



# Effect of triethanolamine on cement hydration toward initial setting time



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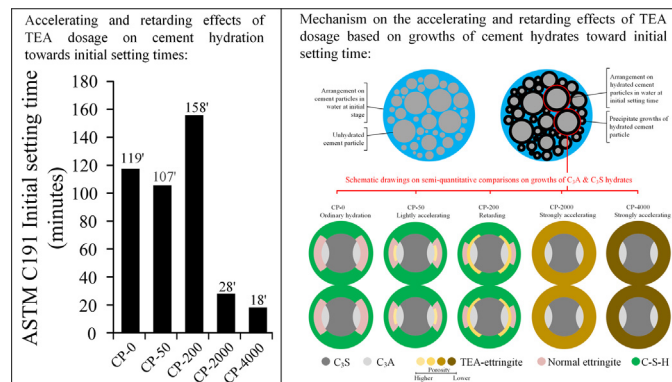
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## HIGHLIGHTS

- Experiments are conducted to examine the effect of TEA toward initial setting time of hydrated cement.
- Working principles of accelerated C<sub>3</sub>A hydration and ettringite formation are discussed.
- Accelerating-retarding effects of TEA on initial setting time of hydrated cement are examined.

## GRAPHICAL ABSTRACT



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## ABSTRACT

The present study investigates the working principle upon the accelerating-retarding effect of triethanolamine dosage on the initial setting time of hydrated cement. We have conducted atomistic simulation to probe the molecular interactions between triethanolamine and dissolved ions of hydrated cement and have performed experiment to examine the effect of triethanolamine upon cement hydration. It is found that the accelerating-retarding effect of triethanolamine on the initial setting time is caused by the different intensity of formed ettringite, which is governed by the triethanolamine dosage. This finding provides the piece of insight information on the roles of triethanolamine and ettringite formation upon the initial setting time.

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## 1. Introduction

In applications of cement based materials, initial setting time is the main parameter governing the operation schedule for transporting, placing, consolidating and finishing processes of concrete [1–3]. Initial setting time which is beyond the specification can

jeopardize the workability for proper placing and the progress of construction schedule. Control over the initial setting time starts from the cement production process. During this process, the sample of cement is taken frequently for finding the initial setting time. The standard test method ASTM C191 is adopted by most cement producing plants to determine the initial setting time. The most viable approach to control initial setting time is to incorporate chemical additive into cement during the comminution process. There are numerous selections in the market for the chemical

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additive that can be incorporated into cement based materials to influence setting time. The chemical additive can be grouped based on the typical applications, *i.e.*, normal [4], shotcrete [5,6] and cement comminution process [7,8]. Normally, those accelerators are used to shorten the setting time. In shotcrete and cement comminution process, the accelerator family is designed with additional features to enhance adhesiveness and cohesiveness (*e.g.* to reduce shotcrete rebound) and to improve the comminution efficiency respectively. The accelerator, which is alkali and chloride free, is preferable. Alkali can cause health issue and chloride induces corrosion in steel reinforcement [9,10]. We arrange the accelerator family with the chemical examples, as presented in Table 1 [11–13].

Triethanolamine (TEA) is the main chemical used as cement additive with multi functions as grinding aid during cement comminution process and setting time regulator for cement hydrations. This couple function is the main advantage from using TEA in addition to the relatively small dosage as well as the alkali and chloride free features. A unique characteristic of TEA is that it can either accelerate or retard cement hydration toward initial setting time depending on the dosage used [14–17]. TEA is respectively identified as light accelerator, retarder and strong accelerator in the typical magnitudes of dosages 200 ppm, 5000 ppm and 10,000 ppm [18–20].

Research studies have been performed to examine the effects of TEA on hydration of cement. Strong accelerating effect on initial setting time caused by relatively high dosages of TEA has been discussed and ascribed to ettringite formation during accelerated tricalcium aluminate ( $C_3A$ ,  $3CaO \cdot Al_2O_3$ ) hydration [20]. The studies involving dosages between 1000 ppm and 10,000 ppm show that TEA accelerates the hydration of  $C_3A$  as well as the formation of ettringite in the  $C_3A$ -gypsum-water system but retards  $C_3S$  hydration [21–23]. The presence of  $C_3A$ , either alone or along with gypsum, largely eliminate the retarding effect on  $C_3S$  hydration [23]. It means that TEA mainly influences  $C_3A$  during cement hydration in which  $C_3A$  and gypsum exist. Studies at molecular levels shows that TEA interacts with metal ions (*e.g.*  $Fe^{3+}$ ,  $Al^{3+}$  and  $Ca^{2+}$ ) in the pore solution of hydrated cement [24–26]. The chelating metal ions ( $Fe^{3+}$  and  $Al^{3+}$ ) by TEA possibly cause the accelerated  $C_3A$  hydration. TEA chelates calcium ions ( $Ca^{2+}$ ) and changes the morphology of formed portlandite. The chelation mechanism of amino alcohols such as TEA with metal ions is based on complexation reaction, wherein amino alcohols act as an electron pair donor [27,28]. It is noticed that TEA chelates different types of metal ions appearing at different stages of cement hydration. Apart from the understanding upon the chelating effects of TEA with metal ions dissolved from cement hydration, the working principle of TEA to control initial setting time is still not fully understood. More specifically, the working principle regarding light acceleration and retar-

ation effects of TEA dosages on initial setting time are still missing. This working principle can provide the essential information for optimizing the use of TEA to control initial setting time.

The aim of present study is to gain a comprehensive insight of TEA in regulating the initial setting time of hydrated cement. In particular, this study provides the working principle for the accelerating-retarding effects toward the initial setting time of cement hydration in the presence of TEA. Noteworthy, the found working principle provides the information on the technique to control the cement hydration toward the initial setting time.

The scope of the present study is the examination of cement hydration with respect to the effect of TEA with different dosages on initial setting time. Herein, we involve both experiments and atomistic simulation. On one hand, we firstly performed the tests to find accelerating and retarding effects of TEA dosages on initial setting time of hydrated cement. Subsequently, exothermic temperature of hydrated cement was monitored to examine the variation in cement hydration with different dosages of TEA. Next, examination on the change in dissolution and precipitation of hydrated cement with TEA was investigated through Fourier transform infrared (FTIR) and thermogravimetric analysis (TGA). On the other hand, atomistic simulation is conducted to probe the interactions between TEA molecules with dissolved ions of hydrated cement. Finally, based on the results from experiments and atomistic simulation, we discover the working principle for the effects of TEA dosages on the initial setting time.

## 2. Experiments

### 2.1. Materials

Materials used in this study were ASTM C150 type 1 cement, distilled water and TEA solution 85% produced by Horizon Chemical Company Limited. ASTM C204 fineness of cement is  $349 \text{ m}^2/\text{kg}$  as obtained from manufacturer specification and no additive was used. The cement used for all the tests was taken from the same commercial 45 kg bag to minimize the variation of cement. The content of calcium silicates is 77% for this cement with the main chemical composition is shown in Table 2.

### 2.2. Methods

Mixing of cement paste followed the procedure of ASTM C187. The composition of cement paste in all specimens was cement 650 g and water 160 g which met the normal consistency as defined in ASTM C187. TEA dosages of specimens were 50 ppm, 100 ppm, 200 ppm, 1000 ppm, 2000 ppm, 3000 ppm and 4000 ppm by weight of cement. A lower concentration in the dosage range (*i.e.* 50 ppm–4000 ppm) was initially chosen based on the reported dosage in literature for CGA and we simultaneously increased the dosage to cover the accelerating-retarding effects of cement on the initial setting [29]. In order to incorporate TEA into specimens, TEA solution 85% was first diluted into TEA solution 10% for more accurate weighing. Afterward, TEA solution 10% was put into the mixing water of each cement paste with its specified TEA dosage. The water in TEA solution was accounted to make the total amount of water in each specimen to be 160 g. Balance with accuracy  $\pm 0.01 \text{ g}$  was used to weigh TEA. This accuracy gave the maximum error 2 ppm of TEA dosage (in the range 50 ppm–4000 ppm) incorporated into the cement paste.

Test on initial setting time followed the procedure in ASTM C191. This test aimed to select the specimen that represented the hydrated cement with lightly accelerated, retarded and strongly accelerated initial setting time. Over the selected specimens, exothermic temperature, FTIR and TGA tests were performed.

**Table 1**  
Setting time accelerating chemical additive.

Typical application	Family	Examples of chemical	Dosage range (bwc)
Normal	Chloride Chloride free	Calcium chloride	~2%
		Calcium nitrate, lithium salts, calcium formate	0.5–6%
Shotcrete	Alkaline	Aluminate bases, sodium silicate	2–4%
	Alkaline-free	Aluminum sulfate	5–11%
Cement comminution	Cement grinding aid	TEA	Up to 0.5% (5000 ppm)

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