



Multi-scale investigation of the performance of limestone in concrete



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HIGHLIGHTS

- The acceleration ability of fine limestone in cements depends on the polymorph.
- Calcite provides favorable surfaces for nucleation and growth, aragonite does not.
- Thermally converting the aragonite to calcite restores its acceleration ability.
- The acceleration ability of limestone produces stronger limestone-based concretes.

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ABSTRACT

Limestone (calcium carbonate, CaCO₃) has long been a critical component of concrete, whether as the primary raw material for cement production, a fine powder added to the binder component, or a source of fine and/or coarse aggregate. This paper focuses on the latter two of these examples, providing a multi-scale investigation of the influences of both fine limestone powder and conventional limestone aggregates on concrete performance. Fine limestone powder in the form of calcite provides a favorable surface for the nucleation and growth of calcium silicate hydrate gel at early ages, accelerating and amplifying silicate hydration, and a source of carbonate ions to participate in reactions with the aluminate phases present in the cement (and fly ash). Conversely, the aragonite polymorph of CaCO₃ exhibits a different crystal (and surface) structure and therefore neither accelerates nor amplifies silicate hydration at a similar particle size/surface area. However, because these two forms of CaCO₃ have similar solubilities in water, the aragonite does contribute to an enhancement in the reactivity of the aluminate phases in the investigated systems, chiefly via carboaluminate formation. In 100% ordinary Portland cement (OPC) concretes, 10% of the OPC by volume can be replaced with an equivalent volume of limestone powder, while maintaining acceptable performance. A comparison between limestone and siliceous aggregates indicates that the former often provide higher measured compressive strengths at equivalent levels of hydration, even when the two aggregate types exhibit similar elastic moduli. This suggests that the interfacial transition zone in the limestone-based concretes exhibits a higher degree of bonding, likely due to the favorable physical (texture) and chemical nature of the limestone surfaces. These observations reinforce the value of utilizing limestone to increase the performance and sustainability of 21st century concrete construction.

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1. Introduction

The 21st century sustainability movement in North America has produced increased interest in replacing a portion of the ordinary Portland cement (OPC) in concrete with limestone powder, thus reducing both the CO₂ and energy footprints of the concrete. While

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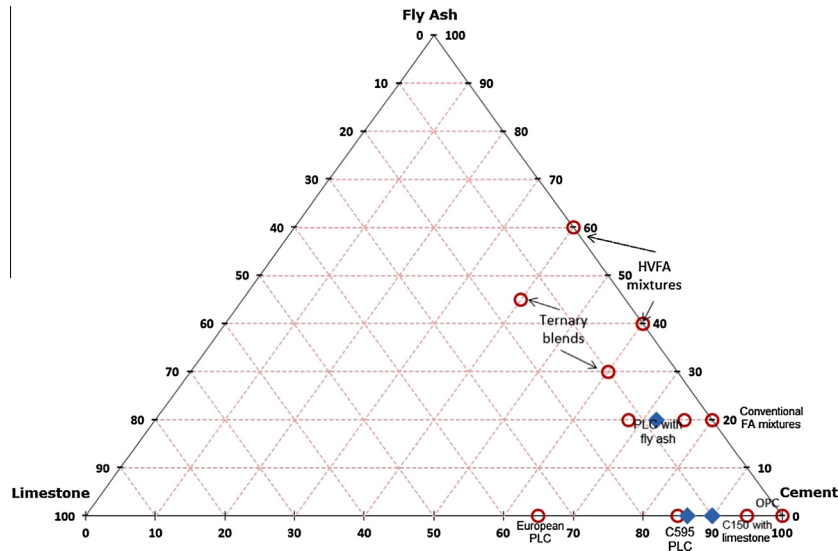


Fig. 1. Ternary plot indicating common mixtures of cement, fly ash, and limestone. Filled diamonds indicate the mass-based mixture proportions investigated in the paste portion of the present study.

Portland limestone cements (PLC) have been used in Europe for many years, it is only recently that U.S. standards have first permitted the incorporation of up to 5% (inter)ground limestone in ASTM C150 Portland cement [1] and subsequently introduced a new class of PLCs into ASTM C595 [2], the standard for blended cements, with the U.S. revisions following after the Canadian implementation in both cases. Type IL in ASTM C595 permits the incorporation of up to 15% limestone by mass in the blended cement. The performance equivalence of these PLC-IL cements with ASTM C150 cements has been recently documented in a series of technical articles [3–7]. As these new PLCs continue to establish market acceptance, a viable alternative to an interground blended cement is the direct addition of a limestone powder to an ASTM C150 cement at the ready-mix or pre-cast production plant, similar to the manner in which slag, fly ash (FA), or silica fume are often introduced by the concrete manufacturer. Since the limestone and cement are processed separately in this case, the particle characteristics (surface area, particle size) of each can be accurately characterized and controlled, and investigations of how these characteristics affect performance in cement-based materials can be conveniently performed [8–12]. These studies have revealed the importance of limestone powder surface area (fineness) in boosting early-age hydration and reducing initial and final setting times, particularly in ternary blends containing fly ash at conventional or high-volume addition rates. The viability of this approach is further reinforced by the ongoing development of a ground limestone (and mineral filler) proportioning guide and a materials specification within ACI and ASTM, respectively.

A variety of potential ternary mixtures of cement, fly ash, and limestone powder are represented in Fig. 1. Because the efficacy of fine limestone powders to improve the setting times and mechanical and transport properties of high-volume fly ash (HVFA) mixtures has been investigated extensively [10,11], the present study focuses on two other types of mixtures, as indicated by the three filled diamond data points in Fig. 1. The two data points on the bottom axis indicate mixtures in which 10% by mass of either a 100% Type I OPC (right point at cement = 90) or an ASTM C150 Type I/II with interground limestone cement (left point at cement \approx 87) is replaced by limestone powder. The third filled diamond data point corresponds to a mixture that contains 20% fly ash and 5% fine limestone powder, replacing 25% of an ASTM C150 Type III with interground limestone cement. In addition, for

a subset of these mixtures, both the surface area/particle size and the crystalline form (aragonite or calcite) of the limestone powder are investigated in studies on pastes. For the 10% limestone mixtures, concretes are prepared, this time with a volumetric replacement of limestone powder for cement, and compared to a 100% ASTM C150 Type I/II with interground limestone cement concrete mixture. Finally, this study of the influence of limestone on cement hydration and performance is extended to a larger length scale by considering the impact of aggregate type (limestone or siliceous) on concrete strength.

2. Materials and procedures

2.1. Cements

Characteristics of the three cements employed in the various parts of this study, as supplied from their manufacturers' mill sheets, are provided in Table 1. The cements consisted of an ASTM C150 Type III cement, a white Type I cement, and a Type I/II cement. The Type III and Type I/II cements, while both meeting ASTM C150 specifications [1], each contained a percentage of limestone powder added directly to the cement clinker prior to the grinding process (interground limestone) as indicated in Table 1. Particle size distributions, characterized by their particle size parameters D_{10} , D_{50} , and D_{90} (representing the 10th, 50th, and 90th percentile, respectively) in Table 1, were determined using laser diffraction equipment, with isopropanol as the dispersant. The particle size parameters in Table 1 are determined by averaging six separate scans, with a typical coefficient of variation being less than 1%.

2.2. Limestones

Two of the limestone powders employed in this study, including the finest material, were supplied by OMYA,¹ while the other two were obtained from Specialty Minerals (SM), including a precipitated calcium carbonate (PCC – denoted as Sturcal F) powder based on the aragonite polymorph of CaCO_3 , as opposed to calcite. In Table 2, limestone powder densities ($\pm 10 \text{ kg/m}^3$ standard deviation) were measured using a helium pycnometer and their BET (Brunauer, Emmett, and Teller [13]) surface areas (coefficient of variation of 2% for three replicate specimens [9]) were measured using nitrogen adsorption. A sample of the aragonite-based Sturcal F limestone powder was subsequently heat treated (HT) at $480 \text{ }^\circ\text{C} \pm 10 \text{ }^\circ\text{C}$ for 4 h to thermally convert the aragonite polymorph to calcite [14]. The converted powder was then evaluated both in a white cement mixture and in a cement/fly ash blend.

¹ Certain commercial products are identified in this paper to specify the materials used and the procedures employed. In no case does such identification imply endorsement or recommendation by the National Institute of Standards and Technology, the Federal Highway Administration, the National Research Council Canada, or Purdue University, nor does it indicate that the products are necessarily the best available for the purpose.

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