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# Study on the hydration and microstructure of Portland cement containing diethanol-isopropanolamine

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ABSTRACT

Diethanol-isopropanolamine (DEIPA) is a tertiary alkanolamine used in the formulation of cement grinding-aid additives and concrete early-strength agents. In this research, isothermal calorimetry was used to study the hydration kinetics of Portland cement with DEIPA. A combination of X-ray powder diffraction (XRPD), scanning electron microscopy (SEM), differential scanning calorimetry (DSC)–thermogravimetric (TG) analysis and micro-Raman spectroscopy was used to investigate the phase development in the process of hydration. Mercury intrusion porosimetry was used to study the pore size distribution and porosity. The results indicate that DEIPA promotes the formation of ettringite (AFt) and enhances the second hydration rate of the aluminate and ferrite phases, the transformation of AFt into monosulfoaluminate (AFm) and the formation of microcrystalline portlandite (CH) at early stages. At later stages, DEIPA accelerates the hydration of alite and reduces the pore size and porosity.

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## 1. Introduction

Faced with global energy-saving and emission-reducing requirements, the cement industry is being confronted with extensive press coverage. To achieve energy savings and emission reductions for cement, in addition to improving equipment, one approach involves increasing the grindability of cement using a grinding agent, thus reducing the power consumption. Another approach is to improve the properties of cement by using additives. Hence, chemical additives are becoming increasingly popular in materials based on cement.

Ethanolamine is a type of chemical additive used primarily in cement grinding agents. Typical ethanolamine components include triethanolamine (TEA), triisopropanolamine (TIPA), methyldiethanolamine (MDEA) and diethanol-isopropanolamine (DEIPA). Several studies have been conducted on TEA and TIPA. It was reported [1] that TEA could significantly increase the early compressive strength, especially before 3 d, and reduce the later strength. However, TIPA could improve the later strength. Gartner [2,3] observed that the effect of TIPA on the compressive strength at 28 d was dependent on the $C_4AF$ content in cement. The compressive strength at 28 d could be increased by 10%–22.8% if the $C_4AF$ content was more than 7.6%. J. Wray and P. Sandberg [4] observed that TIPA could increase the compressive strength to 65 MPa at 28 d if the lime saturation factor was decreased from 0.98 to 0.96. With respect to mechanistic explanations, it has been reported [5–7] that TEA accelerates the dissolution of $C_3A$ and $C_3S$ to form hydration products (AFt, CH and C-S-H) in the early stages, which result in an increase in the compressive strength. However, with the ongoing hydration, TEA is adsorbed on the surface of CH due to its low steric hindrance and precipitates along with CH when the concentration of $Ca^{2+}$ reaches saturation [8,9]. TEA precipitation causes its concentration in solution to decrease, thus reducing its action at later stages. For TIPA [2,10], its concentration in solution does not decrease significantly due to its higher steric hindrance compared with TEA. TIPA can also form a complex with $Fe^{3+}$ and accelerate the hydration of $C_4AF$, even when the gypsum has been fully consumed. Consequently, TIPA can enhance the later compressive strength.

To date, there have been only a few studies on DEIPA. Riding [11] demonstrated the effect of 0.02% DEIPA on the early strength enhancement of blend cement systems. The aim of the research presented herein was to determine the effect of various dosages of DEIPA on the hydration kinetics and microstructure of Portland cement to increase our understanding of the function of DEIPA. It is hoped that this further understanding will lead to more efficient use of Portland cement, with energy-saving and emission-reducing benefits in manufacture and application.

## 2. Experimental details

### 2.1. Materials

The Portland cement used in this work was produced by intergrinding Portland cement clinker with 5.0% gypsum in a 500 mm × 500 mm ball mill. The chemical compositions of gypsum are shown in Table 1. The

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**Table 1**
Chemical compositions of gypsum measured by XRF.

| Chemical composition | CaO | MgO | $Fe_2O_3$ | $Al_2O_3$ | $SiO_2$ | $K_2O$ | $SO_3$ | $P_2O_5$ | $TiO_2$ | LOI |
|---|---|---|---|---|---|---|---|---|---|---|
| | 31.84 | 0.21 | 0.08 | 0.27 | 0.78 | 0.04 | 45.93 | 0.02 | 0.01 | 20.64 |

**Table 2**
The chemical and phase compositions of the clinker as determined by chemical analysis and Bogue calculation.

| Chemical composition | CaO | $SiO_2$ | $Al_2O_3$ | $Fe_2O_3$ | $SO_3$ | MgO | Loss |
|---|---|---|---|---|---|---|---|
| | 65.70 | 21.60 | 5.51 | 3.39 | 0.41 | 1.65 | 0.46 |
| Phase composition | $C_3S$ | $C_2S$ | $C_3A$ | $C_4AF$ | f-CaO | | |
| | 60.76 | 16.96 | 7.24 | 10.67 | 0.42 | | |

chemical and phase compositions are listed in Table 2 for the Portland cement clinker, and its PSD is shown in Fig. 1. Research-grade DEIPA was added using a dosage by weight of cement of 0.01%, 0.02%, 0.03%, 0.04%, 0.05% and 0.1%. Deionised water was used as the mixing water in the paste experiments.

### 2.2. Methodology

#### 2.2.1. Paste preparation

All of the experiments were performed on cement paste with a water-to-cement ratio of 0.5 to study the effect of various DEIPA dosages on the phase and microstructural developments. Cement pastes were prepared by mixing cement and water using a laboratory mixer for 2 min at 500 rpm. The fresh pastes were poured into plastic bottles. The bottles were sealed and then stored in a curing box with a humidity of 98% and a temperature of 20 °C. The hydration was stopped after 1 h, 2 h, 3 h, 6 h, 12 h, 24 h, 3 d and 28 d by submerging small pieces in alcohol. The pieces were stored in an oven at 40 °C for 1 d to dry. Some dried pieces were ground and all particles passed through a sieve with 80 um for the XRD, DSC–TG and micro-Raman spectroscopy experiments. The other pieces were retained for SEM and MIP experiments.

#### 2.2.2. Isothermal calorimetry

An 8-channel isothermal calorimeter (TAM Air from Thermometric AB, Sweden) operating at 20 °C was used to measure the hydration heat flow of cement with and without DEIPA. The hydration experiments were run for 3 d to examine the effect of DEIPA on the hydration kinetics of Portland cement.

#### 2.2.3. X-ray powder diffraction

The phase development was investigated on a Rigaku SmartLab 3000A diffractometer with CuKα radiation (λ = 0.154 nm). The X-ray tube was operated at 35 kV and 30 mA. The optics configuration includes a fixed divergence slit (1/2°) and a D/teX Ultra detector. The measurements were collected using a θ–θ reflection geometry. Data were collected from 10° to 70° in the continuous mode.

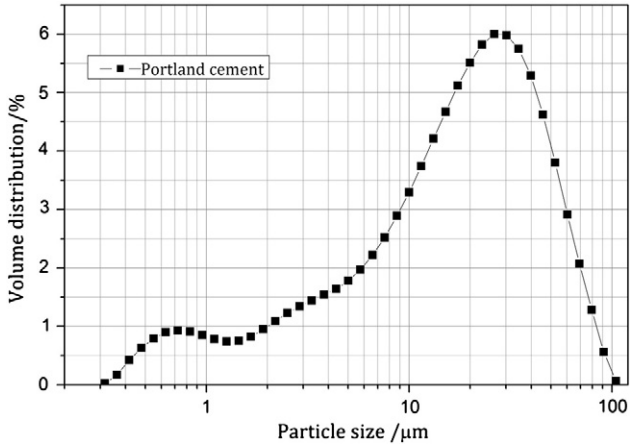


**Fig. 1.** The PSD curve for the Portland cement.

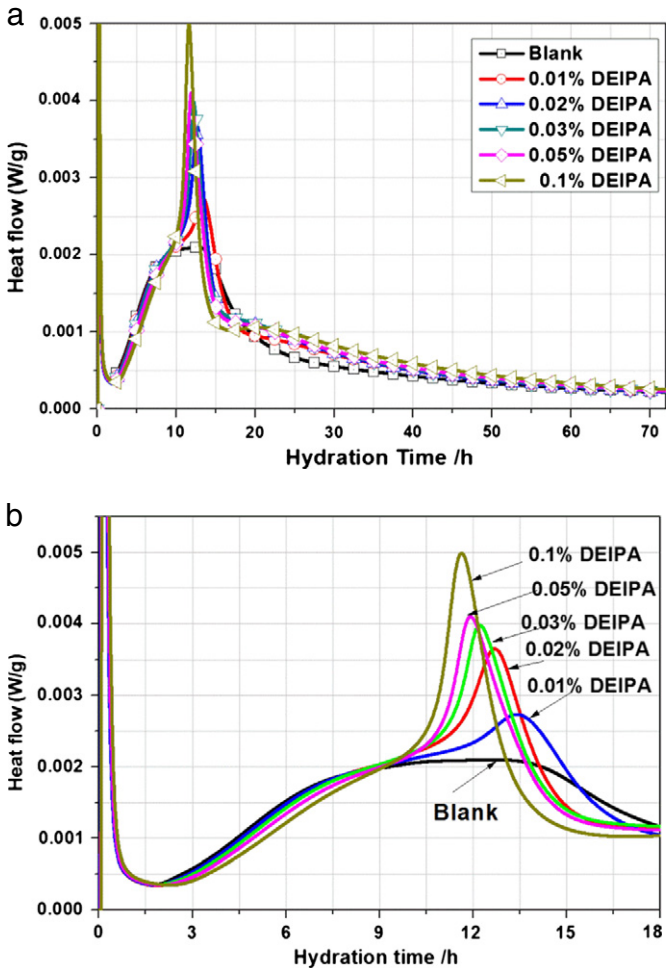


**Fig. 2.** Heat flow vs. hydration time for cement with and without DEIPA.

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

Thermal analysis was performed on a NETZSCH STA-449C instrument with a combined TG and DSC system. A portion of the above powder was heated from 30 °C to 1000 °C at a heating rate of 10 °C/min under $N_2$ atmosphere. The amounts of bound water were deduced from the weight loss between 30 °C and 400 °C. The amounts of CH were deduced from the weight loss between 400 °C and 500 °C.

#### 2.2.5. Micro-Raman spectroscopy analysis

Laser-Raman spectroscopy is complementary to IR spectroscopy, which can also be used to analyse cement minerals and hydration

**Table 3**
Cumulative heat of cement with and without DEIPA hydrated for different times.

| Hydration time/h | Dosages of DEIPA | | | | | |
|---|---|---|---|---|---|---|
| | Blank | 0.01% | 0.02% | 0.03% | 0.05% | 0.1% |
| 1 | 27.26 | 16.16 | 17.44 | 16.89 | 16.89 | 19.41 |
| 2 | 28.64 | 17.51 | 18.76 | 18.28 | 18.23 | 20.86 |
| 3 | 30.32 | 19.05 | 20.26 | 19.76 | 19.65 | 22.17 |
| 6 | 41.51 | 29.73 | 30.68 | 29.90 | 29.107 | 30.57 |
| 12 | 83.28 | 72.49 | 75.23 | 75.75 | 75.857 | 80.97 |
| 24 | 140.07 | 134.15 | 139.55 | 139.79 | 137.00 | 136.76 |
| 48 | 182.16 | 185.48 | 192.16 | 193.58 | 191.90 | 196.8 |
| 72 | 205.97 | 211.28 | 217.78 | 219.87 | 219.22 | 226.57 |

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