

Influence of triethanolamine on the hydration and the strength development of cementitious systems

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The influence of triethanolamine (TEA) at varying dosages on the strength development of pure cement (PC) and fly ash cement (FAC) pastes was systematically investigated. Techniques including mercury intrusion porosimetry, X-ray diffraction, thermogravimetric analysis and scanning electron microscopy were employed to characterise the microstructure of hardened cement pastes and to monitor the cement hydration process with the aim of correlating strength variations with microstructural changes caused by TEA addition. The results showed that 0.03–0.10% addition of TEA visibly enhances the early strength but decreases the compressive strength after 3 d for PC pastes. A high dosage of TEA (1%) leads to a sharp decrease in 28-d strength for both PC and FAC pastes. Mechanistic explanations regarding why TEA affects the strength development differently at different dosages and ages are discussed by considering the impacts of TEA on the hydration degree, pore structure, composition and morphology of hydration products.

Introduction

In the modern cement and concrete industry, almost all cement is produced with grinding aids and nearly all concrete is mixed with superplasticisers. In many cases, triethanolamine (TEA) is a necessary component in grinding aids and in the formulation of superplasticisers. The functions of TEA in grinding aids are to facilitate the grinding process of the clinker by modifying the surface feature of clinker particles and to increase the early strength of cement pastes to enable further addition of supplementary cementitious materials in cement products. TEA is used in superplasticisers to reduce the excessive retardation effect stemming from cement-dispersing components and to increase the early strength of hardened concrete (Aggoun *et al.*, 2008). Irrespective of how TEA is introduced – either in grinding aids or superplasticisers – it will function in the final product to affect the workability, setting behaviour and strength development of the concrete mixture by influencing the kinetics of cement hydration and microstructure of the hydration products.

Given its wide applications, extensive attention has been paid to TEA in order to understand its influence on setting behaviour, the mechanical strength of cement-based materials, the kinetics of cement hydration and the microstructure of hydration products (Ciach and Swenson, 1971a, 1971b; Gartner and Myers, 1993;

Ramachandran, 1972, 1973a, 1973b). It is well known that the effects of TEA in cementitious materials are strongly dependent on both its dosage and the composition of the cement mixtures (Cheung *et al.*, 2011). It acts as a set accelerator and enhances early strength at low dosages (e.g. 0.02–0.05% by weight of cement) (Dodson, 1990; Perez *et al.*, 2003a), while it depresses strength growth by the retardation effect on cement hydration at dosages higher than 0.1% (Heren, 1996).

Mechanistic investigations of TEA in cementitious materials have been carried out for several decades (Cheung *et al.*, 2011). Ramachandran (1972, 1973a, 1973b) concluded that TEA at a dosage of 0.1% retarded the hydration of C_3S by extending the induction period, but accelerated the hydration of C_3A and the formation of ettringite. The hydration rate of C_3S was slightly enhanced after 1 d. The ability of TEA to enhance strength was ascribed to the formation of amine–iron complexes under high pH conditions (Chaberek and Martell, 1959). Increased dissolution of iron at 10–14 h and earlier onset of sulfate depletion were observed by Gartner and Myers (1993) and Perez *et al.* (2003b) at TEA dosages of 0.24% and 0.54%. In the presence of supplementary cementitious materials, TEA was found to enhance the early strength of blended cement, especially fly ash cement (FAC), by either activating the pozzolanic reaction or promoting

fly ash dissolution by chelating Al and Fe (Lee *et al.*, 2003; Scrivener, 2010)

So far, most research regarding a mechanistic understanding of the strength enhancement effect of TEA has concentrated on the influences of TEA on the cement hydration process. As far as strength is concerned, apart from cement hydration factors such as hydration kinetics and hydration degree, morphological factors of hardened cementitious materials (e.g. pore structure and morphology of the hydration products) also play important roles, and these have unfortunately not been given sufficient attention. Some researchers have reported changes in the amount of portlandite (CH) and the morphology of hydration products by the addition of TEA to cement pastes (Ramachandran, 1972; Scrivener, 2010).

As already noted, the effects of TEA on cementitious materials depend primarily on its dosage and the components of the cementitious binders. Why and how TEA affects the strength development of cementitious materials remains incompletely understood. Many questions need to be answered. For example, why does a low dosage of TEA show a strength-enhancing effect at early ages, whereas it loses such an effect at higher addition, even though TEA has the same accelerating effect on the hydration of aluminates at varying dosage? Furthermore, why does TEA produce no enhancement in late strength or even sometimes reduces the late strength of cement pastes?

This paper systematically investigates the influence of TEA at varying dosages on the strength development of pure cement (PC) pastes and FAC pastes. X-ray diffraction (XRD), thermogravimetric analysis (TGA), mercury intrusion porosimetry (MIP) and scanning electron microscopy (SEM) were used to determine cement hydration degree and to characterise the microstructure of hardened cement pastes with the aim of correlating strength variations with microstructural changes caused by TEA addition.

Experimental method

Materials

Two types of cementitious binders were studied – PC and a composite cement (FAC) made by blending PC with fly ash at a mass ratio of 70 to 30. The PC was produced by intergrinding a clinker with 5% gypsum in a laboratory ball mill. Table 1 shows the composition of the PC and FAC, as determined by

	Composition: wt%									LOI: wt%
	CaO	SiO ₂	Al ₂ O ₃	Fe ₂ O ₃	SO ₃	MgO	K ₂ O	TiO ₂	Na ₂ O	
PC	59.79	22.43	4.46	3.94	3.80	3.68	0.99	0.42	0.16	0.87
FAC	2.65	53.64	32.62	5.97	0.75	0.41	1.24	1.43	0.47	6.73

Table 1. Chemical composition of PC and FAC

X-ray fluorescence. Table 2 shows the particle size distribution of the PC measured using a laser particle size analyser. Analytical-grade TEA without further purification was used and deionised water was used for all the experiments.

Paste experiments

Measurements of mechanical strength, hydration degree and microstructural characterisation were carried out on cement pastes prepared with a fixed water to binder mass ratio of 0.32 to minimise any bleeding effects. Cement, water and TEA (at dosages of 0%, 0.03%, 0.10% and 1.00% by weight of cementitious materials) were well mixed in a 2.5 l stirring mixer for about 5 min at 125 rpm. The mixtures were then cast in steel moulds for curing. The samples were demoulded after being cured in a standard curing room for 24 h at 20°C and relative humidity of 95%. After this, they were wet cured in a water bath at 20°C until testing. At the selected ages, the samples were moved into an acetone bath and stored for at least 24 h to terminate cement hydration. Thereafter, they were dried in an oven at 65°C for 24 h.

The compressive strength of the cement pastes was measured at ages of 1, 3, 7 and 28 d to investigate the effects of TEA on strength development. The standard testing procedure (BS EN 196 (BSI, 2005)) was followed. Cubic specimens (40 × 40 × 160 mm) of the pastes were used for strength measurement.

Non-evaporable water (NeW) measurement, XRD and TGA were used to quantitatively or qualitatively characterise cement hydration degree. In the measurement of NeW content, the cement paste samples were pre-dried at 65°C for 24 h and then dehydrated at 1000°C until their weight remained unchanged. The mass loss during dehydration was defined as the content of NeW. TGA is the most accurate method for quantifying the CH content in hardened cement pastes. TGA measurements (DTG-60H, Shimadzu, Japan) of the samples were performed by heating from

	Particle size: μm			
	<3	3–32	32–65	>65
Volume distribution: %	10.43	59.98	29.17	0.45

Table 2. Particle size distribution of pure cement

30°C to 550°C at a heating rate of 10°C/min under a nitrogen atmosphere. The CH content was obtained by determining the mass loss in the temperature range of 400–500°C using the tangent method. XRD is a useful method for semi-quantifying the content of CH in hydration products. FT scanning was carried out at a 2θ range of 17–19° on a Rigaku D/max 2550 X-ray diffractometer. The step length was 0.02° and the settle time of every step was 6 s. The integrated intensity of the diffraction peak of $\text{Ca}(\text{OH})_2$ reflects its amount.

The porosity and pore size distributions of the hardened cement pastes were determined using Hg porosimetry (Autopore, IV 9510, USA) at a maximum pressure of 420 MPa. SEM was employed to observe the morphology of the hydration products in cement pastes at 1 and 28 d in the presence or absence of 1% TEA, a value sufficient to reflect the influence of TEA on the microstructure of the hydration products at different ages.

Results and discussion

Effect of TEA on the strength development of cementitious pastes

The effects of TEA on the strength development of cementitious pastes are shown in Figure 1. It can be seen that 0.03% and 0.1% additions of TEA visibly enhanced the compressive strength of PC pastes at 1 d but decreased the compressive strength after 3 d. The enhancement effect was extended until 7 d for FAC. Adding 1% TEA did not bring about a stronger enhancement effect, but minimally affected or decreased the early strength and strongly reduced the 28-d strength.

It is known that TEA can accelerate the hydration of C_4AF and C_3A phases in cement, as well as facilitate the dissolution and therefore promote the pozzolanic reaction of fly ash (Lee *et al.*, 2003; Perez *et al.*, 2003a). These two aspects may explain the enhancement effect on the compressive strength of PC and FAC pastes at early ages. For the strength development of FAC, a question of whether TEA acts mainly on PC or on fly ash arises. To answer this question, the relative compressive strength is defined as the compressive strength of cement pastes with various TEA dosages divided by the compressive strength of blank cement pastes at the same age. The relative compressive strengths as a function of TEA dosage are plotted in Figure 2. Figure 2(a) illustrates that, at ages of 1 and 28 d, TEA imposes similar effects on the relative compressive strengths of both PC and FAC pastes, suggesting that the effect of TEA is on cement and not on fly ash at the aforementioned ages. On the other hand, for the compressive strengths at 3 and 7 d, TEA manifests different effects on FAC and PC pastes, implying that at these ages, the stimulative effect of TEA on the reaction of fly ash in FAC dominates the effect of TEA on the strength development of FAC pastes.

As far as the strength of cementitious materials is concerned, many factors may play important roles at different ages, including

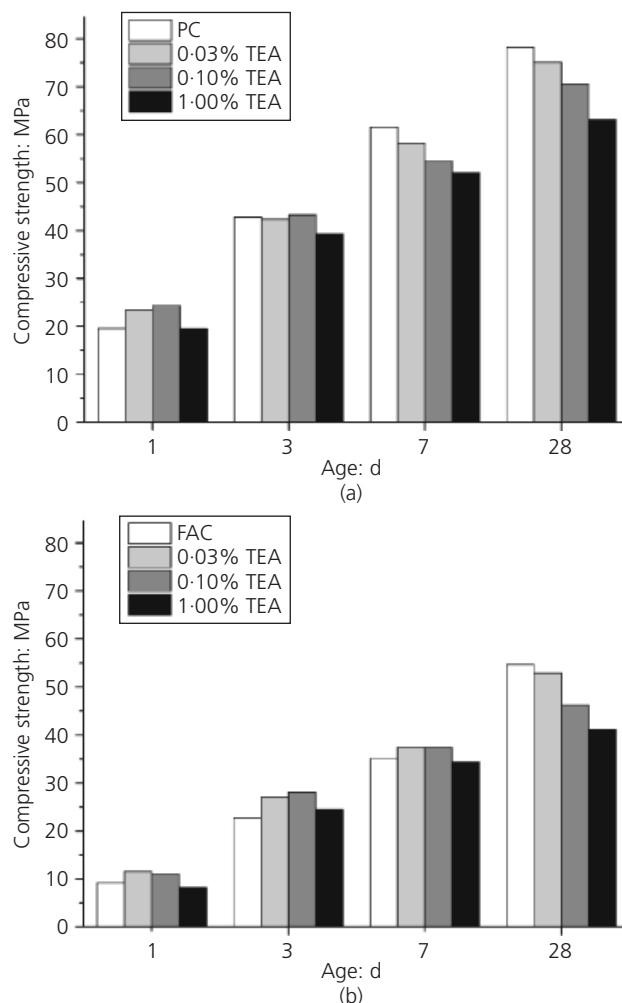


Figure 1. Compressive strength of (a) PC and (b) FAC pastes with varying dosage of TEA

pore structure and bulk strength of the hardened cement paste, which is primarily determined by the hydration degree as well the microstructure of the hydrates. Given that the hardened cement pastes is a porous material, the pore structure (including the porosity and pore size distributions) is naturally one of the most important factors for strength. Another key factor that determines strength is the bulk strength of the hardened cement paste. In this paper, the bulk strength is defined as the intrinsic strength of the hydrates themselves with zero porosity, in which both the hydration degree and microstructure of cement hydrates are two dominant elements (Odler, 1991). For the early strength, the hydration degree of cement is usually considered the most important factor whereas, for late strength, the microstructure of the cement hydrates should be taken more into account. In terms of strength, the morphology of CH must be the focus in many cases – the CH phase is the weakest part in hardened cement pastes and is often responsible for micro-crack initiation and growth.

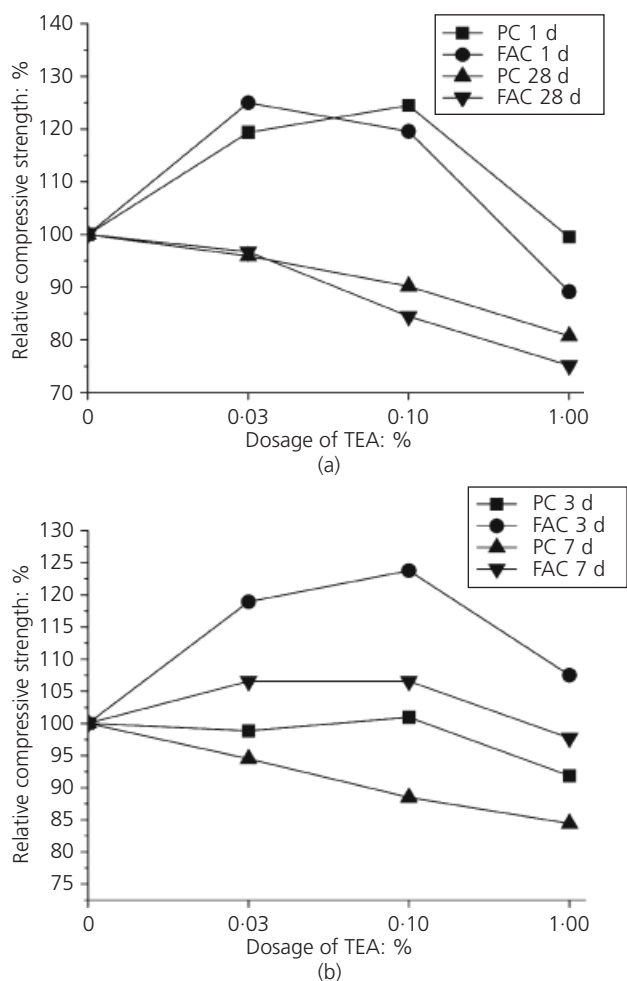


Figure 2. Comparison of the effects of TEA on the strength of PC and FAC pastes: (a) 1 and 28 d; (b) 3 and 7 d

Pore size distribution

The pore size distribution of hardened cement pastes was measured by MIP (Table 3). At 3 d, the cumulative porosity of both PC and FAC pastes clearly increased with the addition of TEA, implying that TEA decreases the degree of hydration or changes the microstructure of hydration products in the pastes. At 28 d, the cumulative porosity of the PC paste was minimally affected by the addition of TEA, whereas that of the FAC paste was obviously reduced by TEA, which accords with the conclusion that TEA facilitates the reaction of the FAC system (Lee *et al.*, 2003). Looking into more detail about the pore size distribution of the pastes, a consistent phenomenon is that TEA addition increased the proportion of pores larger than 100 nm but decreased the volume fraction of smaller pores (<100 nm) for both PC and FAC pastes at 3 and 28 d.

On the basis of the information on the pore structures of the hardened pastes, it can be inferred that the lowered 28-d strength by the addition of TEA could be a result of the larger macroporosity. However, a coherent correlation between early strength enhancement (at 1 and 3 d) by the addition of TEA and pore structure information cannot be found as it is usually understood that a higher porosity and larger pore size lead to lower strength. This suggests that there must be some other factors behind determining variations in strength by the addition of TEA.

Rather than the influence of TEA on pore structure, it is believed that the effects of TEA on the bulk strength of hardened pastes may be crucial to their overall strength. It is well accepted that the bulk strength of hardened cement pastes is determined by the hydration degree of cement and the microstructure of the hydrates. Three methods were thus used to characterise cement hydration degree in this study

	Cumulative porosity:			
	>1000 nm: ml/g	1000–100 nm: ml/g	<100 nm: ml/g	<100 nm: ml/g
3 d				
PC	0.152	0.0084	0.0622	0.0809
PC + 0.1% TEA	0.168	0.0155	0.0737	0.0785
FAC	0.161	0.0109	0.0506	0.0997
FAC + 0.1% TEA	0.181	0.0222	0.0689	0.0898
28 d				
PC	0.088	0.0080	0.0317	0.0486
PC + 0.1% TEA	0.089	0.0123	0.0371	0.0398
FAC	0.134	0.0100	0.0393	0.0851
FAC + 0.1% TEA	0.118	0.0120	0.0443	0.0619

Table 3. Pore size distribution of hardened cement pastes with and without TEA

- measuring the content of chemically bonded water
- determining the CH phase content by TGA and XRD
- measuring the hydration heat by isothermal calorimetry.

Degree of cement hydration

Non-evaporable water (NeW) measurement

The NeW content of the hardened pastes was measured to reflect the hydration degree of PC and FAC (Figure 3). A clear trend is observed: for both PC and FAC, the addition of TEA at various dosages increases the content of NeW in hardened pastes older than 3 d, indicating that TEA accelerates the cement hydration process to some extent. At age 1 d, the NeW content of the pastes is less affected by TEA at a lower dosage, and a 1% addition of TEA decreases the hydration degree for both PC and FAC pastes. Because of the aforementioned effect of TEA on fly ash compared with blank cements, a 0.03% addition of TEA increases the NeW content in the FAC pastes much more than in PC pastes at ages ≥ 3 d. This again verifies the conclusion that TEA accelerates the reaction of the FAC system.

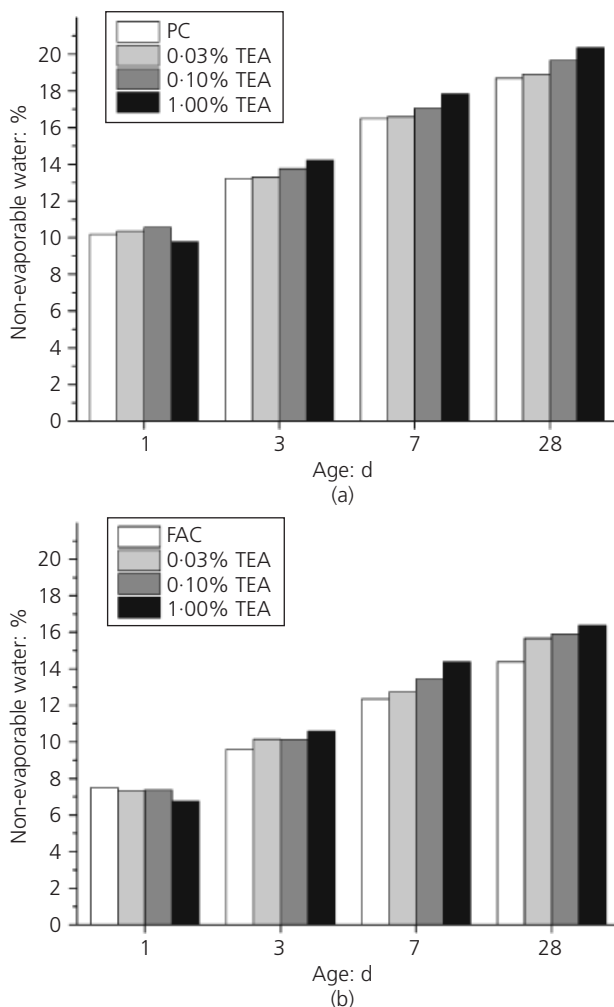


Figure 3. Effect of TEA on the content of NeW of hardened pastes: (a) PC; (b) FAC

As a higher hydration degree of cement usually results in a higher strength of hardened cement-based material (especially at early ages), the results for NeW cannot fully explain the effect of TEA on the strength development of PC and FAC pastes described earlier. For example, a 0.03% addition of TEA in PC pastes slightly increased the NeW amount at 1 d but significantly enhanced the 1-d strength, whereas at 28 d, an evident increase in NeW did not bring any positive effects on strength growth. At 28 d, a 1% addition of TEA still increased the NeW content in both the PC paste and FAC paste, while this dosage was notably harmful for strength development (Figure 3). This implies that the microstructure of hydration products may also be changing considerably.

Ca(OH)₂ content

The content of CH in hardened cement pastes is another indicator of the quantification of the degree of cement hydration. TGA and XRD were carried out to determine the CH content in hardened PC and FAC pastes (Figures 4 and 5). Surprisingly, compared to

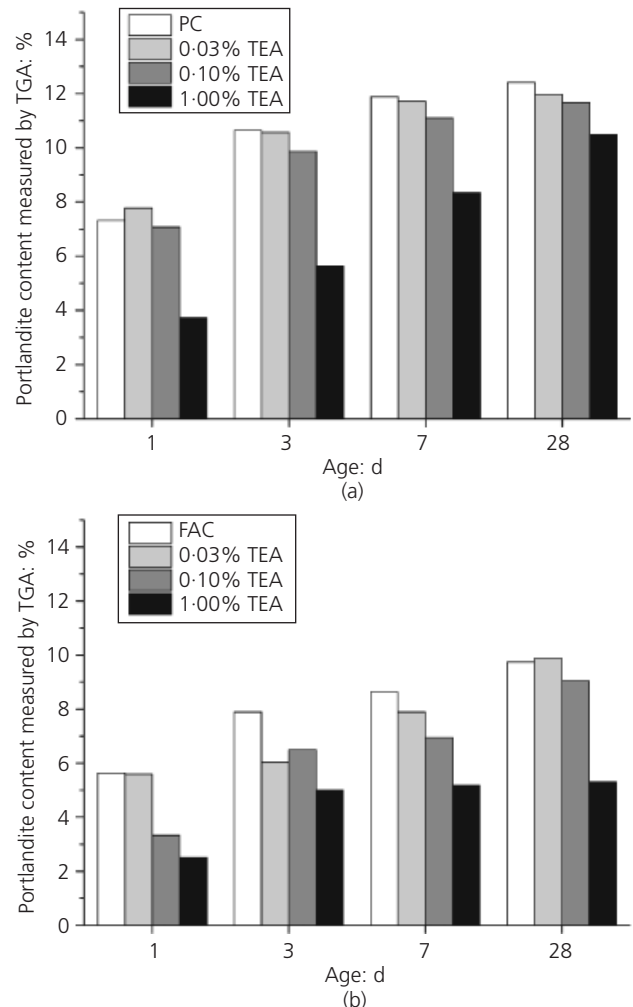


Figure 4. Amount of Ca(OH)₂ in cementitious material pastes with and without TEA determined by TGA: (a) PC; (b) FAC

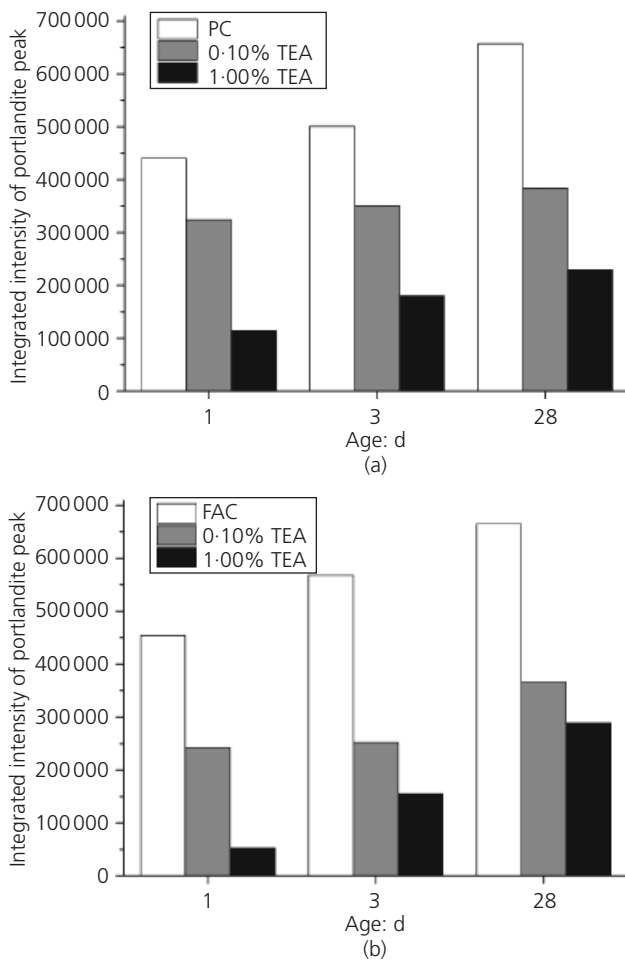


Figure 5. Amount of $\text{Ca}(\text{OH})_2$ in cementitious material pastes with and without TEA determined by XRD: (a) PC; (b) FAC

the blank pastes, the addition of TEA led to less CH content based on the TGA and XRD results. This finding contradicts the increased hydration degree measured by the NeW content. Several researchers have reported a similar phenomenon in which TEA lowers CH production from hydration in the C_3S system as well as in CH cement systems (Ciach and Swenson, 1971b; Ramachandran, 1973b). Scrivener (2010) explained that the reduced amount of CH is a result of the formation of microcrystal CH embedded by calcium silicate hydrate (C-S-H) phases or a heightened C/S ratio in the C-S-H phase.

It should be noted that the reduction in CH content in FAC pastes was higher than in the PC pastes when the effects of TEA at the same dosage are compared. The higher reduction in CH content in the FAC pastes may have resulted from the accelerated pozzolanic reaction of fly ash in FAC pastes by TEA and the accelerated consumption of CH by pozzolanic reaction leads to a lower CH content in FAC pastes than in PC pastes. Another interesting phenomenon is that, at a certain dosage of TEA in the PC and FAC pastes, the reduction in CH content determined by

XRD was higher than the results from the TGA measurement. This would imply that TEA not only reduces the content of CH in cement pastes but may also change the crystal morphology of the CH produced because CH crystals with perfectly ordered lattices are measurable by XRD. Conversely, the CH crystals may have a large amount of flaws and defects or those microcrystals may not be measurable by XRD. The difference in test results between XRD and TGA may imply that – aside from reducing the content of CH produced – the addition of TEA may cause a morphological change in the CH crystals to crystals with more flaws and defects, even to non-crystalline CH.

SEM

SEM observations were conducted to compare the morphologies of the cement pastes with or without TEA at different ages (Figure 6). Figure 6(a) shows typical lamellar $\text{Ca}(\text{OH})_2$ crystals with large dimensions (tens of micrometres). In comparison, with the addition of 1% TEA, the morphology of the $\text{Ca}(\text{OH})_2$ crystals is extremely altered, as shown in Figures 6(b) and 6(c) for the cement paste at an age of 1 d. Most $\text{Ca}(\text{OH})_2$ crystals are distorted and actinomorphic with a much smaller size than the parallel, lamellar crystals with larger sizes in the neat cement paste. Figures 6(d) and 6(e) show that, at 3 and 28 d of age, large amounts of distorted $\text{Ca}(\text{OH})_2$ crystals (Figure 6(d), area1) continue to appear with a minority of the normal lamellar, parallel-arranged $\text{Ca}(\text{OH})_2$ crystals (Figure 6(d), area2), but are considerably smaller in size. These are believed to have been produced after complete depletion of TEA in the cement paste. As a result of the free space limitation, the late-produced $\text{Ca}(\text{OH})_2$ crystals have to be smaller in size.

A completely hydrated cement paste usually contains about 20–25% $\text{Ca}(\text{OH})_2$ phase by mass with parallel-stacked lamellar crystals and dimension of tens of micrometres. The $\text{Ca}(\text{OH})_2$ crystal phase with a lamellar structure is often regarded as the weakest part in a hardened cement paste, responsible for micro-crack initiation and growth. The variation in content and morphology of the CH phase may thus play a key role in the strength variation of cement pastes. This may explain why TEA does not decrease the early strength (or even increase the 1-d strength of PC pastes to some extent) even though it does increase cumulative porosity (especially for pores of diameter >100 nm) and does not largely increase the degree of cement hydration in the PC pastes. Clear and consistent trends in microstructural changes of the C-S-H phase caused by TEA addition have not yet been observed.

Mechanism of TEA influencing strength

The results reported thus far indicate that the addition of TEA to a cement paste exerts an influence over various aspects including hydration kinetics, pore structure and composition and morphology of the hydration products (Table 4). Some effects are beneficial to the strength development of hardened cement pastes, whereas others may be disadvantageous to strength growth. The overall effect of TEA on the strength development of cement

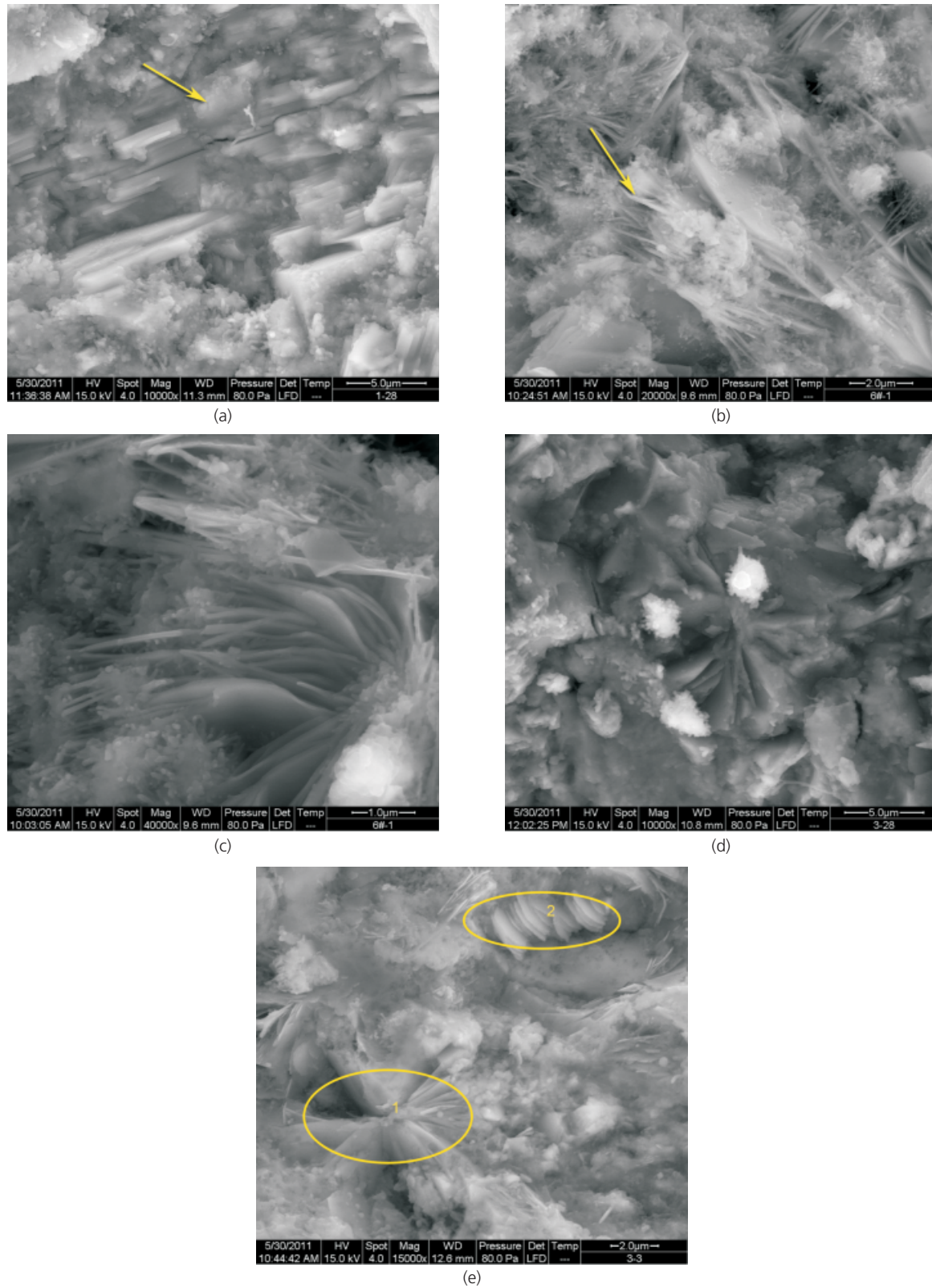


Figure 6. Effect of TEA on the morphology of Ca(OH)_2 crystals in cement hydrates: (a) blank cement, 1 d; (b) cement + 1% TEA, 1 d; (c) cement + 1% TEA, 1 d; (d) cement + 1% TEA, 3 d; (e) cement + 1% TEA, 28 d

	Effect
NeW content	Increases the content of NeW after 3 days
CH content (by XRD and TGA)	Decreases the content of CH phase and alters the morphology of CH crystals
FAC	Accelerates the dissolution of fly ash and pozzolanic reaction after 24 h

Table 4. Effects of TEA on the hydration process and the hydration products of cementitious materials

paste is the counterpoising of these advantageous and unfavourable factors.

Conclusions and future perspectives

To understand the intrinsic mechanism of the influence of TEA on the strength development of cementitious systems, the effects of TEA on pore structure, hydration kinetics and microstructure of hydration products were systematically investigated. The conclusions drawn from this study can be summarised as follows.

- Additions of 0.03% and 0.10% TEA visibly enhance the compressive strength of PC pastes at 1-d age but decrease the compressive strength after 3 d. Enhancement of the early strength due to TEA is more pronounced in FAC pastes than in PC pastes. A 1% TEA dosage leads to a sharp decrease in 28-d strength for both PC and FAC pastes.
- Comparing the PC pastes and FAC pastes, the effects of TEA on strength growth at varying dosages present similar trend at ages of 1 d and 28 d. However, at 3 and 7 d, a stronger strength enhancement was found in FAC pastes than in PC pastes. This must be related to TEA's facilitation in the dissolution of fly ash and the pozzolanic reaction by acting as an ion transporter in the FAC system.
- The addition of TEA results in a variation in the pore structure of hardened cement pastes, which must be a result of changes in the degree of hydration and the microstructure of hydration products. At age 3 d, the cumulative porosity of both PC and FAC pastes clearly increased with the addition of TEA. At 28 d, the cumulative porosity of the PC paste was only minimally affected by the addition of TEA, while that of FAC pastes was obviously reduced. The addition of TEA increased the proportion of pores larger than 100 nm, but decreased the proportion of the pores smaller than 100 nm.
- The NeW measurement suggests that TEA increases the hydration degree of cement at all ages until 28 d for both PC and FAC systems. The TGA and XRD results indicate that the CH content is reduced remarkably by the addition of TEA.
- The addition of TEA alters the morphology of $\text{Ca}(\text{OH})_2$ crystals from the typical parallel-arranged lamellar crystals to actinomorphic distorted crystals of a much smaller size.

On the whole, the effect of a low dosage (<0.1%) of TEA on the enhancement of early strength (1 d) of PC pastes can be explained by accelerated early hydration, reduced CH content and

altered CH morphology. At a higher dosage (1%), the retarding effect of TEA on C_3S hydration accounts for the lowered early strength. The harmful effect of TEA on the late strength (28 d) of PC pastes must be related to unfavourable pore structures. Apart from the aforementioned effects, the facilitated dissolution of fly ash and the pozzolanic reaction in FAC pastes, which is beneficial to strength growth, should be taken into account.

This paper has shown the effects of TEA on the strength development of cement pastes and has given some mechanistic explanations by disclosing the impacts of TEA on the hydration kinetics, pore structure and composition and morphology of hydration products. However, a clearer comprehension of how TEA influences so many aspects is required to fully understand the working mechanism of the strength enhancement. The composition of the pore solution before setting, the complexation of TEA with cations in the pore solution, the crystallisation of C-H, and the nucleation and growth of C-S-H gel are worthwhile topics for further study.

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