



Hydration of ternary cement in the presence of triisopropanolamine



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HIGHLIGHTS

- Mixture design introduced to interpret the strength development of a ternary blend, showing that the interactions of CaCO₃ and slag with TIPA were pronounced.
- Hydration of C₄AF phase promoted by TIPA resulting in consumptions of CH and sulfate in the early stage.
- Reaction of slag improved by TIPA in two ways: acceleration of the silicate reaction yielding more CH, and the faster dissolution of slag under highly alkaline conditions.
- Formation of carboaluminate hydrates favored as the result of boosted C₄AF/C₃A–CaCO₃ reactions by TIPA.

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ABSTRACT

The effects of triisopropanolamine (TIPA) on the strength development and hydration kinetics of a ternary cement containing slag and CaCO₃ were investigated by mixture design. The strength development of the blend was considerably enhanced by the accelerated chemical reactions on each component. A very strong interaction effect of slag and CaCO₃ on the strength enhancement prior to 7 d was observed. The addition of TIPA promoted the hydration of laboratory cement by promoting the dissolution of ferrite, which resulted in more CH being released to participate in the reaction with slag. The dissolution of the slag itself was markedly accelerated by TIPA in a simulated highly alkaline pore solution. The use of TIPA promoted the formation of carboaluminate due to the accelerated C₄AF/C₃A–CaCO₃ reaction, which stabilized the formation of Aft during hydration. In addition, it confirmed that Fe ions were able to enter the Aft structure during hydration with TIPA.

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1. Introduction

In recent decades, supplementary cementing materials (SCMs) have been widely used to partially replace clinker to produce composite cement, which provides both environmental and economic benefits. The physical characteristics and the substitutions of SCMs in cement vary greatly due to their chemical and mineral compositions [1]. Additionally, once the threshold replacement level is surpassed, the performances of the cement, particularly its mechanical properties, are impacted [2]. Limestone filler, which is generally considered to be inert, is also added to cement. According to the general specifications of ordinary Portland cement (OPC), a limestone addition of less than 5% is introduced during clinker milling [3], which is able to modify the performance of the finished cement because its filler effect is able to accelerate early hydration

[4,5]. Due to the shortage of hydraulic SCMs and primarily economic concerns, more limestone is added to produce blended cement, and less than 25% by mass is generally allowed [6]. Under such circumstances, the strength development of the cement changes, and in some cases, the later-age strength markedly decreases [7,8].

Triisopropanolamine (TIPA), a type of alkanolamine discovered in the early 1990s, is widely used as a grinding chemical in the clinker grinding process to reduce agglomeration in the ball mill and to modify the particle distribution of the finished cement [9,10]. TIPA also enhances the mechanical properties of cement. The addition of TIPA to Portland cement significantly improves the strength development after 3–7 d [11–13]. Gartner proposed that TIPA facilitates ion transport by chelating Fe³⁺ and Al³⁺ from the hydration product of ferrite and then releasing it into the aqueous phase, thereby increasing the dissolution of ferrite and promoting the overall silicate reaction [14]. Ichikawa et al. investigated the addition of 100 ppm TIPA in cement with a limestone replacement of up to

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10% by mass [15]. The results indicated that TIPA tended to enhance the compressive strength of the blended cement after 7 d, particularly the 28 d strength, and the rate of strength enhancement increased proportional to the level of limestone replacement.

At the end of 2015, the specification of PC 32.5 was removed from the national standard in China to regulate the use of SCMs in Portland cement and to promote the manufacturing of composite cement with a higher grade (PC 42.5 or above). The incorporation of SCMs is undoubtedly one of the key solutions to enhancing the quality of cement. In addition, efficient use of chemical additives is necessary because the early strength of composite cements is activated by such chemicals in most conditions. The aim of this study is to interpret the reaction mechanisms of SCMs in the presence of TIPA to facilitate their applications in field. The strength enhancement of a ternary blend (~25% slag/CaCO₃ by mass) resulting from TIPA was investigated from the perspective of cement hydration. Insights on the behavior of TIPA in the Portland cement system were also obtained.

2. Materials and methods

To investigate the hydration mechanism of Portland cement in the presence of triisopropanolamine, a Portland cement clinker from a Huaihai cement manufacturer was selected (XRF and QXRD analyses are shown in Table 1), and it was ground in a laboratory ball mill with 5% natural gypsum (CaSO₄·2H₂O, by mass) to obtain a laboratory cement sample (LC, 380 m²/kg). Slag was also ground in a ball mill to a Blaine of 400 m²/kg. Each batch of slag powder was thoroughly mixed to obtain homogeneity. Reagent grade CaCO₃, which was ground to pass through an 80 μm sieve, was used to simulate the limestone substitute to avoid the impacts of impurities (such as clay). The TIPA used in this study was of commercial grade (effective concentration of ≥85%). If TIPA was added to laboratory cement in the grinding process, it would have altered the particle distribution of the cement due to its grinding effect. To study the pure chemical effect of TIPA, different TIPA additions (250, 500 and 1000 ppm) relative to the mass of cement or total cementitious material were added to the mixing water to prepare pastes or mortars (w/c: 0.5).

Approximately 6 g of paste with a w/c of 0.5 was prepared and loaded into an 8-channel TAM air isothermal calorimeter to determine the rate of heat generation during hydration in the first 72 h and the heat evolution for up to 7 d. Pastes were also produced with a w/c of 0.5 and stored at 20 °C in 50 ml sealed plastic vessels for thermal analysis and X-ray diffraction (XRD) analysis. The hydration of the paste was stopped by solvent exchange using ethanol. Then, the sample was dried in a vacuum drying oven at 40 °C for 4 h.

A powder sample of approximately 5 g was analyzed using XRD with a Thermo Fisher ARL 9900 series X-ray workstation with Co K α radiation ($\lambda = 0.1789$ nm) operated in reflection geometry ($\theta/2\theta$) at room temperature. The data were collected from 8° to 80° (2θ) over 120 min with a step size of 0.02°. The X-ray tube operated at 40 kV and 40 mA. The quantitative analysis was performed with the reference intensity ratio (RIR) method using Visual Crystal software supplied by Thermo Fisher.

Thermogravimetric analysis was performed on a Netzsch STA 449C by heating from 50 to 850 °C at 10 °C/min and purging with N₂. The amount of Ca(OH)₂ was calculated from the weight loss of a dry sample from 400 to 500 °C, and the carbonation was evaluated by the weight loss from 600 to 850 °C.

The pore solution was obtained using the expression method. Cylinders of the laboratory cement paste with a w/c of 0.5 were cast with different TIPA additions and were then stored in a humidity chamber (20 °C) until different ages. Thereafter,

Table 1
Analyses of clinker and slag (wt.%).

Oxide	Clinker	Slag
CaO	64.4	38.3
SiO ₂	21.4	34.4
Al ₂ O ₃	5.3	14.2
Fe ₂ O ₃	3.7	0.3
SO ₃	0.4	0.2
K ₂ O	0.1	0.6
Na ₂ O	0.1	0.3
Loss on ignition	0.1	-0.7
C ₃ S	58.9	
C ₂ S	16.7	
C ₃ A	7.8	
C ₄ AF	11.1	

they were demolded and inserted into the expression apparatus, upon which the pressures were applied in the compression test machine. Immediately after the extraction, the solution was filtered through quantitative filter paper and diluted with HNO₃ (6.5%) to prevent the precipitation of solid phases. The concentrations of general metal ions were determined using ICP-AES (PE Optima 2000DV).

Design of experiments (DoE) was used to investigate the influence of TIPA on the strength development of the ternary blend (LC–slag–CaCO₃), and the mixture-factorial model was selected. The strength of the ternary blend was determined using a mortar (4 × 4 × 16 cm³, w/c = 0.5), in which the cementitious–sand–water proportion was 1/3/0.5. Each of the components varied over a given range, which was characterized by three levels (low, intermediate and high). Different concentrations of TIPA were also diluted in the mixing water to prepare the mortars, as shown in Table 2.

3. Results

3.1. Influence of TIPA on the strength development of ternary blends

The strengths of mortars prepared from ternary blends were investigated using a mixture-factorial design, and 14 batches of mortar were prepared (Table 3). The strength results at different curing ages are illustrated by the ternary plot in Fig. 1.

In the absence of TIPA, the 3 d strength of the ternary blend considerably decreased with slag or CaCO₃ replacement (0–25%). The strength development of mortars at 7 and 28 d were similar, in which the lowest strength regions were near the high level of CaCO₃ addition. Because of the latent hydraulic reactivity, the slag provided higher strength than CaCO₃ at the same replacement level. In the presence of TIPA, the strength gains of the ternary blends were considerably higher at all curing ages. Additionally, the very strong interaction effect between slag and CaCO₃ contributed to the strength development prior to 7 d. Although the addition of CaCO₃ resulted in a lower 28 d strength than that provided by the slag, the rate of strength development by TIPA in the LC–CaCO₃ combination was more pronounced than that in the LC–slag combination. For example, the strengths of the blank 75% LC–25% slag combination were 24.0, 35.2 and 52.8 MPa at 3, 7 and 28 d, respectively. The addition of 500 ppm TIPA enhanced the strength development by 5.4%, 9.1% and 13.4%. Furthermore, the TIPA addition increased the strength of the 75% LC–25% CaCO₃ combination by 30.9%, 17.2% and 29.0%, respectively.

3.2. Influence of TIPA on the hydration of laboratory cement

3.2.1. Calorimeter test

The early hydration of LC was monitored by a calorimeter, and three main exothermic peaks appear in the first 72 h, as shown in Fig. 2, which correspond to I: C₃S hydration, II: renewed AFt formation, and III: AFt to AFm conversion [16,17].

The hydration of C₃S was slightly delayed by the increased TIPA addition, which was confirmed by the right-shifted peak I (see Fig. 2). The characteristics of peak II (also called the shoulder peak), which corresponds to the secondary AFt formation in different pastes, were pronounced with different TIPA additions. This

Table 2
Symbols and coded and actual values of the chemicals and the composition of blended cements.

Material	Low level		Intermediate level		High level	
	Coded	Actual	Coded	Actual	Coded	Actual
LC*	-1	75%	0	87.5%	1	100%
Slag*		0%		12.5%		25%
CaCO ₃ *		0%		12.5%		25%
TIPA [☆]		0 ppm		250 ppm		500 ppm

★: mixture component; ☆: continuous factor

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