



Interaction effect of triisopropanolamine and glucose on the hydration of Portland cement



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HIGHLIGHTS

- The Glucose had an effect with TIPA that significantly promoted the hydration of paste after retardation.
- The heat generation of the paste with TIPA and glucose surpassed that of pastes with only TIPA or glucose.
- The addition of glucose promoted C_4AF dissolution by TIPA.
- The types of hydrates were influenced by TIPA or glucose.
- The hydrate products were characterised by a high surface area when the glucose was present.

ARTICLE INFO

Article history:

Received 9 January 2014

Received in revised form 1 April 2014

Accepted 4 April 2014

Available online 24 May 2014

Keywords:

Triisopropanolamine

Glucose

Interaction effect

Hydration

Cement

ABSTRACT

This study addresses the hydration mechanism of a P.II 52.5R cement in the presence of triisopropanolamine (TIPA), glucose or both. Calorimetry tests showed that the interaction effect of TIPA and glucose significantly enhanced the degree of hydration of the cement after 7 days. X-ray diffraction analysis confirmed that the addition of glucose promoted C_4AF dissolution by TIPA. The accelerated C_4AF hydration yielded a reduced CH content and an increased amount of chemically combined water. Due to the delayed acceleration effect of glucose, the hydrate products were characterised by a high surface area. Furthermore, our results showed that the addition of TIPA promotes the formation of carboaluminate and the AFm phase when the limestone is in the presence of cement.

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1. Introduction

Chemical additives are introduced to Portland cement during manufacturing to improve the quality of the finished cement. Triisopropanolamine (TIPA), a tertiary alkanolamine invented in recent years, is widely used as a grinding chemical in the clinker comminution process to reduce agglomeration in the ball mill and to modify the particle distribution of the finished cement [1,2]. TIPA also enhances the mechanical properties of cement. According to various studies, the addition of TIPA to Portland cement significantly improves strength development after 7 days [3–5]. Gartner proposed that TIPA is a facilitated transporter that chelates Fe^{3+} from the hydration product of ferrite and then releases it into the aqueous phase, increasing the dissolution of ferrite and promoting the overall silicate reaction [6]. An organic retarder, such

as sugar, has been used as the concrete chemical admixture to control the set and to improve the rheological properties of the fresh concrete mix [7,8]. Young attributed the retarding effect of sugar to adsorption to both hydrating particles and hydration products [9]. However, different investigations have yielded conflicting experimental evidence regarding the site of adsorption during the hydration process [10,11]. Glucose, a typical monosaccharide, is the basic building block of sugar and other polysaccharides. The retarding effect of glucose can also be explained by an adsorption mechanism. Currently, glucose is mainly used as a set retarder.

In a recent publication by Huang et al., the authors employed response surface methodology and found an interaction effect between triisopropanolamine and a monosaccharide that strongly contributed to the strength development of cement at 28 days [4,13]. However, there was no interaction between monosaccharide and triethanolamine (TEA), which is another popular alkanolamine used as a cement grinding aid [1,14]. In this work, glucose was selected as the monosaccharide in order to investigate its

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potential interaction effect with TIPA on the hydration of Portland cement.

2. Materials and testing methods

A P.II 52.5R Portland cement (Coded as 525 cement, composed of 90% clinker, 5% natural gypsum and 5% limestone) was used. It was analysed by X-ray diffraction (XRD) and X-ray fluorescence (XRF) methods. The XRD pattern and other information for the cement are shown in Fig. 1 and Table 1, respectively. The TIPA used in this study was commercial grade (effective concentration $\geq 85\%$). TEA was also used for a performance comparison in the calorimetry test. Glucose syrup was obtained from a local sugar refinery. The FTIR spectrum of the glucose syrup is presented in Fig. 2. The broad band at $3600\text{--}3200\text{ cm}^{-1}$ is attributed to O–H, and the O–H in-plane bending vibration is observed in the $1440\text{--}1260\text{ cm}^{-1}$ band. The band at $2900\text{--}2840\text{ cm}^{-1}$ is due to the symmetrical CH stretching vibrations in $-\text{CH}_2$, the band observed at $1660\text{--}1600\text{ cm}^{-1}$ is due to C=O stretching vibrations, and the band near 1000 cm^{-1} is attributed to a C–OH stretching vibration. All chemicals were diluted in the mixing water for the paste preparation.

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 days. Pastes were also made with a w/c of 0.5 and stored at $20\text{ }^\circ\text{C}$ in 50 ml sealed plastic vessels for thermal analysis (TG–DSC and ignition method) and X-ray diffraction (XRD). The hydration of the paste was stopped by solvent exchange using ethanol. Then, the sample was dried in a vacuum drying oven at a temperature of $40\text{ }^\circ\text{C}$ for 4 h.

An approximately 5 g powder sample was analysed by XRD using a ThermoFisher ARL 9900 series X-ray workstation with Co K α radiation ($\lambda = 0.1789\text{ nm}$) operated in reflection geometry ($\theta/2\theta$) at room temperature. The analysis of the pure clinker is shown in Table 1. The data were collected from 8° to 80° (2θ) over 120 min with a 0.02° step size. The X-ray tube was operated at 40 kV and 40 mA.

Thermogravimetric analysis was performed on a NETZSCH STA 449C by heating from 50 to $850\text{ }^\circ\text{C}$ at $10\text{ }^\circ\text{C}/\text{min}$ and purging with N_2 . The amount of $\text{Ca}(\text{OH})_2$ was calculated from the weight loss of a dry sample from 400 to $500\text{ }^\circ\text{C}$. The chemically combined water in the hydrated paste was determined by the ignition method. The sample was dried in a vacuum drying oven at a temperature of $105\text{ }^\circ\text{C}$ for 2 h to remove the bound water. Then, the dried sample was loaded into a muffle furnace and calcined at $1000\text{ }^\circ\text{C}$ for 0.5 h. The amount of chemically combined water was calculated from the weight loss of the sample from 105 to $1000\text{ }^\circ\text{C}$.

3. Results

3.1. Heat evolution

To clarify the interaction of TIPA and glucose, another typical alkanolamine, triethanolamine (TEA), was introduced for comparison with the effect of TIPA on the heat evolution of the cement. The hydration heats of the pastes with different chemicals (Table 2) was monitored by calorimetry, and the rate of heat generation during hydration in the first 72 h and the cumulative heat evolution for up to 14 days are shown in Figs. 3 and 4.

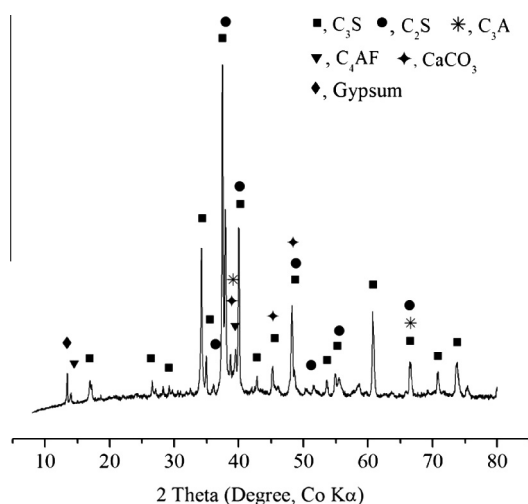


Fig. 1. XRD pattern of 525 cement.

Table 1
Analysis of 525 cement.

Oxide	wt.%	Mineral	wt.%
CaO	63.89	C ₃ S	64.60
SiO ₂	19.80	C ₂ S	7.99
Al ₂ O ₃	4.43	C ₃ A	6.53
Fe ₂ O ₃	3.08	C ₄ AF	9.36
SO ₃	3.77		
K ₂ O	0.067		
Na ₂ O	0.095		
Ignition loss	2.18		

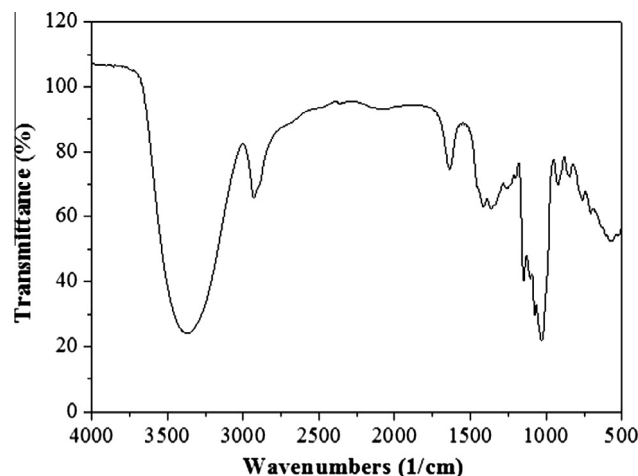


Fig. 2. FTIR spectra of glucose syrup.

Table 2
Symbols and compositions of the samples.

Sample	Composition
1	100% 525 Cement
2	100% 525 Cement + 500 ppm TEA
3	100% 525 Cement + 500 ppm TIPA
4	100% 525 Cement + 500 ppm glucose
5	100% 525 Cement + 500 ppm TEA + 500 ppm glucose
6	100% 525 Cement + 500 ppm TIPA + 500 ppm glucose

The addition of glucose to the pastes significantly delayed the formation of the first main heat releasing peak by approximately 4–5 h, which is associated with the hydration of C₃S [15]. Additionally, the delayed peak was stronger than the peak in the paste without glucose, which suggests that glucose promoted the hydration of C₃S after the retardation. The heights and starting times of the peaks in the pastes with or without TEA were almost identical, which indicates that the addition of TEA did not affect the hydration of C₃S. However, the addition of TEA contributed to the formation of a heat-releasing peak at 12 h, which is in agreement with renewed Aft formation and indicates that the TEA–aluminate interaction occurred during the early hydration period (Fig. 3a) [15]. In the paste with both TEA and glucose, no peak occurred after the first heat-releasing peak, indicating that the addition of glucose inhibited the renewed aluminate reaction. After hydration for 36 h, the rates of hydration in the different pastes were not significantly different from each other. Fig. 3b shows that the addition of TIPA did not affect the hydration of C₃S, as did TEA. However, TIPA promoted the formation of a shoulder peak at 24 h. The formation of the shoulder peak is associated with the conversion of Aft to AFm [15], which indicates that TIPA accelerated the sulphate consumption. In the paste with both TIPA and glucose, the C₃S

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