



# Fly ash particle characterization for predicting concrete compressive strength

Taehwan Kim<sup>a</sup>, Jeffrey M. Davis<sup>c,1,\*</sup>, M. Tyler Ley<sup>b</sup>, Shinhyu Kang<sup>b</sup>, Pouya Amrollahi<sup>b</sup>

<sup>a</sup>Center for Infrastructure Engineering and Safety, School of Civil and Environmental Engineering, University of New South Wales, Sydney, NSW 2052, Australia

<sup>b</sup>Oklahoma State University, Department of Civil and Environmental Engineering, Stillwater, OK 74078, USA

<sup>c</sup>PNDetector GmbH, Otto-Hahn-Ring 6, Munich, Germany

## HIGHLIGHTS

- It was demonstrated that fly ash particles can be grouped into compositionally distinct groups.
- Fly ashes with similar bulk composition may produce different compressive strengths in concrete.
- The compressive strength can be predicted using the particle group composition.
- Linear modeling of the particle group composition has significant predictive power for concrete.
- There are specific particle groups most strongly associated with early strength gain.

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## ABSTRACT

A new classification approach is presented that uses individual fly ash particle measurements to provide improved and more in-depth information about the properties of the concrete. The technique uses machine guided X-ray microanalysis to measure the compositions of 2000 randomly selected particles from 20 different fly ashes. This paper details the methods used to acquire the particle composition data and the derivation of the representative groups of the fly ash particles or classification groups. This method is named the Particle Model. To investigate the utility of these groups, 12 fly ashes were used at a 20% mass replacement of the cement in a series of concrete mixtures, which were tested for compressive strength at various times over 180 d of curing. Seven of the nine particle compositions identified were found to influence the compressive strength of the concrete with a linear model R-squared value of 0.99. The Particle Model showed a statistically significant improvement over the Class C or F classification from ASTM C618 and EN450. This work aims to establish the Particle Model and show that the classification shows promise to be used as a method to predict the physical properties of concretes that contain fly ash.

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## 1. Introduction

Over 98% of the ready mix concrete companies in the United States have used fly ash as a replacement for cement in concrete because of the good performance and economic benefits [1–3]. Fly ash is a by-product of the combustion processes of pulverized coal and is composed of finely divided spherical particles with a diameter of 1  $\mu\text{m}$  to 150  $\mu\text{m}$  obtained from a dust-collection system [2,4]. The major oxide components of fly ash are  $\text{SiO}_2$ ,  $\text{Al}_2\text{O}_3$ ,

$\text{Fe}_2\text{O}_3$ , and  $\text{CaO}$  with various minor oxides [1,2,5]. Applications for fly ash include: a low-cost adsorbent for cleaning flue gas, wastewater treatment, the raw material for synthesis of geopolymers and zeolites, a backfill material in mining, soil stabilization, and a supplementary cementitious material (SCM) in concrete [1,6,7].

Though the demand for fly ash as an SCM in concrete continues to increase, several challenges limit the consistency and the predictability [4,8–10]. Past studies of the reactivity of fly ash largely relied on bulk characterization methods, such as X-ray fluorescence (XRF), X-ray diffraction (XRD), and solution chemistry based on leaching tests [4,11–14]. Both ASTM C618 and EN 450 use bulk composition to classify fly ash as either Class C or F. While understanding a bulk property of a material is useful, it can only describe

\* Corresponding author.

E-mail address: [jeff.davis@pndetector.de](mailto:jeff.davis@pndetector.de) (J.M. Davis).

<sup>1</sup> This author is acting in a volunteer capacity in order to further the field of cement and concrete research.

the average of the system. Moreover, the individual fly ash particles do not have a homogeneous composition [12,15]. Rather, studies have shown that individual particles have a wide range of compositions, which, in turn, affect their performance in concrete. For example, the physical and chemical properties of a high-calcium fly ash were measured from a coal plant for approximately 2 years [16]. After analyzing one hundred and sixty fly ash paste samples, it was found that the bulk chemical compositions were fairly uniform. However, the 7 d compressive strengths of fly ash pastes varied widely. This highlights how bulk composition alone could not explain the change in the compressive strength and it suggests that a more fundamental understanding is needed in order to predict performance.

Potential insights into the performance of fly ash can be gained from a thorough study of the individual particles. Others have tried to take a more fundamental approach to understanding fly ash [8,15,17–21]. Several studies have tried to find unique chemical groups by using scanning electron microscopy with electron dispersive spectrometry (SEM/EDS) to examine polished sections of thousands of fly ash particles [8,15,22]. While the early results were variable, more recent work suggested that the fly ashes investigated are mainly composed of four groups of glasses: a) silicate, b) calcium silicate, c) alumino-silicate with low to moderate calcium, and d) calcium-rich alumino-silicate [15]. Additional work attempted to better understand the reactivity of these glasses by synthesizing glasses that are similar in composition and then testing them [22]. The work found that the different glasses showed different levels of reactivity. Both studies revealed that Ca-rich aluminosilicate has the fastest reactivity and silicate is the slowest [15,22].

Other recent work used lab scale micro-computed tomography ( $\mu$ CT) and SEM/EDS along with synchrotron-based nano-computed tomography (nCT) and nano X-ray fluorescence (nXRF) to study individual fly ash particles [17,19,23]. The fly ash particles investigated consisted largely of a single constituent with only minor inclusions. This means that individual fly ash particles have an almost constant chemical consistency. Thus, if the chemical consistency and elemental composition of individual particles are well understood, then this could provide important insights into fly ash performance. These previous studies made two critical observations: a) certain glass compositions are associated with different levels of reactivity [15,22] and b) single fly ash particles have a nearly uniform chemical composition with only minor chemical inclusions [17,19,23]. Although literature has tended to discuss the glassy and crystalline materials within fly ash, these observations suggest that it may be possible to characterize the reactivity of individual particles based on their chemical composition. In addition, these observations provide an important opportunity to study the variance of elemental oxides contents of individual particles and determine improved ways to classify them. This is the focus of the work in this paper.

The current classification system for fly ash based on ASTM C618 or EN450 is essentially a binary classification (i.e., an ash is either Class C or Class F) and so fly ash will fit within one of these categories. However, this work uses a large collection of data from individual particles and a rigorous data science approach to identify the unique groups of particles and classify them based on their performance in concrete. For this paper, the compressive strength of concrete will be used over the first 180 d. Additional work is underway to compare these results to other concrete properties but they are outside the scope of this work.

It is not the goal of this paper to provide a comprehensive understanding of why each particle group causes certain properties in concrete, nor is it to comprehensively predict the compressive strength of concrete. Instead, this paper aims to provide an outline of the analytical procedures that were used to find these groups,

introduce the groups, and show the usefulness of these groups to predict the compressive strength gain of concrete over time within the materials investigated.

## 2. Materials and methods

### 2.1. Materials

Twenty different fly ashes were investigated with automated scanning electron microscopy with electron dispersive spectrometry (ASEM-EDS). Ten of the fly ashes were classified as Class C and ten were classified as Class F by ASTM C618 [24]. All of these fly ashes were produced in the United States from varying coal sources, boiler designs, and collection conditions. The bulk chemical composition for all fly ashes was analyzed using XRF as per ASTM D4326 [25] and the ASEM-EDS method as per a previous publication [23]. The results are shown in Table 1.

All concrete mixtures used in the study used an ASTM C150 Type I [26] ordinary Portland cement (OPC) and the composition of cement is summarized in Table 2. Limestone and natural sands were used as a coarse aggregate and a fine aggregate, both of which were locally available in Oklahoma. The specific gravities of coarse and fine aggregates were both 2.6 and their absorptions were, respectively, 0.64% and 0.55%.

All of the aggregate, both coarse and fine, were brought into the temperature controlled mixing facility at least a day before and their batch weights corrected for the moisture content. The aggregates and two-thirds of the mixing water are charged into the mixer and mixed for three minutes. Next, any clumped fine aggregate was removed from the walls of the mixer. Then the cement and fly ash were added, followed by the remaining mixing water. The mixer was turned on for three minutes. Once complete, the mixture rested for two minutes while removing the buildup of material along the walls. Mixing continued for another three minutes. Slump, unit weight, and air content were measured [27–29].

Twelve different fly ashes (C1, C2, C3, C4, C5, C6, C7, F1, F2, F3, F4, and F5) were investigated with concrete mixtures. These fly ashes were chosen as they showed a wide range of different particle compositions. A mixture without fly ash was also included for comparison. Each concrete mixture had a water to cement ratio of 0.45. The mixtures with fly ash used a 20% replacement rate by mass of cement. Chemical admixtures are not included to reduce the potential variables. Table 3 shows the mixture proportions for a cubic meter of concrete. Cylindrical samples (10.2 cm in diameter and 20.3 cm in height) were prepared, sealed, and cured at 23 °C and 100% RH for 24 h. The samples were then demolded and placed in the curing room at 100% relative humidity until testing [30].

### 2.2. Automated scanning electron microscopy methods

The ASEM method uses a SEM equipped with an image analysis operating system and an energy dispersive X-ray spectrometry (EDS) system. The instrument used was an Aspex Explorer PSEM-EDS. One advantage of ASEM is the ability to rapidly measure the physical and chemical information of individual particles. This method investigates the composition, size, and shape of approximately 350 particles per hour with no intervention by the user [23]. The Instrument settings and consistency of results are presented in the Appendix A and is also discussed in previous publications [23].

One challenge with this method is that these particles are not flat and so they violate one of the assumptions of classical quantitative EDS analysis. However, EDS correction algorithms can account for geometric effects if the particles are a known shape

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