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# Effect of triisopropanolamine on compressive strength and hydration of cement-fly ash paste



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Baoguo Ma, Ting Zhang, Hongbo Tan\*, Xiaohai Liu, Junpeng Mei, Huahui Qi, Wenbin Jiang, Fubing Zou

State Key Laboratory of Silicate Materials for Architectures, Wuhan University of Technology, Wuhan 430070, PR China

#### HIGHLIGHTS

• TIPA can increase compressive strength of cement-FA system.

TIPA can accelerate the hydration of both cement and FA.

• TIPA can facilitate the dissolution of FA.

• Air-entraining effect of TIPA negatively affects compressive strength.

#### ARTICLE INFO

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#### ABSTRACT

This paper aims to investigate the effect of triisopropanolamine (TIPA) on compressive strength and hydration of cement-fly ash paste. The samples with various dosages of TIPA were prepared with 30% fly ash (FA) and 70% cement (water/binder ratio by weight = 0.38), and cured under the standard condition. The compressive strength, pore structure, hydration process, and hydration products were investigated. The results show that TIPA can obviously increase the compressive strength of cement-FA system at the age of 7 d and 60 d, and the reasons are involved in pore structure and hydration of cement-FA system. Pore structure was characterized with mercury intrusion porosimetry, and the results show that TIPA can reduce total porosity but increase the amount of pore with size more than 50 nm, implying the air-entraining effect with negative effect on compressive strength. The result suggests that TIPA and defoaming agent should be used together to minimize the negative effect in real concrete. Furthermore, analysis of hydration products shows that TIPA can accelerate the hydration of both cement and FA, and this can also be illustrated from solid-state nuclear magnetic resonance. It is noticed that TIPA can hasten the conversion of AFt to AFm, which can be indicated from hydration heat. Additionally, the acceleration of pozzolanic reaction of FA is because TIPA can accelerate the dissolution of aluminate, silicate, and ferric into liquid paste which was demonstrated from morphology characterization and the change of ions in pore solution. Such results would be expected to provide experience for the use of alkanolamine in promoting the performance of cement-based materials.

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#### 1. Introduction

In recent decades, the use of supplementary components in cement-based materials increasingly attracts attention in industrial area, because of both environmental and economic benefits [1–4]. Fly ash (FA), a by-product in coal-fueled power plants, has been developed as one of the most popular supplementary components. Even though many advantages can be found with addition of FA [5–8], one concerned issue cannot be ignored that poor mechanical performance would be found if the threshold replace-

\* Corresponding author. E-mail address: thbwhut@whut.edu.cn (H. Tan).

https://doi.org/10.1016/j.conbuildmat.2018.05.117 0950-0618/© 2018 Elsevier Ltd. All rights reserved. ment was surpassed [9–12]. The effect of FA on mechanical performance is involved in two aspects: one is that finer particles in FA can fill into the pore [13], with positive effect on refining the pore structure to contribute to the mechanical performance. The other is that FA can react with the calcium hydroxide (CH) [14], known as pozzolanic reaction, promoting the formation of hydrates to contribute to the mechanical performance. However, if excessive FA were used, the CH generated by the hydration of cement minerals would be not enough for activating the pozzolanic reaction of FA, and in this case, the hydration degree of whole system would be reduced and the mechanical performance would be significantly declined.

Table 1
Chemical composition of cement and FA.

		Loss	SiO <sub>2</sub>	$Al_2O_3$	Fe <sub>2</sub> O <sub>3</sub>	SO <sub>3</sub>	CaO	MgO	K <sub>2</sub> O	Na <sub>2</sub> O
Cement	wt%	3.82	24.08	4.72	2.46	2.31	58.24	1.95	1.02	0.27
FA	wt%	5.97	48.33	31.69	4.14	1.37	4.12	0.50	1.34	0.37

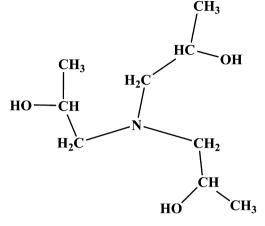


Fig. 1. Schematic diagram of molecular structure of TIPA.

In cement-FA system, in order to improve the mechanical performance, accelerating hydration of both FA and cement should be considered. In fact, the essence of the pozzolanic reaction of FA is the dissolution of ions from FA surface into liquid phase to participate in hydration, resulting in the formation of hydrates, with contribution to mechanical performance. Taking sodium sulphate for instance, sodium sulphate has been accepted as one of the most popular activators for FA [15]. One explanation for the mechanism is that sulphate can facilitate the formation of ettringite with nano particle size as nucleation seeds, which can hasten the hydration of aluminates and also induce the hydration of other phase [16]. Another reason can be explained that sodium ions provide higher alkalinity to accelerate the depolymerization of silicon and aluminum structure in FA, which means that the dissolution of FA can be promoted. Additionally, organic alkali can also facilitate the dissolution of FA to obviously increase the early strength of cement-FA system. Taking triethanolamine (TEA) for example, TEA can accelerate the dissolution of aluminum, ferric, and calcium of FA into pore solution, resulting from the complexation of TEA with  $Al^{3+}$  and  $Fe^{3+}$  [17].

In terms of the acceleration of cement hydration, as reported in the literatures, triisopropanolamine (TIPA) shows high efficiency [18], and the probable reason is due to the accelerated formation of hydrated calcium sulphoaluminate and the induced dissolution of ferric by forming a complex TIPA-Fe [19]. Since the ferric phase, with much lower temperature of melting point, would exist on the surface of clinkers, the promoted dissolution of ferric phase would hasten the dissolution of other phases. It is deduced that in cement-FA system, the promoted cement hydration by TIPA can facilitate the formation CH, and possibly, this would provide stronger alkali environment to hasten the pozzolanic reaction of FA. Additionally, another reason should be noticed that TIPA might induce the dissolution of aluminum, ferric, and silicate into liquid phase, which could also facilitate the pozzolanic reaction of FA. In this case, the addition of TIPA to promote the strength of cement-FA system would be expected. However, no direct evidence can be found.

In this study, the effect of TIPA on compressive strength and hydration of cement-FA system was systematically investigated.

Pore structure was investigated with mercury intrusion porosimetry (MIP). The hydration products were characterized with scanning electron microscope (SEM), thermos-gravimetric analysis (TGA), and X-ray diffraction (XRD). The reaction degree of cement and FA was assessed with solid-state nuclear magnetic resonance (NMR). The dissolution of FA was assessed with SEM and inductive coupled plasma emission spectrometer (ICP). Finally, the mechanism behind the improvement of TIPA in mechanical performance was revealed in the terms of pore structure and hydration process. Such results were expected to broaden the use of alkanolamine in cement-based materials and also provide useful experience to promote the utilization of FA in real concrete.

#### 2. Materials and test methods

#### 2.1. Materials

#### 2.1.1. Cement and fly ash

A Portland cement (P.I 42.5, Wuhan Yadong Cement Co., Ltd.) in accordance with the requirements of GB 175-2007 (Chinese standard) and class F-II fly ash (FA) in accordance with the requirements of GB/T 1596-2005 (Chinese standard) were used in this study. The chemical composition of cement and FA was obtained with X-ray Fluorescence (XRF, Axios advanced, made by PANalytical B.V., Holland), and the results are shown in Table 1.

#### 2.1.2. TIPA

A reagent-grade triisopropanolamine (TIPA, anhydrous white solid,  $\geq$ 95.0% purity, made by Aladdin Biochemical Technology Co., Ltd., Shanghai, China) was used. Additionally, the added dosage of TIPA was recorded as the solid amount. The molecular structure of TIPA are shown in Fig. 1.

#### 2.1.3. Preparation for specimens

Cement-FA paste (C-FA: 30% FA and 70% cement) with different dosages of TIPA (0%, 0.03%, 0.06%, and 0.10%) was prepared with a water/binder ratio of 0.38 by weight. TIPA was dissolved in water in advance and the solution was then mixed with the binder to prepare the paste. The fresh pastes were cast in 40 mm  $\times$  40 mm cubic metallic moulds and cured in the >90% R.H. and 20  $\pm$  1 °C chamber for 24 h, and then demoulded and further cured with the same condition. At the age of 7 d and 60 d, the compressive strength of the sample was measured. The samples were also broken into small pieces and immediately immersed into ethanol in order to stop hydration. The pieces were dried in a vacuum drier at 20 °C, and then prepared for the measurement of pore structure. Additionally, these specimens were also grinded by hand, and the powder, which could pass through a 63 µm sieve, was prepared for analysis of hydration products.

#### 2.2. Test methods

#### 2.2.1. Compressive strength

The samples were tested with the compressive machine at a rate of 0.6 MPa/s. For each mix, three samples were tested, and the average value was the result.

#### 2.2.2. Ions dissolution of fly ash

Firstly, pore solution with different concentrations of TIPA (0–20.0 g/L) was prepared with KOH and NaOH (K<sup>+</sup>/Na<sup>+</sup> = 1:1; pH = 13.0). One gram of FA was added into these solutions (20.0 g), respectively, and mixed. The suspensions was sealed in a plastic container, with constant temperature of  $20 \pm 1$  °C. For each 12 h, the containers were shocked in order to make the suspension to be even.

At the age of 12 h, 1 d, 7 d, 14 d, 28 d, and 60 d, the suspension was centrifuged at 3600 r/min for 10 min in a centrifuge, and the content of Al, Fe and Si in supernatant solution were tested with inductive coupled plasma emission spectrometer (ICP, Optima 4300 DV, made by Perkin Elmer Ltd., USA) to investigate the effect of TIPA on the dissolution of FA.

In addition, the solid was dried in a vacuum drier with the temperature of  $20 \pm 1$  °C, and then the surficial morphology was characterized with field emission scanning electron microscope (SEM).

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