Construction and Building Materials 125 (2016) 210–218

Contents lists available at ScienceDirect

Construction and Building Materials

journal homepage: www.elsevier.com/locate/conbuildmat

Effects of triisopropanol amine, sodium chloride and limestone on the compressive strength and hydration of Portland cement

Zhenguo Shi [a,b], Caijun Shi [a,*], Hui Liu [a], Pingliang Li [a]

[a] College of Civil Engineering, Hunan University, Changsha 410082, PR China
[b] Interdisciplinary Nanoscience Center (iNANO), Aarhus University, Aarhus C 8000, Denmark

HIGHLIGHTS

- The combination effect of TIPA and NaCl on compressive strength and hydration is evaluated for Portland cement and Portland–limestone blended cement.
- Design of Experiment (DoE) was used to evaluate the interactive effect between TIPA, NaCl and limestone.
- No interactive effect was observed between TIPA and NaCl with respect to the strength enhancement.
- Hydration data showed clear interaction between TIPA and NaCl.

ARTICLE INFO

Article history:
Received 29 April 2016
Received in revised form 8 July 2016
Accepted 10 August 2016

Keywords:
Triisopropanol amine
Sodium chloride
Limestone
Compressive strength
Hydration
Design of Experiment
Isothermal calorimetry

ABSTRACT

The effect of triisopropanol amine (TIPA), sodium chloride (NaCl) and limestone (LS) on compressive strength and hydration of Portland cement were studied using Design of Experiment (DoE) and isothermal calorimetry, respectively. The results showed that combination of TIPA and NaCl could increase both early age and later age compressive strength of Portland cement mortars and Portland cement–limestone mortars. No major interactive effects on strength enhancement were observed for these three additives. However, the results of isothermal calorimetry showed clear interaction between TIPA and NaCl. It was found that NaCl increased the effectiveness of TIPA through promotion of two transitions: (i) conversion of AFt to AFm for Portland cement sample, (ii) conversion of carboalumiante phases for Portland–limestone cement sample.

1. Introduction

Chemical admixtures were increasingly employed in manufacture of Portland cement and concrete during the past several decades [1]. It has been developed being a plausible manner to improve both the quality of cement and performance of fresh and finished concrete. Nowadays, concrete with high and even ultra-high compressive strength can be developed to meet the industrial requirement [2]. This reflects the increased research needs for improving the early age compressive strength of concrete for the present expanding civil infrastructure.

Various admixtures have been used to produce high early strength concrete [3]. Triisopropanol amine (TIPA, $C_9H_{21}NO_3$), a well-known effective grinding aid, has also been used as a concrete admixture to accelerate the hydration of cement. An increased compressive strength by adding TIPA has been documented in many published studies [4–9]. Several researchers have proposed that the effect of TIPA on the compressive strength was dependent of the C_4AF content in cement [4,10,11].

Another type of admixture, which has been used for acceleration of hydration, is sodium chloride. Because chloride brings potential corrosion to the steel reinforcement in concrete [12], American Concrete Institute (ACI) has established some practical limits for chloride ions in concrete based on service conditions (ACI 222R; ACI 318/318R). Chinese national code strictly regulates the chloride content in cement should be less than 0.1% for reinforced concrete under moist condition (GB50164-92). However, chloride ions are known not harmful to concrete without steel reinforcement, and the price is much lower compared to most of the other organic additives. Moreover, the dosages of NaCl used for enhancement in compressive strength are usually lower than

* Corresponding author.
E-mail addresses: zshi@chem.au.dk (Z. Shi), cshi@hnu.edu.cn (C. Shi).

http://dx.doi.org/10.1016/j.conbuildmat.2016.08.030
0950-0618/© 2016 Published by Elsevier Ltd.

that specified in the standards. Therefore, use of chloride salt in concrete is still valuable for many field applications. In cementing oil wells, NaCl markedly accelerate cement hydration at lower dosages and retard the hydration at higher dosages [13]. Similar observations were also reported in recent published study [14].

Limestone as a mineral additive has been increasingly used in Portland cement. It is known that addition of small amounts of limestone in Portland cements can increase the early strength, reduces the water demand and improves the rheology of the resulting concrete [15–18]. Furthermore, limestone provides nucleation sites for the formation and growth of the calcium silicate hydrate phase and it is also partially consumed during hydration, resulting in the formation of calcium monocarboaluminate hydrate ($Ca_4Al_2(OH)_{12}CO_3{\cdot}5H_2O$) [16,18,19]. Although minor fraction of limestone is sufficient in Portland cement to stabilize ettringite through formation of monocarbonate, and the rest mainly play a dilution effect, Portland cement partially replaced by limestone with high replacement level (up to 20%) is sometimes still accepted as specified in standards in order to reduce the environmental impact as reviewed in [20,21].

Based on the previous investigations, it is possible to improve the compressive strength of concrete both for earlier ages (up to 3 days) and relatively later ages (up to 28 days or more) when TIPA and NaCl are used together in Portland cement and Portland–limestone cement blends. The impact of these additives on the strength and hydration of cement has been widely investigated individually. However, the interactive effect of these three additives on compressive strength and hydration has not been studied. The aim of this study is to evaluate the interactive effect of TIPA, NaCl and limestone on compressive strength and hydration. The Design of Experiment (DoE) was adopted to model the behaviors of dosed additives and to quantify their possible interactions. The influence of these factors on strength was well discussed by Pareto chart and contour plot. The influence on hydration was analyzed by isothermal calorimetry.

2. Experimental

2.1. Materials

The Portland cement (PC) used in this study was made by grinding 95 wt.% clinker together with 5 wt.% gypsum in a ball mill (MS500 type). Each operation of the grinding, 5 kg of materials was loaded to obtain the resulting cement with a high homogeneity. The mineral composition of the resulting cement contained 62.5 wt.% C_3S, 14.5 wt.% C_2S, 6.4 wt.% C_3A and 10.9 wt.% C_4AF according to Bogue calculation [22]. The cement had a specific surface area of 378 m^2/kg. The used mineral and chemical additives were Limestone (LS, including 96 wt.% of $CaCO_3$), tri-isopropanol amine (TIPA, with concentration of 85 wt.%) and analytical grade sodium chloride (NaCl). The LS was ground into powder to obtain a specific surface area of 492 m^2/kg. Since the present study focused on evaluation of the impact of TIPA, NaCl and LS on the compressive strength and hydration, these additives were not ground together with Portland cement in order to better control the proportions of the mixtures, especially that the dosage of TIPA and NaCl is so small. Intergrinding of these materials may increase the error of the measurements. Moreover, inter-grinding of TIPA with cement clinker may also decompose the molecules of TIPA, leading to reduction of the efficiency to strength enhancement. The chemical compositions for the P, gypsum and LS are given in Table 1.

The maximum dosages determined by experience and were 20 wt.% for LS, 0.08 wt.% for TIPA and 0.1 wt.% for NaCl. TIPA and NaCl were diluted and added into mixing water, respectively. The demineralized water used for the dilution of the chemicals was taken into account the mixing water for paste and mortar preparations.

Table 1
Chemical composition of raw materials (wt.%).

	SiO_2	Al_2O_3	Fe_2O_3	CaO	MgO	SO_3
Clinker	22.22	4.86	3.73	66.18	1.69	0.5
Gypsum	9.4	1.65	0.65	32.65	0.78	38.35
Limestone	1.66	0.55	0.06	53.76	1.08	

2.2. Sample preparation and testing methods

Three mortars for each batch were prepared with 450 ± 2 g cements including additives, 1350 ± 5 g of ISO standard sand and 225 ± 1 g mixing water. The TIPA and NaCl were pre-dissolved in part of the mixing water for a better dispersion. After casting, all the mortars were covered with a plastic membrane and cured in moist curing room at 20 ± 1 °C/95 ± 2% RH for 24 h. Afterward, they were demolded and submerged in water for further curing. The compressive strengths were determined on the hardened mortars after hydration for 1, 3, 7 and 28 days according to the Chinese standard GB/T17671-1999 [23].

The pastes with the proportion of additives as shown in Table 2 were prepared with the same water/cements ratio as for mortars. These samples were divided into two groups: PC blends without (P1-P4) and with (PL1-PL4) limestone. The designed proportion of additives allows evaluating the impact of individual and combination effects of TIPA and NaCl on hydration of the P and PL blends. The hydration process of the cement pastes was analyzed by TAM Air isothermal calorimetry (Thermometric AB, Sweden) at 20 °C. The test duration was 48 h. The changes in phases for PL4 sample after hydration for 12 and 42 h were studied using X-ray diffractometer (XRD, Bruker AXS D8 Advance, Germany) under the following operating conditions: 40 kV and 40 mA with Cu Kα_1 radiation (λ = 1.54187 Å) and acceptance slot at 0.1 mm. The hydration of PL4 was stopped by isopropanol prior to XRD analysis.

2.3. Design of Experiment (DoE)

DOE is a very useful statistical approach to plan the experimental arrangement in order to quantify the correlation of multivariable and responses. It has been successfully used by other researchers to evaluate the effect of other factors on properties of cement and concrete [6,24,25]. In this study, DoE was set up using central composite face-centered design (CCF). For predicting the optimal conditions, a second order polynomial function was fitted to correlate relationship between independent variables and response, which can be expressed as:

$$y = b_0 + \sum_i^k b_i x_i + \sum_i^k b_{ii} x_i^2 + \sum_{i<j} b_{ij} x_i x_j + e(x_1, x_2, \ldots, x_k) \quad (1)$$

Table 2
Paste samples from isothermal calorimetry analysis (wt.%).

Labels	TIPA	NaCl	LS
P1	0	0	0
P2	0.08	0	0
P3	0	0.1	0
P4	0.08	0.1	0
PL1	0	0	20
PL2	0.08	0	20
PL3	0	0.1	20
PL4	0.08	0.1	20