



Hydration kinetics and compressive strength of steam-cured cement pastes and mortars containing limestone filler



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HIGHLIGHTS

- The effect of limestone filler (LF) on hydration kinetics and strength was evaluated.
- LF increased the heat of hydration, $\text{Ca}(\text{OH})_2$ content and early age strength.
- Increasing LF fineness increased the early age hydration and strength.

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ABSTRACT

This paper aims to evaluate the influence of Limestone filler (LF) content and fineness on the hydration kinetics and strength of steam cured cement mortars and pastes. Experimental variables were cement fineness, LF content, LF fineness and steam curing duration. Mortar and paste specimens were steam cured at 55 °C for 12 and 16 h. Hydration kinetics were evaluated using heat of hydration and thermal analysis. The heat of hydration was measured using Isothermal Calorimetry. Calcium hydroxide ($\text{Ca}(\text{OH})_2$) content, calcium carbonate (CaCO_3) content and degree of hydration were measured using Thermal Gravimetric Analysis and Differential Thermal Analysis. Compressive strength of mortars was evaluated at 12 h, 16 h, 3 days, 7 days and 28 days. The results showed increase in the heat of hydration, $\text{Ca}(\text{OH})_2$ content and early age strength (at 12 and 16 h) with the addition of LF. The results also revealed that the influence of LF on hydration kinetics and strength was influenced by the finenesses of the LF and the cement. In general, mix designs containing LF showed improved early age hydration and strength in mix designs made with higher cement fineness. Increasing LF fineness or cement fineness can reduce the negative impact of cement dilution by LF addition on the mechanical properties of mortars.

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1. Introduction

Limestone filler (LF) is a fine powder produced from grinding calcitic limestone rocks obtained from quarries. LF can be utilized as a replacement of cement clinker to reduce energy consumption and reduce CO_2 emission associated with cement production [1,2]. The effects of LF on hydration kinetics and strength when cured at ambient temperature have been the focus of many research efforts [3–5]. The outcomes indicate that LF influences the cement system through physical and chemical effects. The physical effects are modification of particle size distribution, heterogeneous nucleation and dilution [6,7]. The chemical effect is the reaction of LF with calcium aluminate to form calcium mono-carboaluminate.

The addition of LF can improve the particle size distribution of the cement. In addition, LF particles fill the voids between coarser cement particles and thus increase the density and reduce the total pore volume of the cement system [8]. The surface of LF particles act as nucleation sites for the precipitation of the hydration products [9]. The nucleation sites provided by LF reduce the energy barrier and allow the hydration products to precipitate faster from the pore solution; this increases the rate of hydration reactions and early age strength gain which is described as the acceleration effect [10–12].

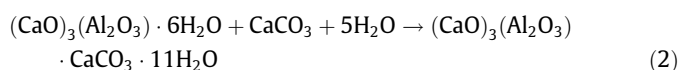
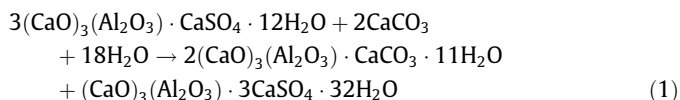
Unlike modification of particle size distribution or heterogeneous nucleation, dilution can have adverse effects on the strength and durability of concrete. Dilution occurs as a result of replacing reactive material such as cement by a nonreactive or relatively less reactive material such as LF [9]. The adverse effect of dilution is mainly due to the reduction in hydration products. When LF is used to replace more than 5% of cement, the dilution effect masks the other effects (i.e. modification of particle size distribution,

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heterogeneous nucleation and chemical reaction). Dilution decreases the strength of concrete at all ages. However, it is mainly observed at later age (i.e. after 3 days) [13]. This is due to the heterogeneous nucleation effect of LF that compensates for the dilution effect at early age (i.e. before 3 days).

There is a general agreement in the literature regarding the reactivity of LF (CaCO_3) with the monosulfate ($(\text{CaO})_3(\text{Al}_2\text{O}_3) \cdot \text{CaSO}_4 \cdot 12\text{H}_2\text{O}$) or calcium aluminate hydrate ($(\text{CaO})_3(\text{Al}_2\text{O}_3) \cdot 6\text{H}_2\text{O}$) to form calcium mono-carboaluminate ($3\text{CaO} \cdot \text{Al}_2\text{O}_3 \cdot \text{CaCO}_3 \cdot 11\text{H}_2\text{O}$) as presented in Eqs. (1) and (2) [14–16]. However, there is disagreement on the amount of the total available LF in the system that can be consumed in these reactions [17].



The above mentioned effects of LF on hydration rate and early age strength gain are agreed upon in the literature for curing at ambient temperature (i.e. 23 °C) [18–20]. This includes inter-ground limestone at replacement levels up to 5% in CSA type GU cement and at replacement levels up to 15% in Portland limestone cement (PLC). However, there is limited data in the literature on how LF influences the hydration rate and early age strength gain when concrete is steam cured [21,22]. The hydration reactions under steam curing conditions are accelerated due to the increase in the temperature. However, during the steam curing, the chemical reaction of LF is reduced due to that fact that the solubility of LF decreases with the increase in the temperature [23]. Therefore, the influence of LF under steam curing conditions is expected to be different compared to moist curing at ambient temperature.

The study of the interplay between cement type, LF content and LF fineness of steam cured cement based material is warranted. The impact of lower solubility of LF and rapid hydration rate on the behavior of LF under steam curing conditions is important to understand. This paper aims to contribute toward further understanding of LF behavior and effects when used as cement replacement under steam curing conditions. This was achieved by evaluating the effects of LF on hydration kinetics and compressive strength of steam cured cement mortars and pastes. In addition, the reactivity of LF under steam curing conditions was assessed. Two types of cements were selected namely: CSA general use (Type GU) cement and high early strength (Type HE) cement to represent the common cements used in the precast/prestressed concrete industry. Three LFs with different sizes (17 μm , 12 μm and 3 μm) were selected to cover a wide range of particle size distribution. The percentage of blended LF was 0, 5, 10 and 15% to evaluate the influence of LF at different cement replacement levels. The maximum steam curing temperature was set at 55 °C to prevent any alteration in the microstructure of cement mortars and pastes due to delayed ettringite formation [23]. The heat of hydration, phase composition using Thermal Gravimetric Analysis and Differential Thermal Analysis (TG/DTA) and compressive strength were investigated. Multiple linear regression analysis was conducted to identify the primary variables that control hydration kinetics and strength evolution from 12 h to 28 days.

2. Experimental program

2.1. Materials and mix design

Two types of cement were used, CSA Type general use (GU) and CSA Type high early strength (HE) cements, both supplied by Holcim Canada. The GU cement had a Blaine fineness of 392 m^2/kg and the HE cement had a Blaine fineness of 514 m^2/kg .

The GU cement had 2.5% interground limestone and the HE cement had 3.5% inter-ground limestone. Three LFs with different nominal particle sizes (17 μm , 12 μm and 3 μm which correspond to Blaine fineness of 475, 380 and 1125 m^2/kg , respectively) were supplied by Omya, Canada. The chemical and physical properties of cements, and the LF are presented in Tables 1 and 2, respectively. The fine aggregate was natural sand with a specific gravity of 2.72 and a fineness modulus of 2.84. The fine aggregate was supplied by Dufferin Aggregates.

Twenty mix designs were examined in this study. LF was used to replace 0–15% of cement by weight. The mix proportions are presented in Table 3. For each mix design, mortars and pastes were prepared. Mortar was used for the cube compressive strength evaluation and the corresponding paste was used to measure the heat of hydration, calcium hydroxide ($\text{Ca}(\text{OH})_2$) content, calcium carbonate (CaCO_3) content and degree of hydration. The water-to-cement ratio and sand-to-cement ratio were kept constant at 0.37 and 2, respectively. Cement, sand and LF (when used) were blended initially for 2 min in a 10 L mortar mixer. After the addition of water the materials were mixed for 4 min. Similar mixing procedures were followed for the corresponding paste mixes. For each mix design, 15 mortar cubes (50 × 50 × 50 mm) were prepared for compressive strength testing and paste was prepared for Isothermal Calorimetry and TG/DTA testing.

2.2. Curing regime

Steam curing was conducted in a 0.45 m^3 environmental chamber manufactured by Cincinnati Sub-Zero. After mixing, the specimens were cured at 23 °C and 98% relative humidity (RH) for 2 h (preset period) to ensure that steam curing was applied after initial setting [24]. The maximum holding temperature of steam curing was 55 °C with RH of 98%. A sample curing regime is presented in Fig. 1. After the preset period, the specimens were steam cured in the following sequence:

- heating to 55 °C in 2 h (16 °C/h) while maintaining 98%RH
- holding the temperature at 55 °C while maintaining 98%RH for 10 h for the 16-h curing regime or for 6 h for the 12-h curing regime
- cooling to 23 °C in 2 h (16 °C/h) while maintaining 98%RH (in the chamber)

The temperature of the chamber was controlled to maintain the required internal temperature of the samples, using Type T thermocouples embedded in the samples at the centroid. After 12 or 16 h, all specimens were moist cured in limewater at 23 °C until tested (i.e. 3, 7 and 28 days). Thermal analysis was conducted after 16 h of steam curing and after 28 days (16 h of steam curing followed by moist curing in limewater until 28 days). The selection of 16 h for thermal analysis was to reflect the common curing regimes used in the precast/prestressed industry in Canada [25].

2.3. Test methods

Initial Setting Time: The normal consistency and initial setting time were measured according to ASTM C187-11 and ASTM C191-08, respectively [26,27].

Heat of Hydration: For each mix design, three cement paste samples were tested for heat of hydration over a period of 72 h. The test was conducted at 23 °C in accordance with ASTM C1702-09 Method B [28]. A TAM Air isothermal calorimeter manufactured by Thermometric was used.

Thermal Analysis: The TG/DTA analysis was conducted to measure the $\text{Ca}(\text{OH})_2$, CaCO_3 and non-evaporable water contents in paste samples. For each mix design, two TG/DTA tests were conducted. Netzsch SA Simultaneous Thermal Analyzer heated to 1145 °C at a heating rate of 10 °C/min was used. The degree of hydration was calculated by dividing the mass loss between 23 °C and 550 °C by the maximum theoretical non-evaporable water (i.e. 0.23), as presented in Eq. (3) [29].

Table 1
Chemical and physical properties of cements.

Chemical and physical properties	Cement type	
	GU	HE
SiO_2 (%)	19.25	19.10
Al_2O_3 (%)	5.33	5.18
Fe_2O_3 (%)	2.41	2.35
CaO (%)	62.78	61.60
MgO (%)	2.36	2.35
SO_3 (%)	4.01	4.26
Na_2Oeq (%)	0.99	1.01
C_3S (%)	58.55	55.15
C_2S (%)	10.04	9.75
C_4AF (%)	7.34	7.14
C_3S (%)	11.03	13.18
LOI at 1150 °C (%)	2.27	2.10
Blaine (m^2/kg)	392	514
Limestone content (CaCO_3)	2.50	3.50

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