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# A methodology to extract the component size distributions in interground composite (limestone) cements



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### HIGHLIGHTS

• A novel method to extract the particle size distributions of the components in the multi-particle systems.

• Adopted to interground Portland-limestone cements.

• Explains the PSD dependence on performance.

#### ARTICLE INFO

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### 1. Introduction

The use of fine limestone as partial cement replacement is a topic of great interest since it improves the sustainability of portland cement-based concrete and provides comparable properties to that of conventional OPC concrete [1,2]. Two methodologies are commonly used to incorporate fine limestone in cementitious systems: (i) intergrinding the limestone with the portland cement clinker to produce Portland-limestone cements (PLCs), or (ii) blending OPC with limestone powder in the concrete production process. Intergrinding of portland cement clinker with limestone results in a finer limestone phase in the composite system due to the fact that limestone is a softer material than the clinker. Blending, on the other hand, would result in a limestone size distribution

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# ABSTRACT

This paper compares the performance of commercial interground Portland-limestone cements (PLC) to those of blended limestone systems. Limestone of four different median sizes is mixed with ordinary portland cement (OPC) to create blends in an attempt to match the particle size distribution of the PLCs. The interground systems are found to outperform the blended systems, plausibly because of the difference in size distributions of the clinker and limestone fractions between the PLCs and the blended systems. A novel methodology to extract the particle size distributions of the components in the interground systems is reported. This method, applicable for several types of multi-component powder systems, considers Rosin-Rammler size distributions for the ground clinker and limestone, and optimizes the parameters of the distribution to obtain a composite distribution of the same fineness as the interground system. The model is verified using a cement hydration and microstructure model.

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that can be controlled more directly by the cement or concrete producer. ASTM C595 cements, along with cements conforming to CSA A 3000 (Canada), AASHTO M 240 and EN 197-1 (Europe) among others are permitted to contain 5–15% (by mass) of fine limestone. In the U.S., cements conforming to ASTM C1157 (a performance based cement specification, as compared to ASTM C595 and C150 which prescribe limits on chemical composition) have also been manufactured with 5–15% of limestone.

Several studies have investigated the influence of fine limestone as a partial cement replacement material, blended or interground with OPC [3,4]. Limestone has also been used in conjunction with other cement replacement materials such as fly ash, slag, or metakaolin [5–9]. The early age hydration response, reaction product formation, rheology, strength development, and durability of the binary and ternary systems containing limestone have been reported in detail. A number of studies have focused on the use of limestone which is finer than the cement it replaces, as this has been shown to accelerate hydration reactions [7,10], increase the packing of particles [11], and minimize the strength loss associated with limestone [7]. The formation of carboaluminates in limestone bearing systems that reduces the overall pore volume has been reported in [1,12]. A number of studies on the properties of interground limestone cements have also been reported [13–15].

It is well known that the particle size distribution (PSD) of the cement (OPC or PLC) influences the early-age hydration, reaction product formation and consequently the performance [16,17]. This paper evaluates whether property equivalence between interground PLCs and OPC-limestone blends can be attained, provided the blend is designed to have a similar specific surface area as that of the PLC and is constituted using the same OPC clinker that made up the PLC. This is of significance for limestone suppliers interested in supplying limestone to concrete producers that blend limestone with the cements they use, because it is the ASTM C150 or equivalent cement that generally is used for general concrete construction. Furthermore, a methodology to extract the PSDs of cement and limestone in an interground PLC is proposed, which will help to choose the parent OPC and limestone particle sizes that permit property equivalence. This methodology is generic enough so as to be implemented to extract the size classes of different particles in a wide array of powder mixtures such as in pharmaceutical, chemical. and food industries.

#### 2. Experimental program

#### 2.1. Materials

The materials used in this study are a commercially available Type I/II ordinary portland cement (OPC) conforming to ASTM C150 [18], nominally pure limestone powders (>95% CaCO<sub>3</sub>, by mass) of four different median particle sizes (0.7  $\mu$ m, 3  $\mu$ m, 10  $\mu$ m, and 15  $\mu$ m), and two different commercially available interground PLCs conforming to ASTM C595 [19] and ASTM C1157 [20]. Fig. 1 shows the particle size distribution of these raw materials, and Table 1 presents their respective

chemical compositions (provided by the manufacturer) and Blaine's fineness. Both the interground cements are finer than the parent OPC (denoted as C150), with the C1157 cement being markedly finer. It is noted that the major difference between these C595 and C1157 cements is in their fineness alone since they are constituted from the same clinker used for the parent C150 cement.

#### 2.2. Experiments

#### 2.2.1. Compressive strength

Compressive strength tests were carried out in accordance with ASTM C109 [21]. The mortar cubes were prepared with a sand volume fraction of 50%. The testing was carried out at ages of 1, 3, 7, 28 and 56 days.

#### 2.2.2. Isothermal calorimetry

The heat evolution from the hydration of cement was determined using isothermal calorimetry (TAM Air microcalorimeter<sup>™</sup> 2700 Series) at a constant temperature of 25 °C for 72 h. The powders were dry blended prior to adding water. To ensure accurate early age measurements and minimize the time required to attain isothermal conditions, the blended powders and water were conditioned in an oven for 12 h at 25 °C. The pastes were then mixed in accordance with ASTM C305 [22]. Approximately 10 g of sample was extracted immediately, placed in a sealed sample vial to minimize evaporation and placed in the calorimeter. Three samples per mixture were tested and average values are reported.

#### 2.2.3. Mercury intrusion porosimetry

Paste samples of approximately 100 g were cured for 3 and 28 days under sealed conditions at a constant temperature of  $23 \pm 1$  °C. At the desired age of testing, the samples were crushed to an approximate size of 2 mm, and then pretreated in an oven for 2 h at 60 °C, as this method was found to produce consistent results in a previous study [23]. Approximately 1 g of material was selected and weighed using a high precision scale. Testing was completed using a 0.5 cc cell in a Quantachrome Instruments PoreMaster<sup>™</sup> mercury intrusion porosimeter to a maximum testing pressure of approximately 410 MPa. The porosity of the sample volume. The relationship between pore diameter and intrusion pressure was determined using the Washburn equation. The surface tension of mercury and the mercury contact angle were assumed to be 0.480 N/m and 117° respectively during distribution frough mercury intrusion have been detailed in [26,27]. Thus, to



Fig. 1. Particle size distributions of: (a) three different cements, and (b) the limestone powders used in this study.

Table 1			
Chemical composition and	fineness of the pare	ent cements used in	n this study

Cement type (ASTM)	Chemical composition (% by mass)						Blaine's Fineness		
	SiO <sub>2</sub>	$Al_2O_3$	$Fe_2O_3$	CaO	MgO	SO <sub>3</sub>	Loss on ignition	Limestone	$(m^2/kg)$
C150 Type I/II	19.60	4.09	3.39	63.2	3.37	3.17	3.17	-	452
C595 Type IL	16.51	3.38	2.66	56.8	2.61	2.80	4.17	11.1	497
C1157 Type HE	16.51	3.41	2.77	56.6	3.01	2.87	3.81	10.5	594

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