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### The influence of ethanol-diisopropanolamine on the hydration and mechanical properties of Portland cement



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#### HIGHLIGHTS

• EDIPA accelerates the hydration of aluminate and ferrite phase, promoting hydration heat release rate.

• EDIPA accelerates the hydration of alite and the formation of CH.

• AFt is converted to AFm with EDIPA present.

• When EDIPA introduced, the mechanical properties of mortars enhances for 28 d.

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#### ABSTRACT

The effect of ethanol-diisopropanolamine (EDIPA) on the hydration and mechanical properties of Portland cement was focused on in this study. A combination of isothermal calorimetry, X-ray powder diffraction (XRD) and differential scanning calorimetry (DSC)-thermogravimetric (TG) analysis was used to investigate the hydration kinetics of Portland cement with EDIPA. Strength tests were conducted to study the effect of EDIPA on Portland cement mortars. The results indicate that EDIPA hinders the formation of ettringite (AFt), decelerates the dissolution of gypsum, and accelerates the hydration rate of the aluminate and ferrite phase and the transformation of AFt to AFm. Additionally, EDIPA also accelerates the hydration of alite and the formation of portlandite (CH), and remarkably enhances the mechanical properties at 28 d. Such strength-enhancing mechanism is probably similar to TIPA.

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#### 1. Introduction

Portland cement has become one of the most important and irreplaceable three traditional building materials in human society, since Joseph Aspdin has obtained the patent for Portland cement in 1824 [1]. Chemical additives are nowadays commonly used to improve the mechanical properties and workability of cement concrete in practice, and are becoming indispensable components in cement industry for energy savings and emission reductions.

Ethanolamine is a type of important chemical additive which is widely utilized as cement grinding agents and cement accelerators. Typical ternary ethanolamines mainly include triethanolamine (TEA), triisopropanolamine (TIPA), diethanol-isopropanolamine (DEIPA) and ethanol-diisopropanolamine (EDIPA). Figs. 1 and 2 show 3D conformer and schematic representation of the chemical structure of ternary ethanolamines. There have been many studies

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on TEA and TIPA. Ramachandran [2,3] showed that TEA could accelerate the formation of ettringite in pure  $C_3A$ -gypsum- $H_2O$  system, increase the induction period and promote the formation of C-S-H gel with higher CaO/SiO<sub>2</sub> ratio and non-crystalline CH in  $C_3S$ - $H_2O$  system. Neubauer et al. [4] also found that TEA could accelerate the hydration of  $C_3A$  and enhance the early compressive strength. Gartner et al. [5] found that the effect of TIPA on the compressive strength at 28 d was dependent on the  $C_4AF$  content in cement and proposed enhancing iron transport as the mechanism of strength enhancement. TEA is an Al and Fe chelating agent and is adsorbed on the surface of CH due to its low steric hindrance [6,7]. However, TIPA has a higher steric hindrance than TEA in Fig. 1, and thus enhance the later compressive strength due to the acceleration of the  $C_4AF$  hydration.

DEIPA and EDIPA are new kinds of alkanolamines. Their molecular structure, which is composed of both hydroxyethyl and hydroxypropyl, is different from TEA and TIPA from Fig. 1. Present investigations have shown that TEA and TIPA are able to dissolve a large amount of aluminate and ferrite phase due to complexation in fresh cement paste, respectively. TEA increases the early

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Fig. 2. Schematic representation of chemical structure of temary ethanolamines.

compressive strength. TIPA improves only the late strength significantly [8]. Hence, the effects of new alkanolamines on the cement hydration process need to be investigated. There have been a few studies on DEIPA, but very few on EDIPA. Riding [9] observed that DEIPA increased the early age mortar strength and affected the morphology of CH. Ma [10] also observed that DEIPA promoted the formation of ettringite and microcrystalline CH at early stages. Nevertheless, the chemical structure of EDIPA is slightly different from other ternary ethanolamines as seen in Fig. 1, and the effect of EDIPA on the Portland cement hydration kinetics and the mechanical properties is also unknown. In this research, the effect of various dosages of EDIPA addition on the Portland cement hydration kinetics and the mechanical properties were studied.

#### 2. Experimental

#### 2.1. Materials

Portland cement 42.5 complying with the Chinese standard GB175-2007 was used, whose chemical and mineral compositions are shown in Table 1. The contents of oxides were measured by X-ray fluorescence. The Bogue method was used to analyze the mineral phases. The fineness of Portland cement according to GB/T1345-2005 is 1.8% and the density is  $3.15 \text{ g} \cdot \text{cm}^{-3}$  with a Blaine value of  $350 \text{ m}^2/\text{kg}$ . Research-grade EDIPA was added with a dosage by weight of cement of 0.01%, 0.02% and 0.03%, respectively. Deionized water was used as the mixing water in the paste experiments.

#### 2.2. Methodology

#### 2.2.1. Cement paste preparation

All of the experiments were performed on cement paste with a water to cement ratio (w/c) of 0.5 to study the effect of various

Table 1

Chemical and mineral composition of cement.

EDIPA dosages on the phase. Cement pastes were prepared by mixing cement and water using over a time span of 2 min into the mixer at 62 rpm. After a 10 s interval, mixing was resumed for an additional 2 min at 125 rpm. The fresh pastes were poured into the mold (20 mm by 20 mm by 20 mm). The molds were then covered with the plastic wrap and stored in a curing box with a relative humidity of 98% and a temperature of  $20 \pm 2$  °C. The hydration was stopped after 1 h, 3 h, 12 h, 24 h, 3 d and 28 d by submerging small pieces in anhydrous ethanol. The pieces were then dried in an oven at 40 °C for 1 d and grounded to pass through the sieve with 80 µm for XRD and DSC-TG experiments.

#### 2.2.2. Isothermal calorimetry

An eight channel TAM AIR isothermal calorimeter was used to investigate the hydration heat flow of Portland cement with and without EDIPA. The calorimeter was first regulated at a constant temperature of  $25 \pm 0.02$  °C and then equilibrated for 24 h. The hydration experiments were measured over a period of 72 h to examine the effect of EDIPA on the hydration kinetics of Portland cement.

#### 2.2.3. X-ray powder diffraction

XRD is a useful method for semi-quantifying the content of AFt, AFm and CH in hydration products. The phase development was investigated via a Bruker AXS D8-Advance diffractometer with Cu K $\alpha$  radiation generated at 35 kV and 45 mA. The powders were step scanned from 5° to 60° with a step size and time per step of 0.01° and 0.2 s, respectively.

#### 2.2.4. Differential scanning calorimetry-thermogravimetric analysis

Thermal analysis was monitored on a Mettler-1600HT instrument with a combined TG and DSC system. A portion of the above powder was heated from 25 °C to 1000 °C at a heating rate of 10 °C/min under Argon atmosphere.

Chemical composition (mass %)						Mineral composition (mass %)					
CaO	SiO <sub>2</sub>	$Al_2O_3$	$Fe_2O_3$	$SO_3$	Na <sub>2</sub> Oeq	f-CaO	LOI	C <sub>3</sub> S	$C_2S$	C <sub>3</sub> A	C <sub>4</sub> AF
65.32	21.98	4.73	3.69	2.42	0.56	0.90	2.94	57.51	20.31	6.32	11.19

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