

Enhanced performance of fly ash-cement mortar through tetraethyl orthosilicate modification

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ABSTRACT

This study investigates the enhancement of fly ash-cement mortar performance through tetraethyl orthosilicate (TEOS) modification, focusing on both strength development and durability parameters. A comprehensive experimental program, including compressive strength tests, water absorption measurements, sorptivity assessments, and ultrasonic pulse velocity (UPV) evaluations, was conducted. The results demonstrated that partial replacement of cement with 30% fly ash not only improved 28-day compressive strength to 46.81 MPa, but also significantly refined the microstructure, as evidenced by reduced water absorption and higher UPV values. The incorporation of TEOS at 0.5%, 1%, and 1.5% further enhanced permeability without compromising strength, with the 1% TEOS mixture (S10) achieving optimal performance. This mixture exhibited the lowest water absorption (2.77%) and reduced sorptivity, attributed to the sol-gel formation of silica networks that densify the pore structure and impede water ingress. UPV results confirmed the structural integrity of TEOS-modified mortars, with velocities comparable to the highest-performing fly ash blend. FESEM analysis shows that incorporating fly ash and TEOS refines the cement mortar microstructure. This results in enhanced matrix densification and improved morphology. These findings underscore the efficacy of TEOS as a chemical modifier for producing durable, high-performance fly ash-cement composites, suitable for sustainable construction applications.

Keywords: fly ash, cement mortar, tetraethyl orthosilicate, sorptivity, ultrasonic pulse velocity, rate of absorption.

INTRODUCTION

Concrete remains the most widely used construction material due to its versatility, durability, and cost-effectiveness. However, concrete often faces durability challenges from water and aggressive agents, leading to significant global maintenance costs [1]. While numerous strategies have been explored to enhance concrete performance, there remains a critical knowledge gap in the internal modification of high-volume supplementary cementitious material (SCM)-based systems using sol-gel precursors such as tetraethyl orthosilicate (TEOS). Most studies to date have focused on surface applications of TEOS for heritage conservation, leaving the potential of in-mixture TEOS incorporation in modern, sustainable cementitious systems largely unexplored. This study directly bridges this

gap by investigating the synergistic effects of internal TEOS addition in high-fly-ash cement mortars – a novel approach that combines sustainability with nano-scale matrix densification, offering a new pathway for durable, low-carbon construction materials.

To address the durability issues, researchers are exploring advanced solutions, including tetraethyl orthosilicate, a silicon alkoxide ($\text{Si}(\text{OC}_2\text{H}_5)_4$), which has drawn attention for its ability to form silica networks via hydrolysis and condensation, thereby densifying cementitious microstructures [2]. Meanwhile, the use of supplementary cementitious materials (SCMs), such as fly ash has become common to reduce cement usage and carbon emissions. Fly ash, a coal combustion by-product, enhances concrete properties but its high-volume use often delays early strength development [3, 4]. Although both

TEOS and fly ash have been individually studied, their combined internal use in mortar systems has not been systematically evaluated, particularly in terms of microstructural refinement and transport property enhancement. This study uniquely bridges this divide by exploring the synergy between TEOS and high-volume fly ash in ordinary Portland cement (OPC) based mortars, aiming to overcome the limitations of delayed strength while significantly improving durability.

TEOS, a silicon alkoxide, is used in sol-gel processes to form silica-based materials. When applied to cementitious systems, it hydrolyzes into silanol groups that condense into a silica network [5]. TEOS has been shown to improve the mechanical strength and water resistance in historical mortars [6] and natural stones [7]. In cement pastes, TEOS densifies the matrix by forming in-situ nano-silica, boosting strength and lowering permeability [8]. Additionally, TEOS-treated composites have shown better sulfate resistance due to pore refinement and extra C-S-H formation [9]. Fernandez et al. [5] found that tetraethyl orthosilicate TEOS is effective in strengthening and protecting cement mortars, making it suitable for conserving cultural heritage structures. TEOS enhances durability by reducing porosity and permeability, while maintaining the original appearance. Its chemical interaction with cement components forms additional binding gels that improve the overall performance of the material. Fernandez et al. [10] demonstrated that tetraethyl orthosilicate TEOS, especially when combined with nanolime, is effective in strengthening and protecting cement-based materials without altering their appearance. The treatment enhances durability by increasing strength, reducing porosity, and improving water resistance. These findings suggest TEOS-nanolime blends are promising for preserving historic cement structures.

Fly ash is widely used to replace OPC, contributing to workability and long-term strength via pozzolanic reactions [11]. However, high fly ash content slows early strength development due to its slower reactivity [12]. To counter this issue, methods such as chemical activation and mechanical processing have been explored [13]. The role of the physical and chemical properties of fly ash in improving its reactivity for better performance in high-volume applications [14].

Yerramala et al. [15] investigated the effect of replacing OPC with Class F fly ash (5–25%) on the strength of cement mortars. Results showed

that while early-age strength decreased for all fly ash replacements, the mortars with up to 15% fly ash developed higher compressive and split tensile strength than the control after 28 days. The optimal replacement level was found to be 10% for maximum strength. Additionally, fly ash mortars exhibited a higher efficiency factor and better tensile strength-to-compressive strength ratio compared to conventional concrete, although existing empirical relationships did not accurately predict their behavior.

Bendapudi et al. [16] highlighted the growing importance of using fly ash as a supplementary cementing material in mortar and concrete, due to its environmental benefits and performance enhancements. Fly ash, a by-product of coal combustion, improves workability, reduces water demand, enhances durability, and increases long-term strength. The study confirmed that fly ash is a reactive pozzolan that improves sulfate resistance as well as contributes positively to setting time and strength development. Overall, replacing a portion of portland cement with fly ash enhances the physical and mechanical properties of concrete, making it a sustainable choice for modern construction. While TEOS and other ethyl-silicates have been widely studied as surface consolidants/protective treatments for historic mortars and hardened concrete, the internal (in-mixture) use of TEOS to modify fresh fly-ash–cement mortars has received far less attention. Therefore, this study makes three distinct and original contributions to the field: (1) it is among the first to evaluate the internal incorporation of low TEOS dosages (0.5–1.5% w/w of binder) in a high-fly-ash (30% FA) mortar system, targeting both performance and sustainability; (2) it integrates comprehensive mechanical, durability, and microstructural analyses – including compressive strength, water absorption, sorptivity, ultrasonic pulse velocity (UPV), and field emission scanning electron microscopy (FESEM) – to establish a direct link between sol-gel-derived silica formation and bulk transport properties; and (3) it identifies an optimal internal TEOS dosage (1% w/w) that significantly reduces water uptake and sorptivity without compromising the 28-day compressive strength.

Collectively, these findings represent a significant scientific advance by extending the application of TEOS from surface conservation to internal modification of sustainable, SCM-rich cementitious systems – a shift that opens new possibilities for durable, low-carbon concrete design.

MATERIAL AND METHODS

The materials used in this investigation comprise several key components, as illustrated in Figure 1. Ordinary Portland cement conforming to ASTM C150 specifications was used as the primary binding agent, as depicted in Figure 1a. Class F fly ash sourced from Eurobuild Flyash and conforming to ASTM C618 requirements was incorporated as presented in Figure 1b. Tetraethyl orthosilicate obtained from Sigma-Aldrich, with properties detailed in Table 3, served as an additional silicate source and is shown in Figure 1d. The fine aggregate consisted of Ottawa sand with a fineness modulus of 2.775, as demonstrated in Figure 1c, which complies with the ASTM C778 standards as illustrated in Table 1. Potable water with a neutral pH of 7 was utilized for all mixing procedures. To optimize workability characteristics, Sika ViscoCrete-180

GS high-range water-reducing superplasticizer was incorporated into the mixtures, as illustrated in Figure 1d. This admixture meets the performance requirements established by ASTM C494. The detailed chemical compositions of both ordinary Portland cement and fly ash are presented in Table 2, while the superplasticizer specifications are provided in Table 4.

TEST PROCEDURE

The experimental program involved preparing three mortar cubes ($50 \times 50 \times 50$ mm) for each test: compressive strength, water absorption, sorptivity, and ultrasonic pulse velocity (UPV). The dry materials were first weighed and mixed thoroughly to achieve a uniform

Table 1. Sieving test results

Sieve size (mm)	Percentage passing (%)
4.75	100
2.36	95
1.18	75
0.6	37.5
0.3	12.5
0.15	2.5
0.075	0

Table 2. Chemical content of the OPC and the FA

Component	OPC	FA
Silicon dioxide (SiO_2)	19.7	48.2
Aluminum oxide (Al_2O_3)	6.3	25.4
Iron oxide (Fe_2O_3)	4.3	10.7
Calcium oxide (CaO)	62.2	8.3
Magnesium oxide (MgO)	1.8	2
Sulfur trioxide (SO_3)	2	1.9
Loss on ignition (LOI)	1.1	1.2
Alkalis ($\text{Na}_2\text{O} + \text{K}_2\text{O}$)	2	1.6
Other oxides	0.6	0.7

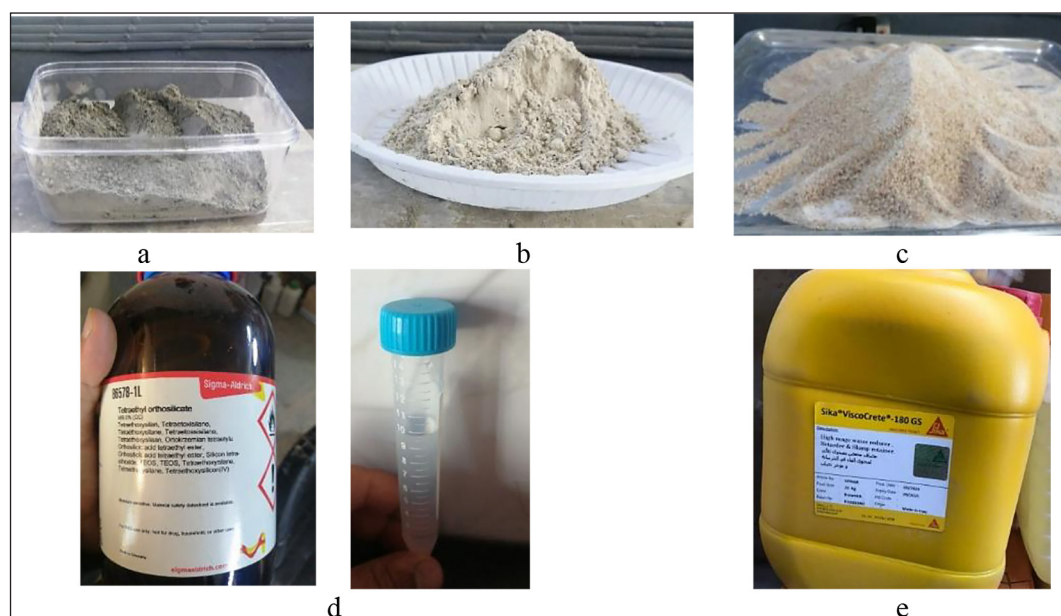


Figure 1. (a) OPC; (b) FA; (c) Ottawa Sand; (d) TEOS; (e) viscocrete

Table 3. Silane compound properties

Property	Description
Appearance	Colorless liquid
Density	0.933 g/mL at 20 °C (lit.)
Refractive Inde N20/D	1.382–1.384
Purity (GC AREA %)	≥ 99.0 %

consistency. Water was then gradually added, while continuously mixing to form a homogeneous mortar. The fresh mortar was carefully cast into molds, vibrated for one minute to remove air bubbles, and leveled. After one day, the specimens were demolded and immersed in water to cure. The average values of the three cubes were reported for each measurement. The compressive strength was determined in accordance with ASTM C109/C109M [21]. Mortar cube specimens ($50 \times 50 \times 50$ mm) were demolded after 24 hours, cured in water at 23 ± 2 °C, and tested at specified ages. Compressive strength was measured at 7 and 28 days, while all other tests were conducted at 28 days. The test was carried out using a compression testing machine with a maximum loading capacity of 300 kN. The load was applied continuously and without shock at a controlled loading rate of 0.9 ± 0.2 kN/s (equivalent to 0.25 ± 0.05 MPa/s) until

specimen failure, as specified in ASTM C109/C109M [21]. Water absorption was measured following ASTM C642 [22], by first oven-drying the samples at 105 ± 5 °C to a constant mass, then immersing them in water (as indicated in Figure 2a) for 24 hours and weighing again. Sorptivity was tested based on ASTM C1585 [23] using the same cube specimens, with only one 50×50 mm face exposed to water while the other faces were sealed, as illustrated in Figure 2b. Both water absorption and sorptivity tests used $50 \times 50 \times 50$ mm cube specimens. Water absorption measures the total water uptake when the specimen is fully immersed, while sorptivity evaluates the rate of water ingress through capillary suction when only one surface is exposed. Finally, the UPV test was carried out according to ASTM C597 [24] using the direct transmission method, where the velocity was calculated by dividing the path length by the pulse transit time. All specimens were prepared and cured under the same conditions to ensure uniformity and reliable comparison across different mixtures. For the FESEM test, mortar samples were dried and then crushed into small fragments. The selected fragments were mounted on aluminum stubs using carbon tape and coated with a thin layer of gold using a sputter coater to ensure conductivity. Field FESEM analysis was performed under


Figure 2. (a) Water absorption; (b) sorptivity

Table 4. Super plasticizing admixture

Property	Description
Composition	Aqueous solution of modified polycarboxylates
Appearance	Light brownish liquid
pH-Value	4–6
Specific gravity	$1.070 \pm (0.005)$ g/cm ³

high vacuum mode at an accelerating voltage of 5–15 kV to observe the surface morphology at high resolution. Table 5 shows the mixture design which is done with a ratio of 1:2.75:0.3.

RESULTS AND DISCUSSION

The 7-day and 28-day compressive strength (CS) tests evaluated early and long-term mechanical performance, critical for assessing hydration kinetics and pozzolanic activity. The reference mixture (Ref) achieved 31.57 MPa (7-day) and 44.34 MPa (28-day), typical of ordinary portland

cement systems due to rapid early hydration [25]. Replacing 25% of cement with fly ash (F25) reduced 28-day strength to 42.78 MPa, reflecting delayed pozzolanic reactions, since fly ash requires time to react with calcium hydroxide [26, 27, 28]. However, at 30% fly ash replacement (F30), 28-day strength increased to 46.81 MPa, exceeding the reference mixture, indicating Fly ash content enhances densification via secondary hydration [29]. TEOS-modified mixtures (S05–S15) maintained comparable 28-day strength (~46.6–46.75 MPa), indicating that TEOS does not hinder hydration but refines pores, maintaining strength [30]. Table 6 and Figure 3 show the test results.

Table 5. Mixture design

Mixture	Cement	Sand	Water	FA	TEOS	HRWR
Ref	1000	2750	300	0	0	12
F25	750	2750	300	250	0	12
F30	700	2750	300	300	0	12
S05	700	2750	300	300	5	12
S10	700	2750	300	300	10	12
S15	700	2750	300	300	15	12

Table 6. Compressive strength and UPV test results

MixtureS	7-day CS(MPa)	28-day CS (MPa)	UPV (m/s)
Ref	31.57	44.34	4380
F25	31.43	42.78	4192
F30	32.96	46.81	4583
S05	32.84	46.75	4564
S10	32.81	46.63	4570
S15	32.76	46.61	4549

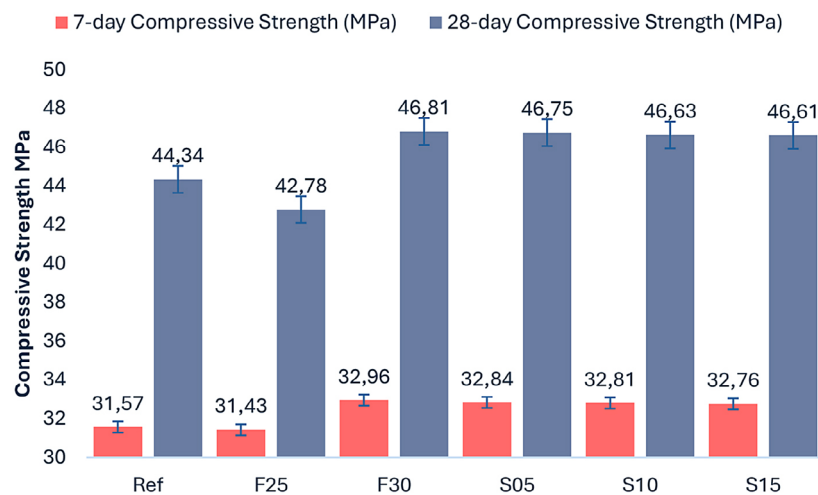


Figure 3. Compressive strength results

Water absorption (in Figure 4) reflects porosity and durability, with 4.56% absorption of the reference mixture indicating a coarser pore structure. Fly ash incorporation reduced absorption (F25: 4.02%; F30: 3.83%) due to the micro-filler effect of fly ash, narrowing capillary pores [26, 31]. TEOS further decreased absorption, with S10 achieving the lowest (2.77%), as hydrolyzed TEOS forms silica gel, sealing surface pores [32]. However, a slight absorption rebound (3.1%) of S15 suggests excessive TEOS may agglomerate, creating localized weak zones (Table 7) [30].

The partial substitution of ordinary portland cement with fly ash at 25% and 30% proportions (designated as mixtures F25 and F30) resulted in a notable reduction in sorptivity values relative to the control mixture. This phenomenon can be attributed to the pozzolanic reaction of fly ash with calcium hydroxide [26], producing additional calcium silicate hydrate (C-S-H) gel that densifies the microstructure [33, 34]. Furthermore, the fine spherical particles of fly ash enhance particle packing density within the cementitious matrix,

effectively restricting the interconnectivity of capillary networks [1, 35].

The incorporation of tetraethyl orthosilicate (TEOS) at concentrations of 0.5%, 1%, and 1.5% (denoted as S05, S10, and S15) demonstrates a significant capacity to mitigate water absorption [36, 37]. Most notably, the S10 formulation exhibits substantially reduced sorptivity compared to the F30 mixture. This enhanced performance can be primarily attributed to the sol-gel reaction mechanism of TEOS, wherein hydrolysis and subsequent condensation reactions generate amorphous silica networks within the pore structure [5, 38]. These siloxane bonds (Si-O-Si) not only physically obstruct capillary pathways, but also impart hydrophobic properties to the concrete surface through the formation of alkyl-substituted silica compounds [39, 40]. The resulting modification of surface energy characteristics significantly impedes the capillary suction forces that typically facilitate water ingress into the cementitious matrix [41, 42]. Figure 5 shows the sorptivity test.

Table 7. Water absorption results

MixtureS	Dry mass (g)	Saturated mass (g)	Water absorbed (g)	Water absorption (%)
Ref	285	298	13	4.56
F25	286	297.5	11.5	4.02
F30	287	298	11	3.83
S05	288	297.5	9.5	3.3
S10	289	297	8	2.77
S15	290	299	9	3.1

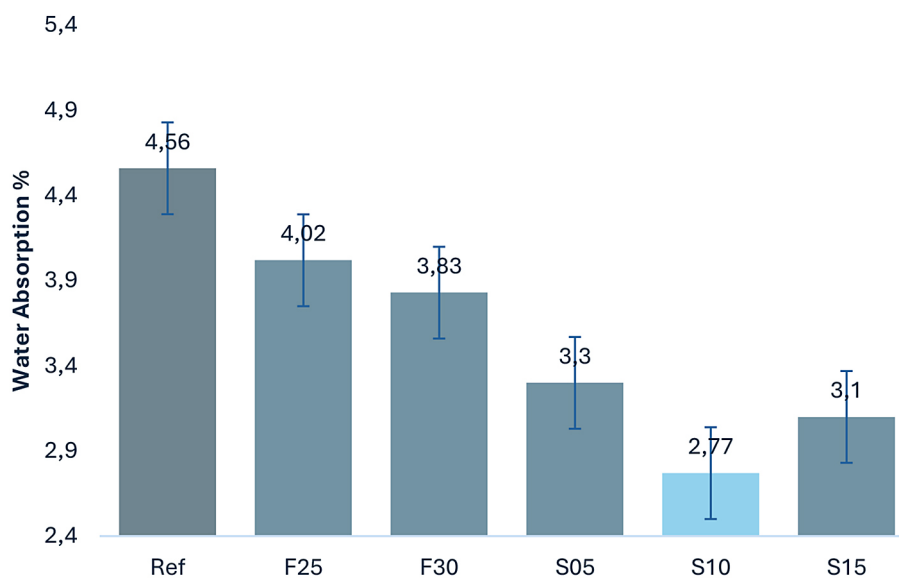


Figure 4. Water absorption test results

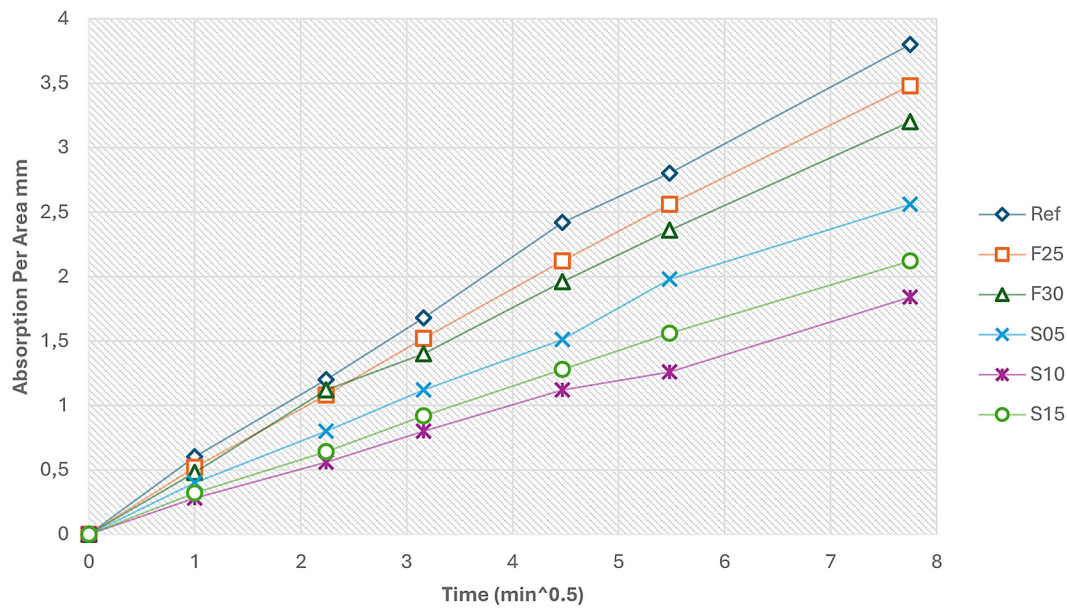


Figure 5. Sorptivity test results

UPV measurement assesses concrete homogeneity and internal flaws, correlating with density and elastic modulus. The reference mix exhibited 4380 m/s, indicative of a dense microstructure with minimal additives. The lower UPV (4192 m/s) of F25 aligned with its reduced 28-day strength, likely due to incomplete Fly ash reaction creating subtle voids [1]. The higher UPV (4583 m/s) of F30 confirms enhanced matrix integrity from the pozzolanic refinement of FA. TEOS blends (S05–S15) showed stable UPV (~4549–4570 m/s), suggesting colloidal silica from TEOS hydrolysis-maintained density without disrupting

the FA-cement matrix [5]. Table 6 and Figure 6 show the test results.

The FESEM results (shown in Figure 7) illustrated the microstructural evolution of cement mortar due to fly ash and TEOS incorporation. The reference sample (Ref) reveals a typical hydrated cement matrix with needle-like ettringite, fibrous calcium silicate hydrate (C–S–H), and visible portlandite crystals. Upon replacing 30% of cement with fly ash (F30), the microstructure became denser and more compact, with fewer crystalline features, indicating that the pozzolanic reaction and filler effect of fly ash, which refines

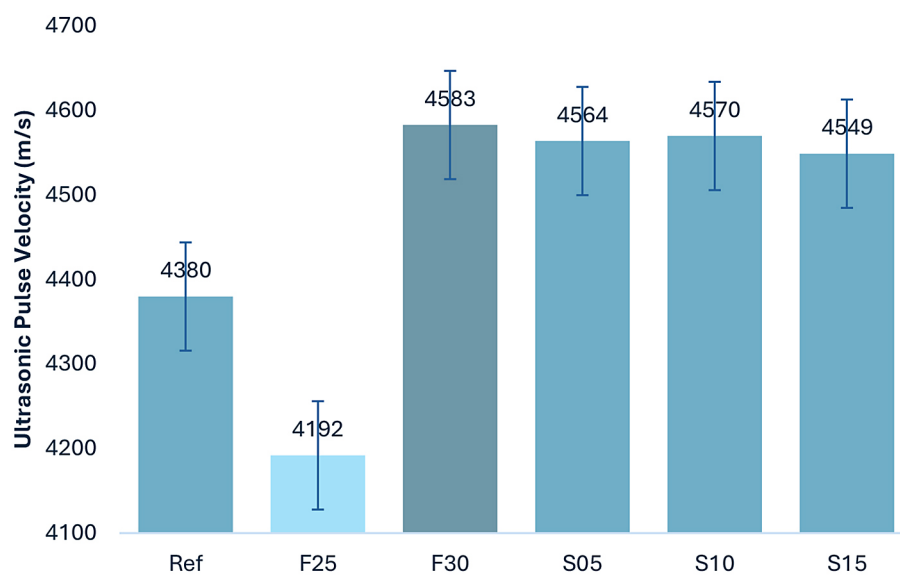


Figure 6. UPV test results

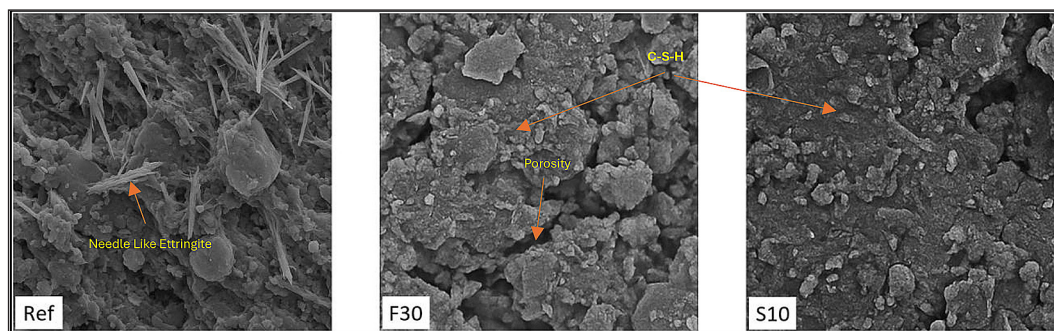


Figure 7. The FESEM test results

the pore structure [43, 44]. The addition of 1% tetraethyl orthosilicate (S10) further enhances the matrix densification, showing fewer voids and a more homogeneous microstructure, suggesting that TEOS may promote additional silica gel formation and improve the packing of hydration products [45, 46].

CONCLUSIONS

This study investigated the effects of incorporating tetraethyl orthosilicate (TEOS) into fly ash–cement mortar to enhance mechanical performance and durability. Results showed that adding 1% TEOS by weight of binder to a mortar mixture containing 30% Class F fly ash led to a slight increase in compressive strength (from 44 MPa to 46 MPa), but a notable reduction in water absorption and lower sorptivity compared to the control mixture without TEOS. FESEM micrographs confirmed a denser microstructure with fewer interconnected pores, indicating that in situ sol–gel silica formation effectively refined the pore network. Unlike most previous research, which has focused on TEOS as a topical surface treatment, this work demonstrated the effectiveness of internal (in-mixture) TEOS addition in fly ash–cement mortar. The combination of mechanical, transport, and microstructural analyses revealed that TEOS at an optimal dosage of 1% reduces capillary connectivity and enhances durability, even though strength gains are limited. This finding provides a practical and scalable approach to improving the performance of sustainable, SCM-rich mortars, expanding the potential applications of TEOS beyond conventional surface treatments. Overall, the synergistic effect of fly ash and TEOS presents a practical pathway for developing advanced cementitious materials with

improved strength and durability, aligning with the goals of sustainable construction and infrastructure resilience.

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