitating Ca^{2+} release from gypsum and cement-derived C_3A , excessive HL induces $\text{Ca}(\text{OH})_2$ supersaturation. This leads to the formation of C-S-H gel with a high Ca/Si ratio and low polymerization degree, which, in turn, increases porosity.

Analysis of variance—Table 10 shows the analysis of variance (ANOVA) results for the performance of the HG-PC-FA-HL system. For the HG-PC-FA-HL system, both the FA and HL contents significantly affect the 3- and 7-day compressive strength. However, for the 28-day compressive strength, softening coefficient, and water absorption, the PC and HL contents have a more significant impact. As observed, the ANOVA results and range analyses are similar, and the performance of the HG-PC-FA-HL system is closely related to HL content.

Performance of gypsum-based cementitious materials

Physical properties of gypsum-based cementitious materials—The ratio of CG0 and CG2 is shown in Table 3. ICMB represents a specimen of the HG-PC-FA-HL system with a ratio of 67 wt. % HG, 20 wt. % PC, 9 wt. % FA, and 4 wt. % HL. As shown in Fig. 9 and 10, the compressive strength and softening coefficient of ICMB were higher than those of CG0 and CG2, and the water absorption rate was lower. When the curing age was 28 days, the compressive strength of ICMB was 41.07 MPa (5955 psi), the softening

coefficient was 0.72, and the water absorption rate after soaking for 48 hours was 12.7%. Compared with the similar products reported by Wan et al.²⁵ and Gou et al.,²⁸ the specimens prepared in this study showed better compressive

Table 9—Test range results of water absorption

Index	Extreme difference and average	A	B	C
Water absorption, %	k_1	14.56	15.18	13.87
	k_2	13.44	15.13	13.51
	k_3	14.93	14.22	14.84
	k_4	15.76	15.14	15.90
	k_5	16.22	15.24	16.79
	R	2.78	1.02	3.28

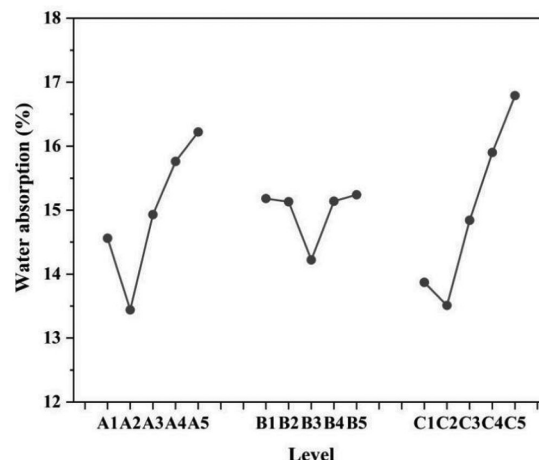


Fig. 8—Relationship between influence factors and average water absorption.

Table 10—Analysis of variance

Dependent variable	Source of variation	Sum of squares	Degree of freedom	Mean square	F	p	Partial η^2
Compressive strength at 3 days	Factor A	44.686	4	11.171	0.958	0.465	0.242
	Factor B	212.876	4	53.219	4.563	0.018*	0.603
	Factor C	253.933	4	63.483	5.443	0.010**	0.645
	Standard error	139.950	12	11.662	—	—	—
	Total sum of squares	651.444	—	—	—	—	—
Compressive strength at 7 days	Factor A	52.644	4	13.161	1.777	0.198	0.372
	Factor B	145.370	4	36.343	4.907	0.014*	0.621
	Factor C	232.075	4	58.019	7.833	0.002**	0.723
	Standard error	88.883	12	7.407	—	—	—
	Total sum of squares	518.972	24	—	—	—	—
Compressive strength at 28 days	Factor A	172.189	4	43.047	6.883	0.004**	0.696
	Factor B	12.588	4	3.147	0.503	0.734	0.144
	Factor C	189.584	4	47.396	7.578	0.003**	0.716
	Standard error	75.052	12	6.254	—	—	—
	Total sum of squares	449.413	24	—	—	—	—
Softening coefficient	Factor A	0.026	4	0.006	4.469	0.019*	0.598
	Factor B	0.008	4	0.002	1.408	0.290	0.319
	Factor C	0.030	4	0.007	5.168	0.012*	0.633
	Standard error	0.017	12	0.001	—	—	—
	Total sum of squares	0.080	24	—	—	—	—
Water absorption	Factor A	23.399	4	5.850	4.928	0.014*	0.622
	Factor B	3.644	4	0.911	0.767	0.567	0.204
	Factor C	37.538	4	9.384	7.905	0.002**	0.725
	Standard error	14.246	12	1.187	—	—	—
	Total sum of squares	78.827	24	—	—	—	—

Note: The asterisks next to some values are statistical significance markers, with the following definitions: $p > 0.05$ (not significant, denoted by *); $0.01 < p \leq 0.05$ (significant, denoted by **); $p \leq 0.01$ (highly significant, denoted by ***).

strength and softening coefficient. Wan et al.²⁵ developed a green binder from flue-gas desulfurization gypsum incorporating PC and large-volume FA. This binder had a softening coefficient greater than 0.6 and a compressive strength of up to 19.4 MPa (2813 psi). Gou et al.²⁸ found that the ternary systems consisting of gypsum, cement, and mineral powder exhibited good water resistance compared with pure gypsum. The softening coefficient and the compressive strength at 28 days reached 0.71 and 24.7 MPa (103 and 3582 psi) at the optimal proportion.

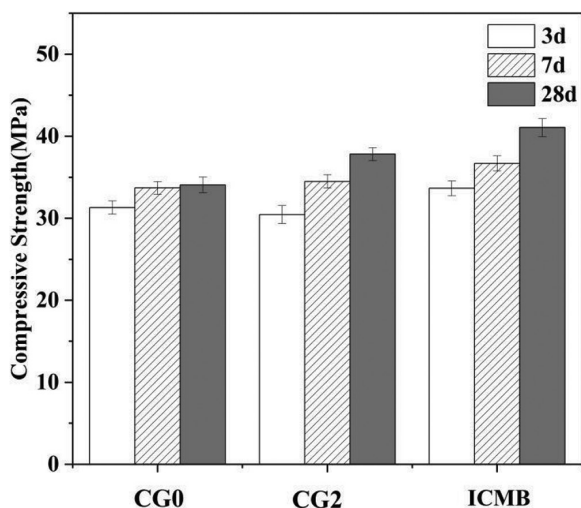


Fig. 9—Compressive strength of modified gypsum. (Note: 1 MPa = 145 psi.)

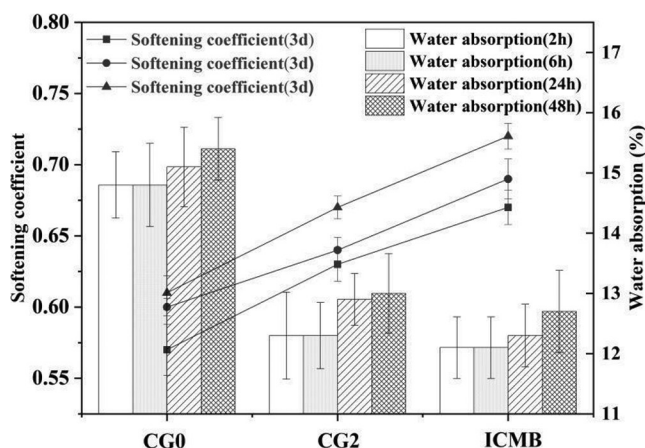


Fig. 10—Water resistance of modified gypsum.

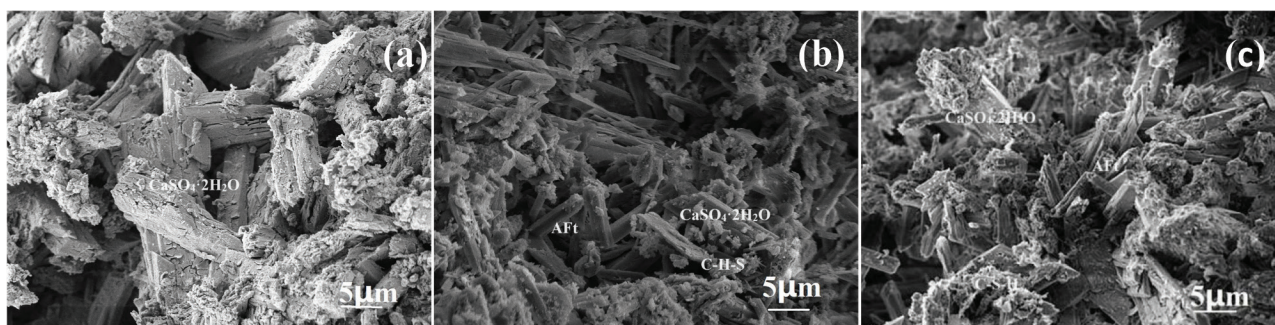


Fig. 11—SEM images of modified gypsum: (a) CG0; (b) CG2; and (c) ICMB.

According to previous literature reports,²⁵ the flocculent C-S-H gels and long, rod-shaped AFt are interwoven together on the surface of gypsum crystals. In this way, a dense network structure is formed, which explains the good water resistance of ICMB. Figure 11(c) in this study also proves this point. The FA in ICMB had pozzolanic activity and a small particle size. The C-S-H gel and AFt were formed by activating the pozzolanic properties of FA, and the activator was HL and $\text{Ca}(\text{OH})_2$ produced during PC hydration. Some of the hydration products of PC and FA filled the pores, improving the compactness of the system. Others wrapped the $\text{CaSO}_4 \cdot 2\text{H}_2\text{O}$ crystals, reducing structural damage caused by water infiltration. The compressive strength and softening coefficient of the system increased, and water absorption decreased.

Microstructural and mineral composition analysis of gypsum-based cementitious materials—The microstructures and XRD patterns of hydration products of CG0, CG2, and ICMB after 28 days of curing are shown in Fig. 11 and 12. The hydration products of CG0 (Fig. 11(a)) were mainly composed of coarse, rod-shaped crystals, which were intertwined to form a hardened structure. When the PC content was 10 wt. %, the long, rod-shaped AFt and the reticular and fibrous C-S-H gel appeared in the hydration products of CG2 (Fig. 11(b)). These hydration products were distributed around the short, rod-shaped $\text{CaSO}_4 \cdot 2\text{H}_2\text{O}$ crystals. This arrangement increases the contact points between the crystals and enhances the compactness of the structure. More tiny, fibrous, and reticular C-S-H gels appeared around the long, rod-shaped AFt and short, rod-shaped $\text{CaSO}_4 \cdot 2\text{H}_2\text{O}$ crystals in the hydration products of ICMB (Fig. 11(c)).

From Fig. 12, it can be seen that the main component of the hydration products of CG0 was $\text{CaSO}_4 \cdot 2\text{H}_2\text{O}$. When PC content was 20%, the diffraction peaks of C-S-H gel and AFt began to appear in CG2, and the diffraction peak intensity of $\text{CaSO}_4 \cdot 2\text{H}_2\text{O}$ decreased. The diffraction peaks of $\text{CaSO}_4 \cdot 2\text{H}_2\text{O}$ in the hydration products of ICMB were lower than those of CG0 and CG2. In contrast, the diffraction peaks of C-S-H gel and AFt were higher than those of CG2. When PC is added, the tricalcium silicate (C_3S) and dicalcium silicate (C_2S) in PC will undergo a hydration reaction to generate C-S-H gel. At the same time, the tricalcium aluminate (C_3A) will immediately react with HG to form AFt crystals. As a result, the amount of $\text{CaSO}_4 \cdot 2\text{H}_2\text{O}$ in the hydration products was reduced compared with HG. In the HG-PC-FA-HL system, FA generated more AFt and C-S-H

gels through the joint excitation of PC, HL, and HG. They interweave with each other to form a denser structure, which improves the strength and water resistance of gypsum.

Reaction mechanism of gypsum-based cementitious materials—The reaction mechanism of modified gypsum is illustrated in Fig. 13. The introduction of PC is one of the essential measures to improve the mechanical properties and water resistance of HG. The hydration products of CG0 were mainly composed of coarse, rod-shaped $\text{CaSO}_4 \cdot 2\text{H}_2\text{O}$ crystals that intertwine to form a hardened structure. When the PC was added, long, rod-shaped AFt and reticular and fibrous C-S-H gel were formed. They either overlapped with the thick, rod-shaped $\text{CaSO}_4 \cdot 2\text{H}_2\text{O}$ crystals or coated the surface of $\text{CaSO}_4 \cdot 2\text{H}_2\text{O}$. This makes the cementitious system structure more compact and reduces the exposure of $\text{CaSO}_4 \cdot 2\text{H}_2\text{O}$ to water. When PC content is low, the formation of C-S-H gel and AFt is limited, which is not enough to greatly improve the performance of HG. However, excessive PC content will generate a large amount of AFt, which causes expansion and cracking.

If better HG performance is desired, introducing FA is a viable choice. The co-introduction of PC and FA can significantly improve the compressive strength and water resistance of HG. The pozzolanic activity of FA is activated by HL and $\text{Ca}(\text{OH})_2$ produced by PC hydration. The tiny C-S-H gel and AFt produced by FA hydration interconnect with

the hydration products of PC. This can prevent water from entering the interior of the specimen, avoid the dissolution of $\text{CaSO}_4 \cdot 2\text{H}_2\text{O}$ crystals, and optimize the pore structure at the same time.

Properties of SiO_2 aerogel composites

The same SiO_2 aerogel slurry with a content of 30 wt. % was added to the ICMB and cement, and their properties were tested. As shown in Fig. 14, after the introduction of the SiO_2 aerogels, the density, thermal conductivity, strength, and water absorption of gypsum-based cementitious materials were 1136.4 kg/m^3 (70.91 lb/ft^3), $0.1889 \text{ W/(m}\cdot\text{K)}$ ($0.1091 \text{ Btu/[h}\cdot\text{ft}\cdot\text{°F}]$), 17.16 MPa (2488 psi), and 20.37% , respectively. Compared with cement, the density, thermal conductivity, and strength were reduced by 30.85% , 63.19% , and 36.98% , respectively. The water absorption was increased by 21.11% . Therefore, gypsum-based cementitious materials are more suitable for the preparation of SiO_2 aerogel inorganic composite materials than cement-based cementitious materials.

Limitations and improvement direction

This study demonstrates that adding cement, FA, and lime improves the mechanical properties and water resistance of the gypsum-based binder. However, it still has several limitations. First, applying the optimal mixture proportions from laboratory-scale experiments to industrial production may be challenging. Issues such as consistency in raw material quality, mixing uniformity, and curing conditions could affect the application. Second, the research focused on short-term mechanical properties and water resistance, but the long-term durability of the composite materials in practical applications remains uninvestigated. Finally, the experimental design prioritized mechanical properties and water resistance as primary evaluation indexes. It fails to explore how higher FA content, within the allowable range of maintaining basic performance, could further enhance the sustainability of the composite material. Future work should integrate industrial by-products to enhance the environmental compatibility of the composite binder. It should also systematically assess the binder's long-term durability under real-world exposure conditions to ensure sustainable application in green construction.

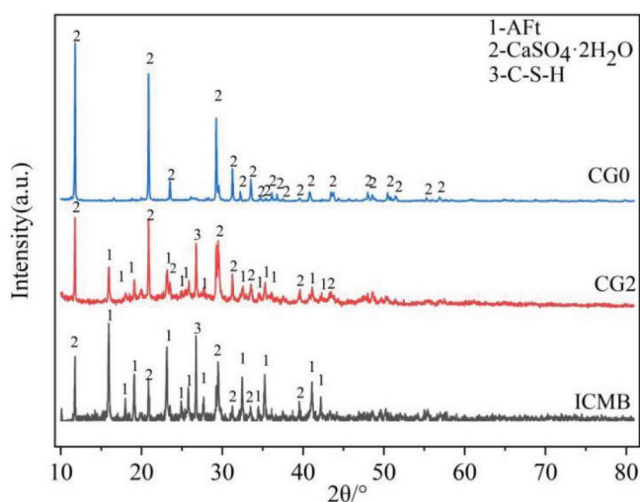


Fig. 12—XRD patterns of inorganic cementitious materials.

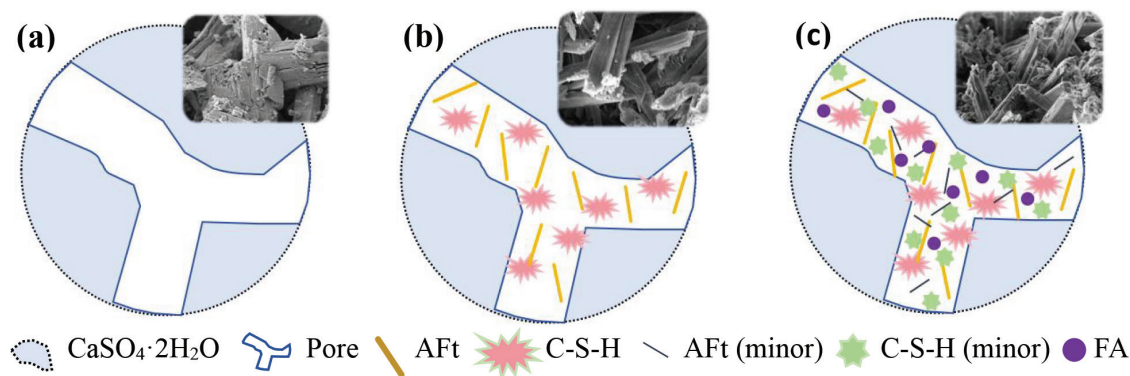


Fig. 13—Reaction mechanism of modified gypsum: (a) CG0; (b) CG2; and (c) ICMB.

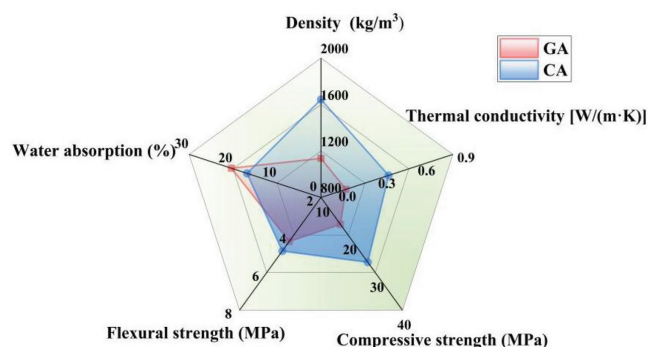


Fig. 14—Properties of SiO₂ aerogel composites. (Note: 1 MPa = 145 psi; 1 W/(m·K) = 0.5778 Btu/(h·ft·°F); 1 kg/m³ = 0.0624 lb/ft³.)

CONCLUSIONS

This study analyzes the synergistic effects in the hemihydrate gypsum (HG)-portland cement (PC) system and HG-PC-fly ash (FA)-hydrated lime (HL) system. Through this experiment, the following conclusions can be drawn:

1. Adding a certain amount of PC can effectively improve the performance of HG. When the PC content was 20 wt. %, the 28-day compressive strength of the HG-PC system was higher than that of the other specimens, while the water absorption was lower. The softening coefficients of HG-PC at the early stage of hydration showed a trend of first increasing and then decreasing with the increase in PC content. When the curing age was 28 days, the softening coefficient of HG-PC increased with the rise in PC content.

2. Compared with the HG and HG-PC systems, the HG-PC-FA-HL system showed better mechanical properties and water resistance. When the FA content was 9 wt. %, PC was 20 wt%, and HL was 4 wt. %, the 28-day compressive strength reached 41.07 MPa (5955 psi). After soaking for 48 hours, the water absorption was 12.7%, and the softening coefficient was 0.72.

3. The study examined the evolution of hydration products and microstructural development in both the HG-PC and HG-PC-FA-HL systems. It also explored how these changes relate to mechanical properties and water resistance. The mechanical properties and water resistance of the system are determined by the filling ratio of ettringite (AFt) and calcium-silicate-hydrate (C-S-H) gel within the intercrystalline pores of CaSO₄·2H₂O. Their coverage on the surface of CaSO₄·2H₂O crystals also plays a key role. FA exhibits both pozzolanic activity and fine particle-size characteristics. Upon activation, FA facilitates the formation of C-S-H gel and AFt phases. These reaction products enhance the microstructure of the HG-PC-FA-HL system, thereby improving its mechanical performance and water resistance.

4. After incorporating SiO₂ aerogel, the gypsum-based cementitious materials exhibited a 30.85% reduction in density, 63.19% decrease in thermal conductivity, and 36.98% decline in strength compared to cement. Meanwhile, their water absorption increased by 21.11%. Therefore, gypsum-based cementitious materials are more suitable for the preparation of SiO₂ aerogel inorganic composite materials than cement-based cementitious materials.

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Synergizing Ultra-High-Performance Concrete and Conventional Concrete for Economical and Sustainable Three-Dimensional-Printed Composite Section

by A. Tripathi, S. Surehali, A. S. Nimbalkar, B. Mobasher, and N. Neithalath

Ultra-high-performance concrete (UHPC) is composed of a high volume fraction of binder and steel fibers, and a very low water content, resulting in enhanced strength and ductility along with higher cost and environmental impacts. This study develops a UHPC mixture amenable for three-dimensional (3-D) printing, with 30% of cement (by mass) replaced with a combination of replacement materials. The proportioned UHPC mixture with 1.5% fiber volume fraction demonstrates 28-day compressive strengths of >120 MPa (17.4 kip), and limited anisotropy when tested in the three orthogonal directions. Furthermore, 3-D-printed layered composites are developed where UHPC (with and without fiber reinforcement) and conventional concrete layers are synergistically used in appropriate locations of the beam to achieve mechanical performance that is comparable to 3-D-printed UHPC sections. Such manufacturing flexibility offered by 3-D printing allows conserving resources and attaining desirable economic and environmental outcomes, as is shown using life cycle and techno-economic analyses (LCA/TEA). Experimental and theoretical analyses of load-carrying capacity and preliminary LCA/TEA show that >50% of the fiber-reinforced UHPC beam volume (in the compression zone) can be replaced with conventional concrete, resulting in only a <20% reduction in peak load-carrying capacity, but >35% reduction in cost and >20% reduction in CO₂ emissions. These findings show that targeted layering of different materials through 3-D printing enables the development and construction of 3-D-printed performance-equivalent structural members with lower cost and environmental impacts.

Keywords: composite sections; economic structure; sustainable construction; three-dimensional (3-D)-printed concrete; ultra-high-performance concrete (UHPC).

INTRODUCTION

Three-dimensional (3-D) printing of concrete is an emerging technology where rapid advances in materials development and automation, and efficient integration with construction processes, are paving the way for its enhanced adoption in construction. The selection of appropriate materials and printing parameters is crucial toward attaining desirable properties of 3-D-printed elements.¹⁻⁷ Reinforcing the printed elements with fibers, textiles, or particulates,^{4,8-10} especially because layer-wise printing does not lend itself well to conventional reinforcement strategies, and reducing the self-weight and controlling volume changes and cracking using lightweight aggregates^{11,12} have been explored as potential methods to enhance the properties of 3-D-printed components. The use of cement replacement materials and cement-free concretes (for example, alkali-activated

materials) as approaches to enhance the sustainability of 3-D-printed concretes is also well explored.^{13,14}

While most studies related to 3-D printing of concrete use conventional mortars or concrete, the unique attributes of the technology can be beneficially employed in shape- and topology-optimized structures and/or for specific high-performance applications (for example, blast and impact resistance, targeted repair, and so on). This often requires the development of material formulations with improved strength and toughness. Fiber reinforcement is typically used to enhance toughness, but the lower strengths of 3-D-printed concrete (generally attributed to a lack of consolidation as well as the presence of weaker inter-layer and inter-filament interfaces^{15,16}) also need to be mitigated. It is in this context that ultra-high-performance concrete (UHPC) can be an appropriate material for 3-D-printing applications. UHPC is proportioned with carefully selected materials that synergize to deliver a high degree of particle packing and improved reactivity, a very low water-binder ratio (w/b), and generally incorporates metallic fiber reinforcement.^{17,18} The preferential alignment of steel fibers in 3-D-printed UHPC layers is noted to significantly enhance its flexural strength and toughness.^{19,20} The low w/b and the high volume of fibers present significant challenges in 3-D printing with UHPC.^{21,22} Moreover, the need for high cement, admixture, and fiber contents make UHPCs more expensive and carbon-intensive, even though otherwise unattainable mechanical performance can be achieved. For the enhancement of flexural capacity, the replacement of lower-strength material with higher-strength material in the tensile section of the beam has been explored in previous studies.²³ In the case of UHPC, the opposite of this—that is, the substitution of part of the compression section—offers a promising approach to reduce costs and associated carbon emissions while still striving to attain identical mechanical response.

This paper explores the development of 3-D-printable UHPC mixtures where 30% of the cement is replaced by cement replacement materials such as fly ash, slag, and limestone powder, to make 3-D-printed UHPC more cost-effective and sustainable. In addition, to make structural

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Table 1—Chemical composition and physical properties of mortar components

Components of binders	Chemical composition, % by mass							d_{50} , μm (mils)	Specific gravity
	SiO ₂	Al ₂ O ₃	Fe ₂ O ₃	CaO	MgO	SO ₃	LOI		
Cement (OPC)	19.60	4.09	3.39	63.21	3.37	3.17	2.54	12.6 (0.5)	3.15
Slag (S)	36.00	10.50	0.67	39.80	7.93	2.10	3.01	7.9 (0.3)	2.07
Fly ash (F)	54.93	20.44	4.57	9.93	2.81	0.86	0.99	22.4 (0.9)	2.28
Limestone (L)	CaCO ₃ > 99%							1.5 or 3.0 (0.06 or 0.12)	2.70
Medium sand (M)	SiO ₂ > 99%							600 (23.6)	2.40

Note: LOI is loss on ignition.

Table 2—Properties of fibers used in study

Type of fibers	Diameter, mm (mils)	Length, mm (mils)	Specific gravity	Tensile strength, GPa (ksi)	Young's modulus, GPa (ksi)
Short steel fiber (SF)	0.20 (8)	13 (512)	6.80	3.00 (435)	210 (30,458)

systems using UHPCs more sustainable and economical, the formulated UHPC mixtures are synergized with conventional 3-D-printed concrete mixtures to develop layered composite systems that perform comparably in flexure to that of 3-D-printed UHPC specimens. This is predicted by the fact that 3-D printing allows the use of targeted materials in requisite layer heights in different locations of the beam, enabling the development of layered multi-material composite systems that are likely to be more cost- and carbon-efficient, yet not compromising the performance. Life cycle and techno-economic analyses (LCA/TEA) demonstrate the beneficial impacts of such 3-D-printed layered composite systems as compared to traditional UHPCs as well as conventional 3-D-printed concrete solutions.

RESEARCH SIGNIFICANCE

The rationale behind this study is to demonstrate that using multiple materials in 3-D-printed concrete sections, rather than a single type, offers a cost-effective and sustainable way to enhance mechanical performance. In this work, the flexural behavior of an ultra-high-strength mixture (UB) is compared to a composite section where UB is partially replaced with a normal-strength mixture (L₃₀). Replacing 50% of UB with L₃₀ reduces peak flexural load by ~20% but lowers cost by ~35% and CO₂ emissions by ~20%.³ Experimental results and analysis confirm the feasibility of combining UHPC with normal-strength mixtures for stronger, more sustainable construction.

EXPERIMENTAL PROGRAM

Materials and mixtures selection

The experimental program was devised to design UHPC mixtures (defined here as mixtures with 28-day compressive strength ≥ 100 MPa [14.5 ksi], though the conventional definition of UHPC is somewhat different) that fulfill the printability requirement (extrudability and buildability). The materials used in this study included a Type I/II ordinary portland cement (OPC) conforming to ASTM C150/C150M, Class F fly ash (F) conforming to ASTM C618, ground-granulated blast-furnace slag (GGBFS) conforming to ASTM C989/C989M, limestone powder (L) with two different median particle sizes (d_{50}) (1.5 and 3.0 μm [0.06 and 0.12 mils])

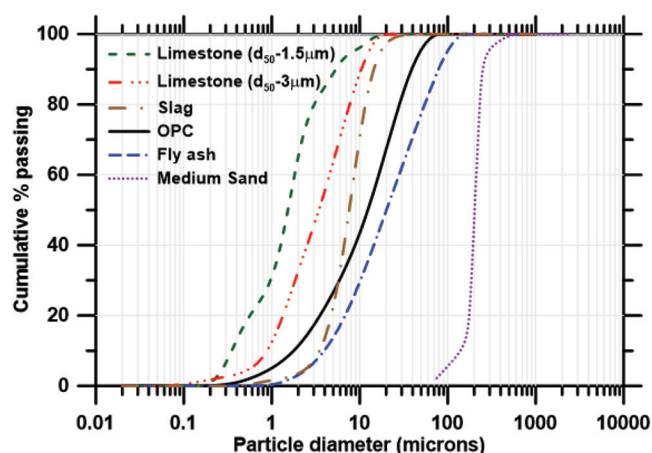


Fig. 1—PSD of mortar components.

conforming to ASTM C568/C568M, and commercial sand (M), having a median particle size of 0.60 mm (23.6 mils), conforming to ASTM C778. Table 1 shows the chemical and physical properties of the different binder components used. Short steel fibers (SF), the properties of which are shown in Table 2, are used as fiber reinforcement. All the mixtures used a polycarboxylate ether-based high-range water-reducing admixture (HRWRA). The particle-size distribution (PSD) of the materials used is shown in Fig. 1.

Several unreinforced mixtures were evaluated to obtain an ideal matrix for the UHPC mixture (details of which are provided in the Appendix*), with a 30% (by mass) replacement of cement by a combination of cement replacement materials based on extensive past work.^{17,24} Table 3 shows the mixture proportions for all the final selected UHPC mixtures, plain and fiber-reinforced (hereinafter referred to as UB or UB-SF_{1%} and UB-SF_{1.5%}, respectively; w/b of 0.18 and HRWRA dosage of 1.1%).

Mixing and 3-D printing

A high-shear rotating pan mixer was used to prepare the UHPC mixtures. All the powder constituents were mixed for

*The Appendix is available at www.concrete.org/publications in PDF format, appended to the online version of the published paper. It is also available in hard copy from ACI headquarters for a fee equal to the cost of reproduction plus handling at the time of the request.

Table 3—Mixture proportions used in study

Mixture ID	Mass fraction of ingredients					Short steel fiber (SF)*
	Cement (OPC)	Fly ash (F)	Limestone ($d_{50} = 1.5 \mu\text{m}$) (L_f)	Slag (S)	Sand (M)	
UB	0.42	0.06	0.06	0.06	0.40	—
UB-SF _{1%}	0.42	0.06	0.06	0.06	0.40	1.0
UB-SF _{1.5%}	0.42	0.06	0.06	0.06	0.40	1.5

*Percentage by volume of solids in mixture.

Note: w/b of 0.18 is used for both cast and printed mixtures; HRWRA dosage used was 1.1% by mass of binder for 3-D-printed mixtures and 1.5% for companion cast mixtures.

5 minutes to obtain a homogeneous mixture. Once the ingredients were mixed, water (mixed with HRWRA) was slowly added to the mixture while the mixer was still rotating. Mixing continued for 8 minutes, after which the top lid was opened and the mortar adhering to the sides of the mixer was scraped. Mixing then continued for another 5 minutes, after which the mixture was transferred to the printer. In the case of a fiber-reinforced mixture, fibers were added through the trapdoor just after introducing the required amount of water. Because the workability requirements are different for conventional mold-cast and 3-D-printed specimens, an additional 0.4% of HRWRA (by mass of the binder) was added to the mold-cast mixtures so that they would be uniformly formed into beams and cubes without having to resort to changes in w/b .

A gantry-based printer equipped with a screw extrusion system was used to print the mixtures shown in Table 3. A circular nozzle diameter (ND) of 20 mm (0.79 in.) and an in-plane print speed of 50 mm/s (1.97 in./s) were used, in line with several of the authors' previous works.^{1,25} A layer height (LH) of 10 mm (0.39 in.), which is close to the fiber length, was selected for all the prints in this study, based on the authors' past work.¹ The stepper for the screw extruder was calibrated for a flow rate of ~10 mL/s (0.34 fl oz.) to obtain uniform filaments with LH of 10 mm (0.39 in.) ($0.5 \times \text{ND}$) and layer width (LW) of 20 mm (0.79 in.) (equal to ND). The mortars were printed into 400 x 400 x 60 mm (15.75 x 15.75 x 2.36 in.) slabs. The printed slabs were moist-cured in a curing chamber at $23 \pm 2^\circ\text{C}$ ($73 \pm 3.6^\circ\text{F}$) and >98% relative humidity (RH), after which they were cut into prisms and beams for compression and flexural tests using a diamond-tipped saw.

Printing of layered composite UHPC specimens

To evaluate the behavior of composite UHPC conventional concrete printed specimens in flexure, slabs of size 400 x 400 x 60 mm (15.75 x 15.75 x 2.36 in.) were printed with 10 mm (0.39 in.) LH filaments and 20 mm (0.79 in.) LW. Initially, the lower 30 mm (1.18 in.)—that is, three layers—were printed with the chosen UHPC binder with or without 1.5% by volume of steel fibers (UB and UB-SF_{1.5%}; refer to Table 3). Then, the auger head containing the UHPC binder was replaced by another auger head containing a conventional 3-D-printable mixture, which had a 30% limestone-replacing cement (referred to as L_{30} ; used in many of the authors' previous studies on 3-D printing^{1,25}). The switching of the auger head was carried out with minimal time intervals (~2 minutes) to ensure that cold joints were avoided. Finally, the remaining upper three layers were printed with

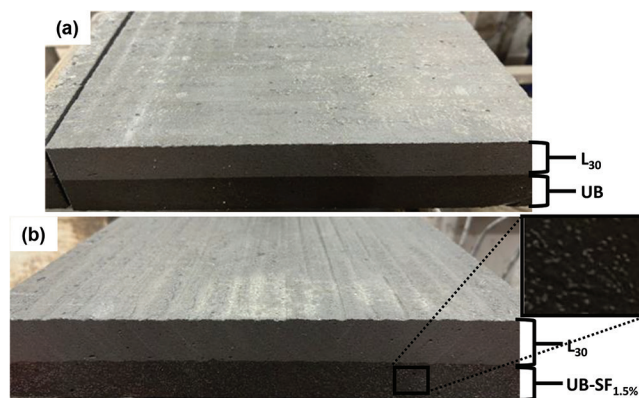


Fig. 2—Composite slabs consisting of: (a) L_{30} mixture and unreinforced UB (CS); and (b) unreinforced L_{30} and fiber-reinforced UB-SF_{1.5%} (CS_F). L_{30} section is lighter in color due to use of large volume of limestone powder. Light-colored regions in UHPC section, highlighted in (b), correspond to steel fibers. Note that L_{30} mixture is not fiber-reinforced.

the L_{30} mixture. The paste content for the L_{30} mixture was maintained at 50% (as opposed to the UB mixtures with 60% paste content) and w/b at 0.35. This 3-D-printed mixture was observed to attain a 28-day compressive strength of ~70 MPa (10.15 ksi) in its strongest direction and ~50 MPa (7.25 ksi) in the weakest direction. The beams were cured for 28 days in a curing chamber at $23 \pm 2^\circ\text{C}$ ($73 \pm 3.6^\circ\text{F}$) and >98% RH. Figure 2 shows the printed composite slabs. The printed composite section consisting of layers made of the unreinforced UB mixture and unreinforced L_{30} mixture is hereinafter referred to as CS, and those consisting of the fiber-reinforced UB-SF_{1.5%} and unreinforced L_{30} as CS_F. Note that the CS_F section contains only half the volume of fibers as in the UB-SF_{1.5%} beam.

Compression testing in three orthogonal directions

Prisms of size 50 x 50 x 60 mm (1.97 x 1.97 x 2.36 in.) were prepared by sectioning the 3-D-printed UHPC slab. Each prismatic specimen consists of three filaments (each of 20 mm [0.79 in.] width) adjacent to each other, and five layers (LH of 10 mm [0.39 in.]). The prisms were subjected to uniaxial loading using a servo-controlled testing machine with a maximum load capacity of 489 kN (110 kip). Compression tests were carried out after 7, 14, and 28 days of curing, with the printed samples tested in all three directions. Direction-1 (D1) is along the direction of printing, direction-2 (D2) is along the direction of layer buildup, and

direction-3 (D3) is perpendicular to D1 and D2. More details, including an extensive description of testing in orthogonal directions, have been provided in the authors' earlier work.²⁵ Companion 50 x 50 x 50 mm (2 x 2 x 2 in.) cubes were also cast and tested in compression. Loading was carried out at a stroke displacement rate of 0.125 mm/min (5 mils/min) (cast, D1, and D2) or 0.15 mm/min (6 mils/min) (D3), corresponding to an initial strain rate of 0.25%/min.

Flexural testing and digital image correlation

Beams of 60 x 60 x 300 mm (2.36 x 2.36 x 11.81 in.) size were cut from the slab and tested in flexure after 28 days of curing, with a span length of 240 mm (9.45 in.). The filament print direction was oriented along the longer side of the beam. One of the longer faces (300 x 60 mm [11.81 x 2.36 in.]) parallel to both the print and the layer buildup direction was painted white and then speckled with black dots to prepare the specimen for digital image correlation (DIC)-based strain analysis. Cast beams measuring 60 x 60 x 300 mm (2.36 x 2.36 x 11.81 in.) were also used for comparison. The flexural response was obtained per ASTM C78/C78M-18²⁶ under four-point bending. A servo-controlled testing machine with a capacity of 100 kN (22 kip) was used, with loading applied on the middle-third of the span of the beams. A minimum of six beams were tested for each mixture. The test was carried out under midpoint deflection control using linear variable differential transformers (LVDTs) at a rate of 0.004 mm/min (0.16 mils/min) until failure occurred. For mixtures containing steel fibers, the beams were initially tested at a midpoint deflection rate of 0.004 mm/min (0.16 mils/min) until first cracking, after which the rate was switched to 0.24 mm/min (9.45 mils/min) until a total midpoint deflection of 3.75 mm (0.15 in.). Beam samples (60 x 60 x 300 mm [2.36 x 2.36 x 11.81 in.]) extracted from composite slabs (CS and CS_F) were also subjected to flexural loading after 28 days of curing.

Flexural testing of the composite sections was carried out with the UHPC layers on either the bottom or top of the beams, hereinafter referred to as Configuration A (UHPC layers predominantly in tension) or Configuration B (UHPC layers predominantly in compression), respectively. 3-D-DIC testing for beams' surface displacement and strain fields consisted of two cameras focused on a speckled area of interest, illuminated by high-intensity floodlights. The frequency of image capture was fixed at 1 Hz. The process of obtaining Lagrangian strain fields from the displacement field captured by DIC is described in detail elsewhere.^{27,28}

RESULTS AND DISCUSSION

Compressive strength of 3-D-printed UHPC mixtures in orthogonal directions

As discussed earlier, uniaxial compressive tests for the mixtures shown in Table 3 were carried out on 50 x 50 x 60 mm (1.97 x 1.97 x 2.36 in.) printed prisms and 50 mm (2 in.) cast cubes after 7, 14, and 28 days of curing. For the 3-D-printed specimens, strength determination in the three orthogonal directions helps quantify the anisotropy in

compression. At least six samples were tested in each direction for each age, and the results are shown in Fig. 3.

Previous studies have reported that the 3-D-printed samples demonstrate lower compressive strength as compared to cast samples,²⁹⁻³¹ which is generally attributed to the presence of weaker inter-filament and inter-layer interfaces,¹⁵ and improper compaction due to lack of vibration or consolidation,¹⁶ consequently resulting in higher overall specimen porosity.^{15,16,32} However, the 3-D-printed UHPC mixtures shown in Table 3 show comparable strengths to the companion mold-cast mixtures at all ages, as shown in Fig. 3. The higher strength of the mixtures, aided by a lower w/b and thus reduced porosity, along with appropriate extrudability and buildability, aided by careful particle packing^{17,24,33} and high admixture dosage, likely helps in a more consistent microstructure for the 3-D-printed mixtures. The enhanced fluidity of these mixtures, even at a low w/b , results in superior inter-filament and inter-layer interfaces in the UHPC mixtures as compared to the more porous interfaces in less workable (stiffer) mixtures. This is shown in Fig. 4 using optical microscopy images. The non-zero standoff distance used in this study further helps to enhance the inter-layer strength as well as the consolidation of the filaments. Figure 4(a) shows a representative image of an unreinforced UHPC specimen (UB mixture), with the solid white line indicating the inter-layer interface and the dotted line indicating the inter-filament interface. These interfaces are indistinguishable except for a small void along the inter-filament interface and closer to the inter-layer interface. The surface profile map shown in Fig. 4(b) does not identify any significant voids in the bulk region. On the contrary, the image of an L_{30} specimen shown in Fig. 4(c) and its corresponding surface profile in Fig. 4(d) show higher porosity both in the inter-filament and inter-layer interfaces.

While the unreinforced mixtures (UB) attain compressive strengths of ~100 MPa (14.5 ksi) after 28 days, fiber-reinforced specimens—both having 1 or 1.5% by volume of steel fibers—attain ~120 MPa (17.4 ksi) compressive strengths. The fiber-reinforced specimens attain ~100 MPa (14.5 ksi) even in the 7-day cured samples. In a previous work by the authors, a smaller volume fraction of steel fibers (~0.3%) was found to result in similar strengths as that of their unreinforced counterparts.²⁵ However, at higher fiber volume fractions such as those used in this work, the presence of steel fibers enhances the compressive strength compared to their unreinforced counterparts (note that fibers are used in UHPCs to primarily enhance their tensile and flexural properties, though crack bridging and confinement effects at higher volume fractions result in increased compressive strengths). The volume fraction of fibers used in this work, despite being lower than the generally recommended dosage (~3%) needed to enhance the performance of UHPCs in compression,^{34,35} is noted to bring beneficial effects; such observations are also not uncommon.³⁶ While cast-in-place UHPCs designed and implemented by the authors^{24,37,38} for bridge deck connections and overlays consisted of up to 3% of fibers by volume, this study limits the fiber volume to 1.5% to eliminate issues with extrudability during printing.

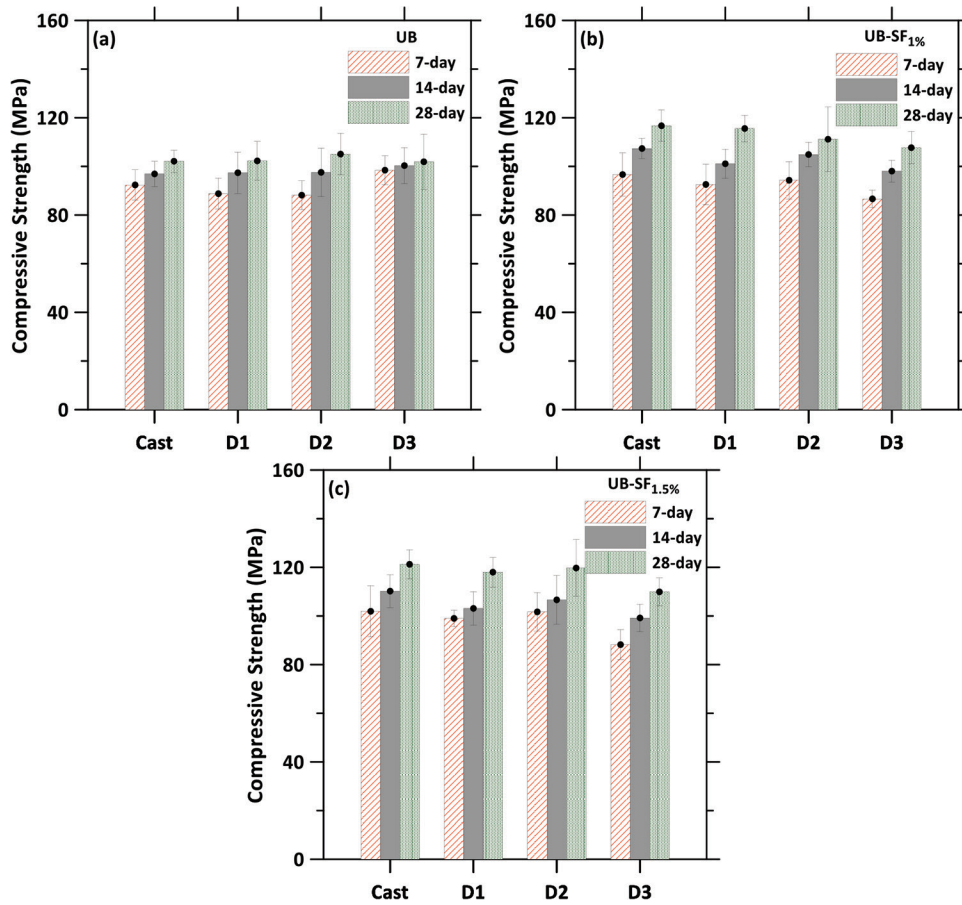


Fig. 3—Compressive strength development for: (a) UB; (b) UB-SF_{1%}; and (c) UB-SF_{1.5%} mixtures when tested in three orthogonal directions. (Note: 1 MPa = 145 psi.)

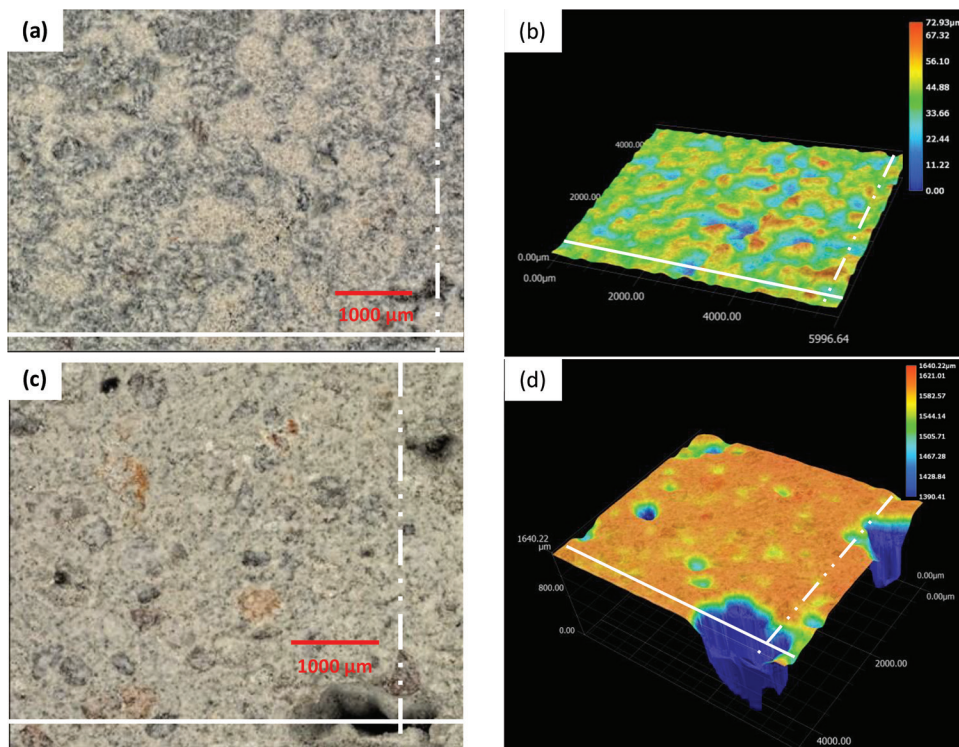


Fig. 4—(a) and (b) Representative optical images of UB specimen and surface profile map that help identify pores in bulk and interfacial regions; and (c) and (d) corresponding images for L₃₀ 3-D-printed specimen. Solid white line indicates inter-layer interface, and dashed line represents inter-filament interface. All images were taken at 40× magnification.

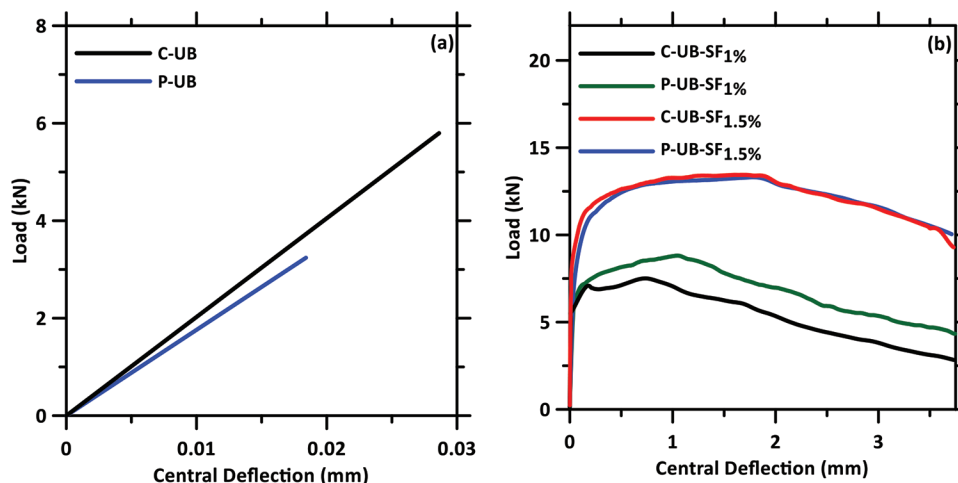


Fig. 5—Representative load-deflection response of cast and printed specimens for: (a) UB; and (b) UB-SF_{1%} and UB-SF_{1.5%} (note that x-axis and y-axis limits are different in two figures); ‘C-’ represents cast specimen and ‘P-’ represents printed specimen. (Note: 1 kN = 224.9 lbf; 1 mm = 0.039 in.)

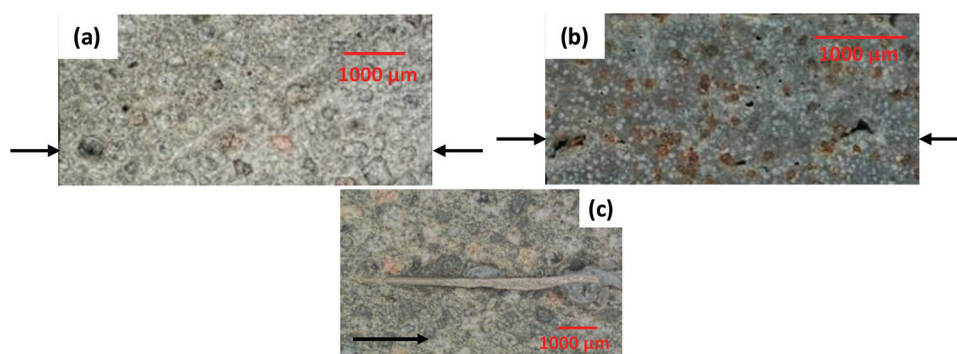


Fig. 6—Representative cross section of: (a) UB; and (b) UB-SF_{1.5%} mixtures along direction of layer buildup (black arrow indicating location of inter-layer interface); and of (c) UB-SF_{1.5%} mixtures along print direction (black arrow indicating print direction). UB mixture does not show distinct voids along inter-layer interface, while they are distinctly visible for UB-SF_{1.5%} mixture. Bulk filament for UB-SF_{1.5%} mixture also shows more pores compared to UB.

Flexural response of 3-D-printed UHPC beams and composite sections

3-D-printed UHPC beams in four-point bending—Flexural tests were performed, as described in the “Flexural testing and digital image correlation” section. Figure 5 shows the representative load-deflection responses obtained from the four-point bending test.

Figure 5(a) shows that the 3-D-printed unreinforced UHPC mixture shows lower peak loads than its cast counterparts in flexure, contrasting with its response in compression reported in the earlier section. The plausible reason for this behavior is that smaller voids—both in the bulk and inter-layer regions—that are present in the 3-D-printed samples are likely not critical in compression,³⁹ while they are potential nucleation points for crack extension under tensile stresses. When fiber-reinforced mixtures are 3-D-printed, small voids exist at the interface (note that voids are relatively absent at the interface in the UB mixture shown in Fig. 6(a), while UB-SF_{1.5%} shown in Fig. 6(b) shows significant inter-layer voids), yet the fibers provide more resistance in flexure because they are aligned perpendicular to the direction of the applied load (as shown in Fig. 6(c) and also

reported elsewhere^{36,40}), as opposed to those in cast samples where the fibers are randomly oriented.³⁶

The midpoint deflection for the 3-D-printed beams is smaller than its cast counterpart for the corresponding unreinforced specimens. As expected, steel fibers significantly enhance the ductility, as shown in Fig. 5(b). Even at a deflection of 3 mm (0.12 in.) (100 times the failure deflection of the unreinforced specimens), 50 to 80% of the peak strength is retained, depending on the fiber volume fraction. An interesting observation is that, at a fiber volume fraction of 1%, the 3-D-printed samples perform better in flexure than the companion fiber-reinforced cast specimens. The aligned nature of fibers results in more fibers (and higher fiber lengths) being engaged in load transfer in the 3-D-printed case in the tested direction. When the fiber volume increases to 1.5%, the beneficial attribute is not observed, plausibly due to the compromise in print material quality due to higher fiber content. In either case, it can be safely stated that the flexural performance is not compromised compared to cast specimens when UHPC mixtures containing steel fibers up to 1.5% by volume are 3-D-printed. Interface defects that generally play a predominant role in lowering the mechanical performance of 3-D-printed concrete are countered in

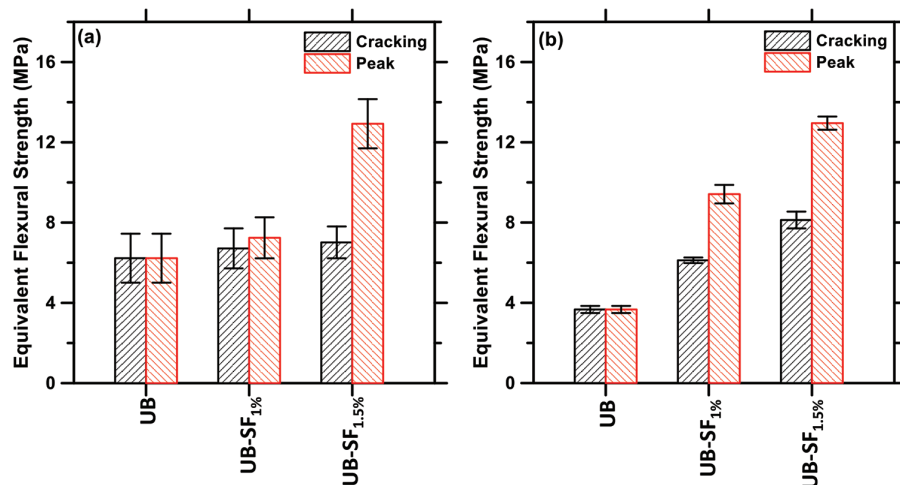


Fig. 7—Equivalent flexural strength at cracking and peak capacity for different mixtures: (a) cast specimens; and (b) printed specimens; cracking strength and peak strength are different only for ductile beams (steel fiber-containing mixtures). (Note: 1 MPa = 145 psi.)

this case, enabling the use of 3-D-printed UHPC mixtures in unique configurations, as shown in a later section.

The average flexural strength at cracking and peak load-carrying capacity for the different UHPC beams are shown in Fig. 7(a) for the mold-cast samples and Fig. 7(b) for the 3-D-printed samples. For the brittle beams (UB), cracking strength and peak strength are obviously the same. Using fibers in 3-D-printed specimens can be employed as a strategy to mitigate strength loss due to the presence of interfaces when elements are 3-D-printed; this is depicted in Fig. 7. While the flexural strength is ~45% lower for the 3-D-printed unreinforced mixture (UB) than the cast counterpart, the strength reduction is mitigated when reinforced with steel fiber or, in some cases, reversed. In the experiments carried out herein, the 3-D-printed UB-SF_{1.5%} mixture shows similar strength as its cast counterpart, while the 3-D-printed UB-SF_{1%} mixture shows higher flexural strengths than its cast counterpart. The fibers are aligned perpendicular to the direction of testing (Fig. 6(c)), as discussed earlier, resulting in this observation. The use of 1% steel fibers by volume enhanced the flexural strength in printed samples by ~170% as compared to the unreinforced sample, and at 1.5% fiber volume fraction, by ~270%.

Figure 8 shows representative Lagrangian strain profiles at different instances during the flexural test for the 3-D-printed beams. At the peak load, the maximum strain is 0.1% for the unreinforced beam (Fig. 8(a)). Figures 8(b) and (c) depict the strain profiles at the peak load and at a central deflection of 3.75 mm (0.15 in.) for the UB-SF_{1%} specimens. Note that the Lagrangian strain at the peak load is approximately two orders of magnitude higher than that of the unreinforced specimen, and the crack deflection and branching effects of fibers have been mobilized. While there are multiple zones of strain concentration before cracking, the dominant crack is found to have localized in the fiber-reinforced beams by the time of peak load. At the end of the test (3.75 mm [0.15 in.] deflection), the maximum strain recorded using DIC is more than three times that at the peak, indicating significant ductility—more than what is typically noted in

randomly oriented short steel fiber-reinforced concrete^{41,42}—which is attributed to the preferential orientation of fibers. The beam retains >50% of its peak load-carrying capacity at this deflection. The behavior of the UB-SF_{1.5%} specimens is similar, as shown in Fig. 8(d) and (e), albeit with an even greater enhancement in strains and post-peak load-carrying capacity, attributed to the higher volume of steel fibers. The post-peak DIC images of the steel fiber-reinforced beams (Fig. 8(c) and (e)) show sizeable crack openings and very high Lagrangian strains. Crack branching observed in these figures is most likely due to the initiation of compression failure prompted by the large deformations rather than branching or deflection of the crack, due to layer interfaces seen in beams where the layer interface is significantly weaker than those in the UHP beams described here. Steel fibers arrest crack growth at higher volume fractions by temporarily bridging cracks, leading to multiple cracking and branching.^{43,44} Multiple cracking for UHPC at similar volume fractions of steel fibers has also been observed in the case of cast UHPC specimens.^{24,43}

Composite beams—3-D-printed UHPC combined with conventional 3-D-printed concrete—One of the major advantages of 3-D printing is that multiple materials can be combined in unique geometrical configurations to exploit beneficial attributes of the components; several examples can be found in Tripathi et al.,²⁵ Edelen and Bruck,⁴⁵ and Mansouri et al.⁴⁶ Here, 3-D-printed UHPC is synergized with normal-strength 3-D-printed concrete in layered configurations to obtain performance-, cost- and CO₂-optimized structural elements as compared to traditional UHPC systems. As discussed in the “Printing of layered composite UHPC specimens” and “Flexural testing and digital image correlation” sections, composite sections constituting multiple layers of L₃₀ mixture and multiple layers of UB or UB-SF_{1.5%} mixtures were tested in flexure under Configurations A or B. Test results are shown here for the cases where the L₃₀ and UB binders each occupy three layers of 10 mm (0.39 in.) layer height. Figure 9 shows the representative load-deflection response obtained from the four-point bending test of the

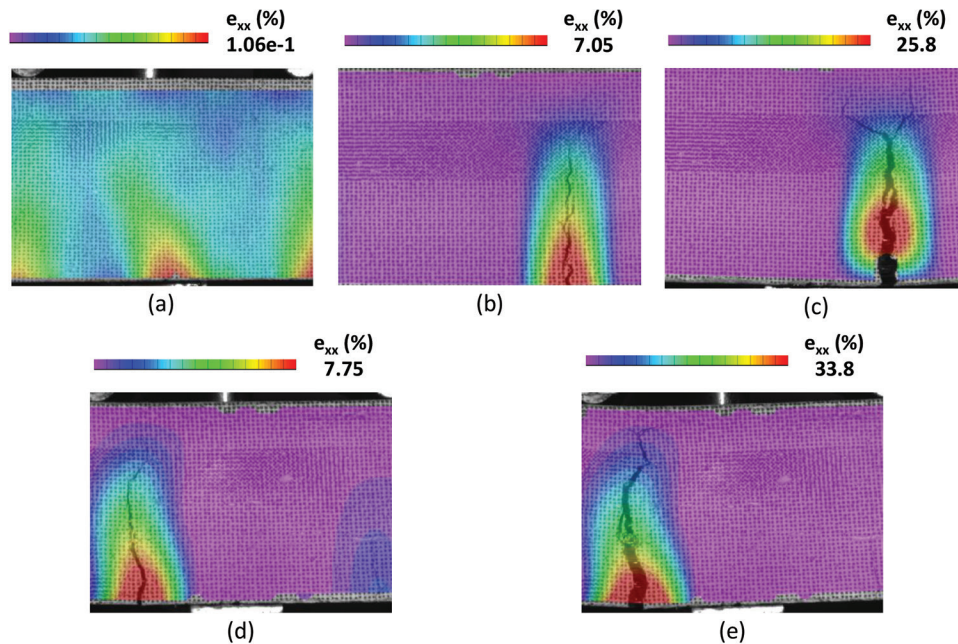


Fig. 8—Representative Lagrangian strain profiles in middle-third span of beam for: (a) UB at peak; (b) UB-SF_{1%} at peak; (c) UB-SF_{1%} at 3.75 mm (0.15 in.) central deflection; (d) UB-SF_{1.5%} at peak; and (e) UB-SF_{1.5%} at 3.75 mm (0.15 in.) central deflection. Note that lowest strain value is set to -6.00×10^{-2} for all figures.

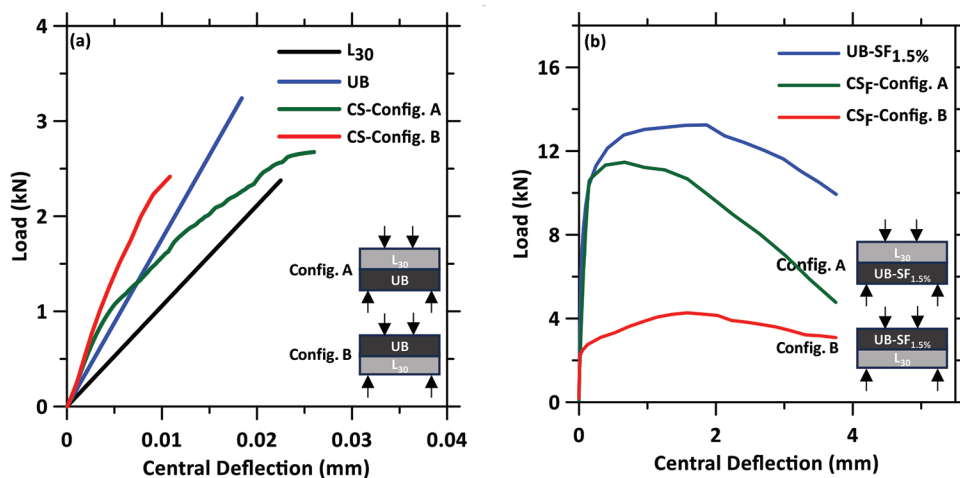


Fig. 9—Load-deflection response of printed composite section for: (a) CS; and (b) CS_F; both print types contained L₃₀ as normal-strength mixture. Configurations A and B indicate different locations of mixtures in beam as shown. Note that axis limits are different in both figures. (Note: 1 kN = 224.9 lbf; 1 mm = 0.039 in.)

composite sections, along with the flexural response of the 3-D-printed plain L₃₀ and/or UB sections that constitute the composite section. Figure 9(a) shows the response of the CS beams (unreinforced) and the L₃₀ and UB mixtures, while Fig. 9(b) shows the response for the CS_F beams and the UB-SF_{1.5%} beams.

The load-deflection responses for the unreinforced beams shown in Fig. 9(a) indicate marginally high brittleness of the UB beam as compared to the L₃₀ beam of a lower strength. For the composite sections, the beams were able to resist nearly similar loads at failure for both configurations, with a marginally lower (~10%) value for Configuration B. The peak load of the CS beam under Configuration A is only ~20% lower than the peak load experienced by the homogeneous UB beam, and under Configuration B, the peak load

of the CS is almost the same as that of the homogeneous L₃₀ beam. This is expected, because the bottom fibers are dominant in the response until the peak—UB layers constitute the fibers below the centroidal axis for Configuration A, and L₃₀ layers for Configuration B (with a small portion of UB also below the neutral axis for Configuration B case as the neutral axis is at ~34 mm [1.34 in.] from bottom, as shown later in the paper). The load capacity is not directly indicative of the stress at failure as the elastic moduli of the materials in the section are different. Strain-hardening response is noted for the UB-SF_{1.5%} and CS_F beams. For the CS_F beams shown in Fig. 9(b), the peak load capacities are significantly different for the two configurations (with the capacity for Configuration A being significantly higher, owing to the presence of fiber-reinforced UHPC in the bottom layers). Even when the

top half of the beam is replaced with the L_{30} mixture, the CS_F beam demonstrates a peak load capacity that is only ~15% lower than that of the UB-SF_{1.5%} beam for Configuration A. However, the post-peak ductility of the CS_F beam, in this case, is compromised with respect to the UB-SF_{1.5%} beam, with the residual strength at 3.75 mm (0.15 in.) deflection at only ~40% of its peak load capacity, which is only half of the residual strength of the UB-SF_{1.5%} beam. For Configuration B, the peak load capacity of the CS_F beam is higher than that of the unreinforced UB and L_{30} beams, and nearly 30% of the UB-SF_{1.5%} beam. At a deflection of 3.75 mm (0.15 in.), the CS_F beam retains approximately 70% of its peak capacity, which is much lower than that of the UB-SF_{1.5%} beam. In the CS_F Configuration B case, once a crack initiates, it propagates through the L_{30} portion of the beam, and the post-cracking response to the beam is provided by the UB-SF_{1.5%} portion. This results in the beam acting more like a reduced depth UB-SF_{1.5%} printed beam which is the major reason for the reduced load capacity of the CS_F beam under Configuration B.

The load values cannot be directly correlated to the stresses at the extreme fibers of the composite beams, unlike for a homogeneous section. From the calculated elastic moduli of the three different mixtures that constitute the composite sections (24 GPa [3500 ksi] for L_{30} ; 40 GPa [5800 ksi] for UB; and 45.33 GPa [6600 ksi] for the UB-SF_{1.5%} based on axial compression tests, as well as inverse analysis of flexural test results of homogeneous printed beams as detailed elsewhere³⁷), the neutral axis depth and the extreme fiber stresses are calculated using the transformed section method,^{47,48} which converts a beam cross section consisting of two different materials into an equivalent homogeneous beam. The stresses at cracking and the peak load capacities thus determined for the plain and fiber-reinforced composite sections are shown in Fig. 10.

For the 3-D-printed unreinforced beams with the same material for all layers (L_{30} and UB beams), the cracking stress and peak stress are the same and the UB beam shows higher strength. For the unreinforced composite beams (CS), the strength under Configuration A is higher than that for Configuration B, because the bottom fibers are made of the UB mixture of higher strength. The composite section attains peak strength similar to that of the UB beam under Configuration A and the L_{30} beam under Configuration B. Thus, the peak load capacity for the composite beams is a function of the placement of different material types to create the composite section, rather than both materials achieving similar stress at failure. Similar is the response for the CS_F beam, which is a combination of the UB-SF_{1.5%} mixture and L_{30} mixture. Under Configuration A, this composite beam with half the total amount of fibers as that of the UB-SF_{1.5%} beam, and reduced cement content attributed to the L_{30} mixture, attains similar flexural strengths as that of the UB-SF_{1.5%} beam. Under Configuration B, the CS_F beam shows a higher load-carrying capacity because even after the cracking stress of the L_{30} mixture, the fiber-reinforced UHPC mixture can carry more load.

Figure 11(a) shows the Lagrangian strain at peak load for the homogeneous L_{30} beam. Figures 11(b) and (c) are

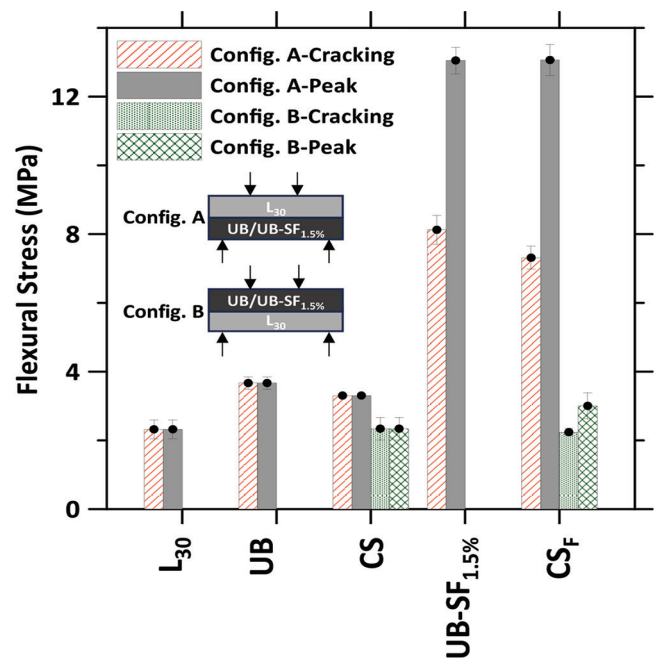


Fig. 10—Flexural stress at extreme bottom fiber for different composite section combinations, at cracking load and/or peak load capacity. (Note: 1 MPa = 145 psi.)

the Lagrangian strains at peak load for the unreinforced composite beam (CS) for both configurations respectively, while Fig. 11(d) to (g) denote the strain profiles for the CS_F beam at peak load and at 3.75 mm (0.15 in.) central deflection. The flexural capacity of the overall section is governed by the mixture that is under tensile strain. In Configuration A, UB or the UB-SF_{1.5%} layers (depending on whether the composite section is CS or CS_F) are under tensile strains, and in Configuration B, the L_{30} layers are under tensile strains. Hence, strain comparison for the CS beam is made with the UB single material beam under Configuration A, and with the L_{30} single material beam under Configuration B. The CS_F sections demonstrate ductile response for both configurations (refer to Fig. 9), and hence, the strains are compared to the UB-SF_{1.5%} single-material beam. The maximum strain at peak for the L_{30} beam was determined as 0.13% using DIC. The strain at peak load for the CS beams (Fig. 11(b) and (c)) and their single-material counterparts is similar. This further indicates failure initiation at similar stress conditions in the extreme fiber for the composite beam compared to those in the corresponding single-material beams, which has also been shown before in Fig. 10. DIC thus helps to validate that the change in load-carrying capacity of the composite section, as compared to the homogeneous section, is an artifact of the combined effect of different moduli systems working in synergy, rather than the actual printed mixture achieving higher strain (or stress) at failure. For the CS_F beams, for Configuration A, the strain observed at peak load (Fig. 11(d)) is nearly half that of the UB-SF_{1.5%} beam (Fig. 8(d)). This is similar to the observation from the load-deflection response where the load for the composite beam is seen to peak at approximately half of the deflection of the corresponding homogeneous beam (refer to Fig. 9(b)). At 3.75 mm (0.15 in.) deflection, the Lagrangian

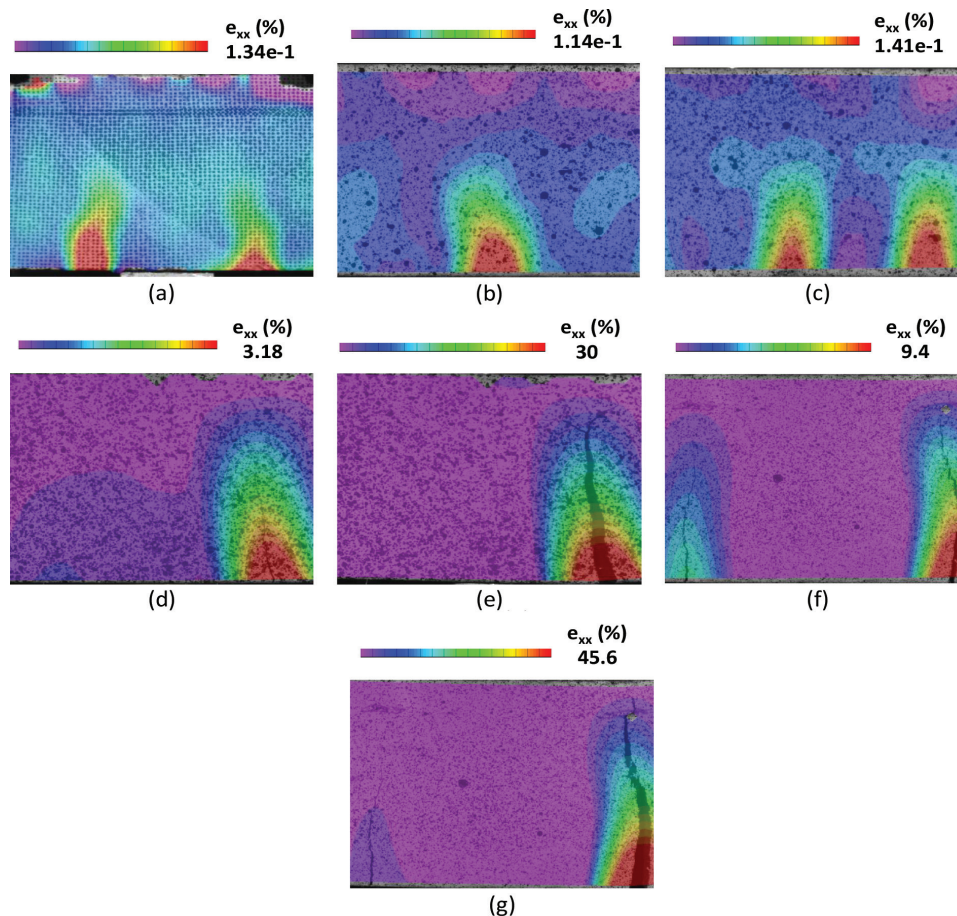


Fig. 11—Representative Lagrangian strain profile in middle-third span of beam for: (a) L_{30} at peak load; (b) CS at peak under Configuration A; (c) CS at peak under Configuration B; (d) CS_F at peak under Configuration A; (e) CS_F at 3.75 mm (0.15 in.) central deflection under positive Configuration A; (f) CS_F at peak under Configuration B; and (g) CS_F at 3.75 mm (0.15 in.) central deflection under Configuration B. Note that lowest strain value is set to -6.00×10^{-2} for all figures.

strains for the CS_F (Fig. 11(e)) and UB-SF_{1.5%} beams are comparable (Fig. 8(e)). The rapid decrease in load-carrying capacity for the CS_F beam as compared to the UB-SF_{1.5%} beam can be attributed to the presence of fibers only in the lower half of the CS_F beam under Configuration A. Here, the L_{30} mixture does not provide appreciable load-carrying capacity after cracking. The UB-SF_{1.5%} beam, with fibers throughout its depth, benefits from increased load transfer capacity as cracking initiates and the neutral axis shifts upward.

To determine the position of the neutral axis, compression-tension force equivalence was used. Determining the compressive and tensile forces requires the stress-strain response of the L_{30} and UB-SF_{1.5%} mixtures. An analytical quadlinear tensile constitutive model, as presented in Nair et al.,⁴⁹ was employed for this purpose, derived using inverse analysis of flexural test results of homogeneous printed beams, as detailed elsewhere.^{37,49} The stress-strain response was then used to calculate the neutral axis locations for the CS_F beams. Figure 12 shows the neutral axis location from the bottom of the beam, as a function of the strain in the bottom fiber. The neutral axis remains at a constant height until cracking begins, after which it starts shifting upward. When the neutral axis moves beyond the midpoint of the beam's height (30 mm [1.18 in.]), the L_{30} portion enters tension in the CS_F beam in Configuration A, while more of

the UB-SF_{1.5%} layers experience tension in the homogeneous beam. Due to the lower load-carrying capacity of the L_{30} portion in tension compared to the UB-SF_{1.5%}, the CS_F beam consequently exhibits a reduced load capacity relative to the UB-SF_{1.5%} beam.

The CS_F beam in Configuration B exhibits a larger strain at peak load (Fig. 11(e)) compared to the UB-SF_{1.5%} homogeneous beam. Once a crack initiates in the CS_F beam in this configuration, it quickly propagates through the lower half of the beam where the brittle L_{30} mixture is present. As the crack reaches the UB-SF_{1.5%} layers, additional load-carrying capacity is mobilized. This behavior is reflected in the shift of the neutral axis for the CS_F beam in Configuration B, as shown in Fig. 12, where the neutral axis rapidly shifts to ~ 45 mm (1.77 in.) from the bottom of the beam, as the lower half provides little load transfer efficiency. The CS_F beam under Configuration B shows a comparable central deflection to the UB-SF_{1.5%} homogeneous beam at peak load capacity (refer to Fig. 9(b)), while the strain at 3.75 mm (0.15 in.) deflection (Fig. 11(f)) is slightly higher.

Preliminary LCA-TEA of composite sections

Previous sections have shown that composite sections (both CS and CS_F) can achieve load capacities comparable to those of the dominant homogeneous mixtures (that is,

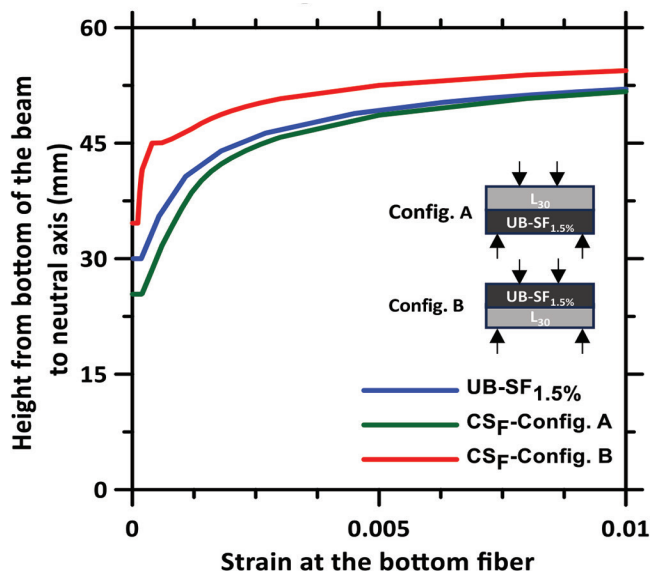


Fig. 12—Height from bottom of beam to neutral axis with respect to strain at bottommost fiber of beam obtained through force equivalence. (Note: 1 mm = 0.039 in.)

UB or UB-SF_{1.5%}). In addition to structural performance, composite 3-D-printed sections offer economic and environmental advantages. Conventional casting of such composites is often challenging due to the need for separate casting of different layers, resulting in issues such as cold joints and dimensional inaccuracies. Cold joints, formed when there is a delay between successive placements, can lead to critical defects under flexural loading. Also, keeping exact layer heights in formwork can be difficult. These issues are mitigated in 3-D-printing through continuous, automated layering, which eliminates cold joint formation and improves dimensional control and fabrication efficiency.

Cost, emissions, energy demand, and performance of homogeneous and composite sections—This section presents the cost and environmental impact (that is, CO₂ emission and primary energy demand) for various 3-D-printed mixture combinations and configurations used in composite sections. The material cost for 1 m³ (35.3 ft³) of different concrete mixtures (that is, not accounting for mixing or printing costs), considered for the composite section, is shown in Table 4. The prevalent material costs in the United States, provided by various manufacturers, are used here; regional variations are not considered. A cradle-to-gate LCA was performed using the Concrete LCA Web Tool,⁵⁰ and the total CO₂ emissions (kgCO₂-eq) and primary energy demand (PED) were obtained for all the mixtures and are shown in Table 4. The web tool does not account for CO₂ emissions and PED for steel fiber production; hence, an additional 1.89 kgCO₂-eq and 27.6 MJ per kg of steel fiber, based on the data provided by the fiber manufacturer,⁵¹ was considered for the fiber-reinforced mixtures.

Table 4 also presents the peak loads resisted by beams of 60 x 60 mm (2.36 x 2.36 in.) cross section and a span length of 240 mm (9.45 in.) made using the corresponding mixtures, calculated using the flexural strength, which was discussed in an earlier section. While the cost and CO₂ intensity of

the UB-SF_{1.5%} mixture are three and one and a half times, respectively, that of the conventional L₃₀ mixture, the flexural strength is enhanced six times, providing opportunities to optimize cost, CO₂ emissions, and performance.

Using the obtained data for homogeneous mixtures, cost, CO₂ emissions, and PED for the composite material sections were calculated, and are also shown in Table 4. Two combinations are explored here: (a) section with three layers of L₃₀ and three layers of either UB or UB-SF_{1.5%} mixture (referred to as CS and CS_F respectively; the experimental results for which were shown in “Composite beams” section; and (b) modified sections with four layers of L₃₀ and two layers of either UB or UB-SF_{1.5%} (referred to hereinafter as CS-M and CS_F-M, respectively; for which no experimental studies were carried out, but performance can be theoretically estimated). The modified sections with only 33% of UHPC (CS-M and CS_F-M) are obviously more cost- and carbon-efficient as compared to their counterparts (CS and CS_F, respectively) because they are composed of more layers of the conventional L₃₀ mixture. Based on the flexural strengths of the beams made with single materials, the flexural peak loads carried by the modified sections were calculated using the transformed section method.^{47,48} Both Configurations A and B were considered in the calculations and are shown in Table 4. The cost, CO₂ emissions, and PED for 1 m³ (35.3 ft³) of the composite sections (when the sections contain three layers of L₃₀ and UB-SF_{1.5%} mixtures—that is, 50% of each, or four layers of L₃₀ and two layers of UB-SF_{1.5%} mixtures—that is, 66% of the former and 34% of the latter) are significantly lower in comparison to those of UB-SF_{1.5%} and comparable to or lower than those of the unreinforced UB sections. When the composite sections contained four layers of L₃₀ mixture instead of three layers, there was only a marginal reduction in the load-carrying capacity, justifying the use of more layers of the sustainable mixture in the cross sections without major loss in structural performance.

Sustainability analysis for 6 m long beam—Based on the load-carrying capacity of the different homogeneous and composite beams shown in Table 4, a simply supported 6 m (19.7 ft) long beam with a uniformly distributed unfactored load of 3 kN/m (205.6 lbf/ft) is designed. Keeping the width constant at 200 mm (7.87 in.), the minimum depth required to resist this load was calculated, along with the volume of material required to construct the beam. Table 5 shows the volumes of different concrete mixtures needed based on the sections considered (both homogeneous as well as composite), and the associated cost and CO₂ emissions related to materials for this beam. Note that for the composite beams, only Configuration A is considered. By using a UHPC mixture with steel fibers (UB-SF_{1.5%}), the material cost required for a homogeneous section to resist the imposed load is increased by ~30% as compared to conventional concrete, but the lower section dimensions needed to carry the same load results in ~33% lower carbon impact. When fiber-reinforced composite sections are used, with 50% or 33% of UB-SF_{1.5%} (CS_F and CS_F-M, respectively), the material costs are much lower than the homogeneous UB-SF_{1.5%} and the carbon impacts are also lower by 10 to 20%. Appropriate structural design and techno-economic

Table 4—Cost, CO₂ emissions, and PED for 1 m³ (35.3 ft³) volume of different mixtures and composite sections considered

Combination	Cost, \$/m ³	CO ₂ emissions, kgCO ₂ -eq/m ³	Primary energy demand, MJ/m ³	Peak load, kN (lbf); for 60 x 60 mm beam with span of 240 mm	
				Configuration A	Configuration B
L ₃₀	178	728	3410	2.09 (469.9)	
UB	281	1012	5143	2.97 (667.7)	
UB-SF _{1.5%}	540	1148	7250	11.75 (2641.5)	
CS	230	870	4277	2.59 (582.26)	2.38 (535.05)
CS-M	212	823	3988	2.57 (577.76)	2.36 (530.55)
CS _F	359	938	5330	9.87 (2218.86)	2.94 (660.94)
CS _F -M	299	868	4690	9.83 (2209.87)	2.42 (544.04)

Note: Peak load capacity under four-point bending for beam of cross section 60 x 60 mm (2.36 x 2.36 in.) and span length of 240 mm (9.45 in.) for different combinations and configurations, calculated using transformed section method. Note that, for composite sections, UB or UB-SF_{1.5%} mixture is in tension when subjected to Configuration A, and in compression when subjected to Configuration B.

Table 5—Material volume required and associated cost and CO₂ emissions for different homogeneous and composite beams of 6 m (19.7 ft) length

Material or composite section details	Volume, × 10 ⁻² m ³	Material cost*, \$	CO ₂ emissions, kgCO ₂ -eq	% difference in cost†	% difference in CO ₂ emissions†
L ₃₀	50.11	89.14	365.06	0.00	0.00
UB	42.04	118.28	425.42	32.68	16.53
UB-SF _{1.5%}	21.14	114.10	242.73	27.99	-33.51
CS	45.02	103.37	391.75	15.96	7.31
CS-M	45.19	95.97	371.92	7.66	1.88
CS _F	23.06	82.76	216.42	-7.16	-40.72
CS _F -M	23.11	68.98	200.68	-22.61	-45.03

*Material cost accounts only for cost of ingredients.

†In comparison to conventional mixture (L₃₀).

Note: Composite beams are designed only for Configuration A.

analysis will be helpful in developing desirable cross sections using combinations of multiple materials because 3-D printing offers the flexibility to do this efficiently and cost-effectively.

CONCLUSIONS

Ultra-high-performance plain and fiber-reinforced concrete mixtures suitable for three-dimensional (3-D) printing applications were proportioned with 30% of cement (by mass) replaced by supplementary cementitious materials, enabling the attainment of 28-day compressive strengths of 100 to 120 MPa (14.5 to 17.4 ksi). To exploit the advantages of layer-wise 3-D printing to enable cost-efficiency and sustainability of concrete without compromising performance, 3-D-printed ultra-high-performance concrete (UHPC) layers (unreinforced, termed UB, or steel fiber-reinforced at 1.5% by volume, termed UB-SF_{1.5%}) were printed along with a conventional unreinforced 3-D-printable mixture where limestone powder replaced 30% of cement by mass. Composite sections were printed, which comprised three layers of conventional concrete (termed L₃₀) and three layers of UB or UB-SF_{1.5%}, from which beams were cut to evaluate flexural performance. The fibers were found to be mostly aligned in the print direction, and therefore perpendicular to the direction of crack propagation in

flexure, further enhancing the performance. The following conclusions were derived from this study:

- The differences in flexural strengths and toughness between conventionally cast and 3-D-printed beams were negligible for the fiber-reinforced UHPC specimens, while the influence of defects was more dominant in reducing the strength of the unreinforced 3-D-printed specimens. The preferential orientation of steel fibers in 3-D-printed specimens offered more ductility than is commonly observed in the case of cast specimens where the fibers are randomly oriented.
- The use of UB-SF_{1.5%} in the tension zone along with a conventional mixture (L₃₀) in the compressive zone provided peak load capacities and toughness that were only slightly lower than the case of a homogeneous UB-SF_{1.5%} beam. The strain at peak stress, obtained from digital image correlation (DIC), for the composite and homogeneous sections is comparable, while at a deflection of 3.75 mm (0.15 in.), the residual stress is lower for the composite beam showing lower toughness, primarily attributed to the reduction in overall fiber volume in the composite beam. The neutral axis of the composite beam as a function of the strain in the extreme bottom fiber also demonstrated similarity with the homogeneous

UB-SF_{1.5%} beam, indicating that structural performance similarity can be attained in a more sustainable manner.

- Life cycle and techno-economic analyses (LCA/TEA) found the UB-SF_{1.5%} mixture to be significantly more expensive and carbon-intensive as compared to the conventional mixture, even though flexural strength was enhanced by more than five to six times. When composite beam sections consisting of either 50 or 33% by volume of UB or UB-SF_{1.5%} in the tension zone were tested, the peak load capacities remained comparable, but the cost and carbon impact were reduced to the tune of 30 to 40%.
- The results of this study showed that the use of targeted layering of different materials, achievable through 3-D printing, to obtain composite sections allows better exploitation of beneficial material properties to reduce the overall cost and environmental impacts associated with structural concrete. Optimization of material types and properties and fiber volume fraction, number and thickness of layers, and their positioning enable the development and construction of 3-D-printed performance-equivalent structural members with lower cost and environmental impacts.

This study focuses on the peak load capacity in flexure for LCA/TEA, as the transformed section method allows for estimating the peak load capacity of multi-material composite sections. However, accurately predicting the complete load-deflection response is essential for a more comprehensive analysis. While closed-form solutions for the flexural response of homogeneous beams are well-established, a similar approach for composite material sections is still lacking and remains a topic for future research. To address this, the authors are currently developing a MATLAB-based algorithm aimed at predicting the full load-deflection response for any combination of materials. This will facilitate comparisons not only in terms of peak load capacity but also in terms of post-peak behavior and toughness.

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