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Effect of TEOS on the Properties of Cementitious Mortar

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ABSTRACT

In this research, the influence of tetraethyl orthosilicate (TEOS) on the mechanical and durability properties of fly ash blended cementitious mortar is investigated. Six mortar mixes were formulated: reference mix (without TEOS and fly ash), two 25% and 30% fly ash mixes (F25 and F30), and three mixes with constant percentage of fly ash (30%) and varying amounts of TEOS (0.5%, 1%, and 1.5%) labelled as S05, S10, and S15, respectively. The key properties that are tested are indirect tensile strength, impact resistance, rapid chloride permeability (RCPT), and drying shrinkage. Results indicate that the addition of fly ash alone lowered early-age strength to some degree, whereas the addition of TEOS enhanced tensile and impact properties significantly, predominantly at the 1% dosage. TEOS enhanced durability too, as indicated by reduced chloride permeability and reduced shrinkage strains. The optimal performance was observed in the S10 mix, and it is proven that the moderate addition of TEOS enhances microstructure and durability of fly ash-based mortars. These findings suggest that TEOS is a promising contender for an additive to create high-performance, long-lasting, and sustainable cementitious composites.

Keywords: TEOS, Tetraethyl Orthosilicate, Cementitious mortar, Fly ash, Durability

تأثير رباعي إيثيل أورثوسيليكات على خصائص الملاط الأسمنتي

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الخلاصة

في هذا البحث، تم دراسة تأثير رباعي إيثيل أورثوسيليكات (TEOS) على الخواص الميكانيكية والمتانة لملاط الأسمنت المخلوط بالرماد المتطاير. صُممت ست خلطات من الملاط: الخلطة المرجعية (بدون TEOS والرماد المتطاير) ، وخلطتان من الرماد المتطاير بنسبة 25% و30 % (F25وF30)، وثلاث خلطات بنسبة ثابتة من الرماد المتطاير (30%) وكميات متفاوتة من TEOS (0.5% و1% و1.5%)، مُسمّاة بالرموز S05 و S10و S15على التوالي. الخصائص الرئيسية التي تم اختبارها هي قوة الشد غير المباشرة، ومقاومة الصدمات، ونفاذية الكلوريد السريعة(RCPT) ، وانكماش التجفيف. تشير النتائج إلى أن إضافة الرماد المتطاير وحده قللت من قوة التحمل في مرحلة مبكرة إلى حد ما، بينما عززت إضافة TEOS خصائص الشد والصدمة بشكل كبير، وخاصة عند جرعة 1%. كما عزز TEOS المتانة، كما يتضح من انخفاض نفاذية الكلوريد وانخفاض انفعالات الانكماش. لوحظ الأداء الأمثل في خليطS10 ، وثبت أن الإضافة المعتدلة لمادة TEOS تُحسّن البنية الدقيقة ومتانة ملاط الرماد المتطاير. تشير هذه النتائج إلى أن مادة TEOS تُعدّ خيارًا واعدًا لإنتاج مركبات أسمنتية عالية الأداء وطويلة الأمد ومستدامة.

الكلمات المفتاحية: TEOS، رباعي إيثيل أورثوسيليكات، ملاط أسمنتي، رماد متطاير، متانة

INTRODUCTION

The building construction industry constantly looks for new materials and methods to improve the performance and durability of structures made from concrete. Tetraethyl Orthosilicate (TEOS), a silicon alkoxide, has been identified as a future pore-blocking agent that can greatly enhance the durability properties of cementitious materials [1]. TEOS is hydrolysed and undergoes condensation reactions in the alkaline condition of concrete, resulting in silica gel, which occupies the pore structure and lowers permeability [2].

Using supplementary cementitious materials (SCMs) such as fly ash has become a norm in modern concrete technology because of environmental and economic factors [3]. But in utilizing fly ash, there are conditions associated with permeability in the initial hydration stage where TEOS starts to offer protection because of opportunities available in this stage [4].

The main goal of this research work is to explore the effect of TEOS on the mechanical properties of fly ash-modified cementitious mortar. Some of the specific targets of this research work include: (1) to examine the effect of TEOS on the tension and impact strength, (2) to examine the effect of TEOS on chloride penetration resistance, (3) to examine the effect of TEOS on shrinkage properties, and (4) to explore the optimal quantity of TEOS.

The research thus makes a contribution to existing knowledge regarding TEOS properties in a cementitious system and gives practical advice concerning its use for enhanced durability performance in construction works.

CHEMISTRY AND MECHANISM OF TEOS

TEOS: $Si(OC_2H_5)_4$ — This compound is categorized as an organosilicon compound. This particular compound finds use as a source material for producing silica using a sol-gel process [5]. In the basic environment of concrete with pH values > 12:

$$Si(OC_2H_5)_4 + 4H_2O \rightarrow Si(OH)_4 + 4C_2H_5OH$$

The resulting silicic acid then undergoes condensation reactions to form a silica gel network that precipitates in a cementitious material pore [6]. This leads to a reduction in porosity and permeability properties with subsequent enhancement of durability properties.

PAST RESEARCH ON TEOS IN CONCRETE

A few studies have explored the use of TEOS in concrete. Franzoni et al. [7] found that TEOS had a prominent effect in reducing the penetration of water and chlorides in concrete. They inferred that TEOS worked even better when used in porous concretes with additional cementitious materials.

Pan et al. [8], investigating TEOS consolidation properties for surface-deteriorated concrete, achieved a marked enhancement in surface durability and hardness. The results were such that penetration resistance of TEOS depends on initial porosity values of concrete and methods of application.

In experiments conducted by Wheeler [9], TEOS-modified concrete was shown to reduce carbonation and have enhanced freeze-thaw resistance. This study emphasized that incorrect dosage may lead to undesirable adverse effects.

FLY ASH-TEOS INTERACTIONS

Fly ash and TEOS offer a combination that gives exceptional opportunities for performance enhancement in concrete. While fly ash causes later durability-related developments because of pozzolanic reactions, TEOS brings about short-term pore-blocking action [10].

A study conducted by Li et al. [11] indicated that TEOS had a pronounced effect in a fly ash concrete environment because of a relatively higher degree of initial porosity resulting from lower reactivity of the fly ash. They observed a synergistic effect of TEOS pore-blocking action along with fly ash pozzolanic activity.

RESEARCH GAPS

While previous studies have validated the beneficial effects of TEOS on concrete durability, few systematic investigations have examined optimum levels of dosage for fly ash-modified systems. In addition, cumulative effects on mechanical properties such as tensile and impact strength remain poorly understood. This research addresses these lags by providing comprehensive evaluation of TEOS performance over a range of dosage levels and determination of properties.

MATERIALS AND METHODS

Materials

Cement

Normal Portland Cement (OPC) according to ASTM C150 [12] Type I was used throughout the entire research work. The physical and chemical properties of the cement are presented in Table 1.

Fine Aggregate

River sand with a fineness modulus of 2.65 was used as fine aggregate. The sand was sieve-analyzed according to ASTM C136 [13] and had the gradation requirement according to ASTM C33 [14].

Fly Ash

Class F fly ash according to ASTM C618 [15] was obtained from a local coal power station. Fly ash properties are presented in Table 1.

Table 1: Material Properties

Component	OPC	FA
Silicon Dioxide (SiO_2)	19.7	48.2
Aluminium 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
Sulphur Trioxide (SO_3)	2	1.9
Loss on Ignition (LOI)	1.1	1.2

Alkalis ($Na_2O + K_2O$)	2	1.6
Other Oxides	0.6	0.7

TEOS

Technical grade Tetraethyl Orthosilicate of at least 98% purity (table 2) was obtained from a chemical supplier. The TEOS was stored in sealed bottles to prevent premature hydrolysis.

Table 2: Silane compound properties

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

High-Range Water Reducer

A polycarboxylate-type HRWR fulfilling ASTM C494 [16] Type F (table 3) was used to provide workability in all the mix designs.

Table 3: Super plasticizing admixture

Property	Description
Compositio n	Aqueous solution of modified polycarboxylates
Appearance	Light brownish liquid
pH-Value	4 - 6
Specific gravity	1.070 ± (0.005) g/cm^3

Mix Proportions

Six mortar mixes were prepared using the proportion specified in Table 4. Ref mix consisted of cement and sand only. Mix F25 and F30 contained fly ash replacement of 25% and 30% by weight of cement, respectively. Mixes S05, S10, and S15 contained TEOS between 0.5%, 1%, and 1.5% by weight of fly ash, respectively, and a constant percentage of HRWR at 1.2% (12 gm).

Table 4: Mix Proportions

ID	Cemen t	San d	Wate r	Fly Ash	TEOS
Ref	1000	2750	300	0	0
F25	750	2750	300	250	0
F30	700	2750	300	300	0
S05	700	2750	300	300	5
S10	700	2750	300	300	10
S15	700	2750	300	300	15

Specimen Preparation

Mixing of the mortar was carried out using a mechanical mixer in accordance with ASTM C305 [17]. The dry materials were first mixed for 30 seconds before being added incrementally with water that had dissolved HRWR. Some of the mixing water was pre-dissolved with TEOS and added during the last mix for TEOS-modified mixes. A total mixing time of 3 minutes was used to allow for uniform distribution.

Specimens were pressed into steel moulds with the assistance of a vibrating table. Specimens were extracted and cured in a humid room at 23±2°C and 95±5% relative humidity for 24 hours until testing.

Test Methods

Tensile Strength Test

Splitting tensile strength was tested according to ASTM C496 [18] on cylindrical specimens with a diameter of 100 mm and height of 200 mm. Tests were conducted at ages of 7, 28, and 90 using a universal testing machine with the test rate of 1.0 MPa/min.

Impact Strength Test

Impact resistance was performed by drop-weight testing on 150 mm cube specimens according to procedures in ACI 544.2R [19]. A 4.5 kg hammer was dropped repeatedly from a distance of 305 mm until specimen failure. Impact strength was measured as total energy absorbed at failure.

Chloride Penetration Test

Rapid chloride penetration test (RCPT) was performed on 100 mm diameter × 50 mm thick samples according to ASTM C1202 [20]. The test measured total charge passed through samples in 6 hours under 60V applied voltage. Results were ranked according to ASTM C1202 [20] ratings of chloride penetrability.

Shrinkage Test

Drying shrinkage was measured on 40 × 40 × 160 mm prismatic specimens following the method provided in ASTM C596 [21]. First curing for 7 days, followed by exposure to laboratory air at 23±2°C and 50±5% relative humidity, was carried out. Periodic interval length readings for 120 days were obtained with a digital length comparator.

RESULTS AND DISCUSSION

Indirect Tensile Strength (ITS)

The indirect tensile strengths (ITS) results shown in figure 1 and table 5 show a consistent decrease for both 7-day and 28-day strengths for all modified mixes compared to the reference mix. The control mix recorded the highest ITS of 4.5 MPa at 28 days, while the lowest was that of S15 at 3.7 MPa.

Fly ash replacement (F25, F30) led to lower early-age strength owing to enhanced initial reactivity of fly ash [22]. The trend is as predicted since fly ash takes longer to react pozzolanically. However, its addition with tetraethyl orthosilicate (TEOS) seems to reverse the strength loss. Mix S10 (1% TEOS) achieved 4.2 MPa, higher than F25 and F30, suggesting enhanced pozzolanic reactivity and increased microstructure density upon TEOS addition [23].

Table 5: Indirect tensile strength test results

Mix ID	Indirect Tensile Strength MPa	
	7 Days	28 Days
Ref	3.8	4.5
F25	3.2	4.1
F30	2.9	3.8
S05	3	3.9
S10	3.1	4.2
S15	2.8	3.7

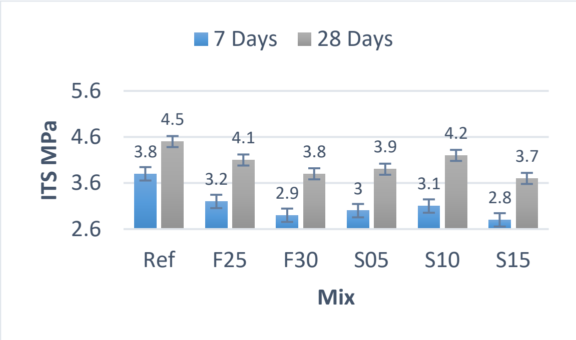

Figure 1. ITS test result

Impact Strength

Impact resistance of the mortars had similar trends as illustrated in figure 2 and table 6. The control mix attained 162 J at 90 days. The mixes modified with TEOS, S10 was optimal with 165 J, meaning that an intermediate content of TEOS (1%) enhances toughness and energy absorption. Such enhancement is likely due to enhanced particle packing and matrix densification brought about by silica gel formation from hydrolysis of TEOS [24].

Single fly ash mixtures (F25 and F30) alone showed lower values due to inadequate early-age matrix development, while high TEOS compositions (1.5%) in S15 caused marginal performance reduction, likely due to an excess amount of unreacted silica, which caused brittleness [25].

Table 6: RCPT test results

Mix ID	Impact (Joule)	
	28 Days	90 Days
Ref	145	162
F25	132	158
F30	118	148
S05	125	152
S10	138	165
S15	115	142

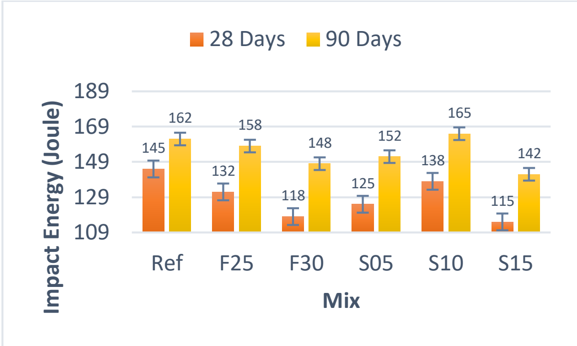

Figure 2. RCPT test results

RCPT (Rapid Chloride Penetration Test)

RCPT results indicate dramatic improvement in durability upon incorporation of TEOS (table 7 and figure 3). The control sample was 2850 Coulombs, establishing a moderate permeability to chloride as per ASTM C1202. All the amended mixes, especially S10 (2432 C) and S15 (2180 C), showed lower permeability, indicating improved pore structure fining.

TEOS generates a secondary C–S–H gel network upon hydrolysis, which reduces capillary porosity and retards ion transport [26]. Permeability was reduced by fly ash in isolation (F25: 2680 C), but the coupling with TEOS worked better, especially at higher dosages, confirming its synergic effect.

Table 7: RCPT test results

Mix ID	RCPT (Coulombs)
	28 Days
Ref	2850
F25	2680
F30	2920
S05	2450
S10	2432
S15	2180

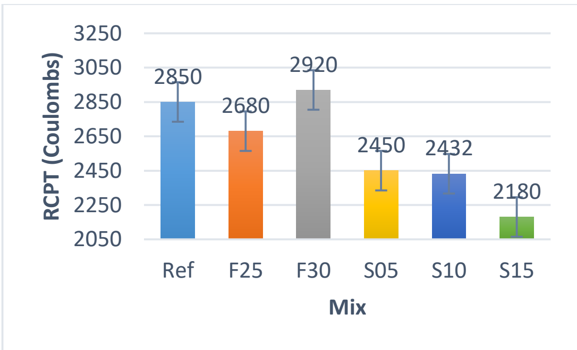

Figure 3. RCPT test results

Shrinkage

The shrinkage values (microstrain) decreased by incorporating fly ash as well as TEOS. The control mix recorded 424 με at 28 days, while S15 recorded the lowest value of 311 με. The reduction is because of the better pore structure and reduced rate of water loss due to the gel formation of TEOS [27].

Moreover, progressive hydration and pozzolanic reaction of fly ash created less internal stress and reduced autogenous shrinkage [28]. TEOS seems very effective in minimizing shrinkage regardless of the effect of fly ash alone.

Table 8: Results of shrinkage

Mix ID	Shrinkage (microstrain)	
	7 Days	28 Days
Ref	180	424
F25	161	379
F30	140	353
S05	144	352
S10	137	324
S15	132	311

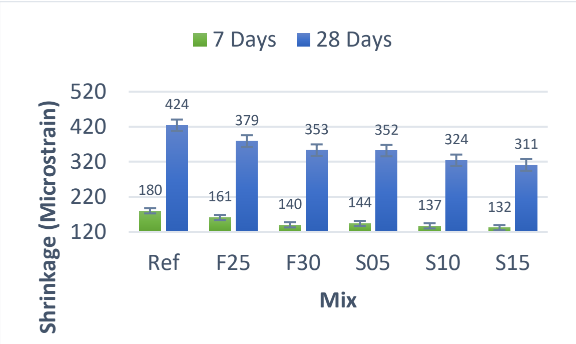

Figure 4. Results of shrinkage

CONCLUSION

The current study examined the effect of incorporating tetraethyl orthosilicate (TEOS) into fly ash-based cement mortar on mechanical properties, durability, and shrinkage behavior. The results validate that even though the addition of fly ash alone (25% and 30%) caused a decrement in indirect tensile strength as well as impact resistance due to its slow pozzolanic activity, the introduction of TEOS significantly improved these values, especially at 1% dosage. The S10 combination possessed the optimum mechanical properties among the modified combinations with the 28-day tensile and 90-day impact resistance nearing or surpassing that of the control mixture. As for durability, TEOS greatly enhanced resistance to chloride ion penetration as attested by the reduced RCPT values. The permeability through the mortar material decreased cumulatively as the content of TEOS increased, and showed the densification of pore structure and enhanced refinement of microstructure. Drying shrinkage values were also reduced remarkably for the TEOS-modified samples and were lowest in S15. This indicates that TEOS not only enhances strength and toughness but also plays a major role in controlling shrinkage volumes. This might happen because of secondary C-S-H gel formation. Among various samples S15 exhibits most favorable shrinkage and permeability properties. However, this sample revealed a slight reduction in tensile and impact strength values. This indicates that TEOS content beyond 1% makes a material stiff and/or prone to micro-cracking. Thus, a TEOS content of 1% (S10) was taken as optimal. The results imply that TEOS may be used effectively as a chemical additive sourced from a silica material. However, future studies based on studies concerning microstructure assessment studies, durability for a prolonged period in aggressive environments, and cost-benefit ratio studies are recommended.

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CONFLICT OF INTEREST

The authors declare no conflict of interest.

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