

Evaluation of the Physical and Chemical Properties of Fly Ash Products for Use in Portland Cement Concrete

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ABSTRACT

The physical and chemical properties of forty-nine different fly ash products from different sources within the United States were evaluated. The evaluation was conducted based on the standard physical and chemical requirements of ASTM C 618, "Standard Specification for Coal Fly Ash and Raw or Calcined Natural Pozzolan for Use in Concrete." Physical properties including fineness, density, water requirement and strength activity index (SAI), and major chemical properties including acidic elemental analysis and loss on ignition (LOI) were characterized in accordance with the testing procedures described in ASTM C 311, "Standard Test Methods for Sampling and Testing Fly Ash or Natural Pozzolans for Use in Portland-Cement Concrete." In this paper, the effects of fineness and carbon content on water requirement; and the effects of fineness, acidic oxide content, calcium content, and carbon content (derived from thermogravimetry) on 7-day strength activity index are described. In addition, the relationship between 7-day and 28-day SAI is presented.

INTRODUCTION

Fly ash is a by-product of the combustion of pulverized coal in electric power generating plants. It is the most widely used supplementary cementitious material in concrete. It is used in more than 50% of ready mix concrete. The replacement level of Class F fly ash is often at 15-25% by mass of cementitious materials, and it is between 15-40% by mass of cementitious materials for Class C fly ash. The properties of fly ash can greatly affect the properties for both fresh concrete and hardened concrete. Many countries have their own national standards and specifications regulating the use of fly ash in concrete. In the United States, ASTM C 618¹, "Standard Specification for Coal Fly Ash and Raw or Calcined Natural Pozzolan for Use in Concrete," is the common specification to assess the suitability of fly ash and pozzolan for use in concrete. The standard test methods related to this specification are contained in ASTM C 311², "Standard Test Methods for Sampling and Testing Fly Ash or Natural Pozzolans for Use in Portland-Cement Concrete."

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The standard physical requirements of ASTM C 618 include fineness, SAI with standard portland cement, water requirement, and soundness. The standard chemical requirements of ASTM C 618 include the sum of SiO₂, Al₂O₃ and Fe₂O₃ content (≥70% for Class F and ≥50% for Class C), SO₃ content, moisture content and loss on ignition.

Recently, CTLGroup evaluated forty-nine different fly ash products from different sources in the United States. The evaluation was based on the standard physical and chemical requirements of ASTM C 618. Results presented in the current paper revealed correlations between powdered fly ash and fresh and hardened properties when used in combination with portland cement and water in final products. Our observations can serve as a guide to allow ash industries to optimize the use of these by-products in portland cement concrete. In addition, the findings will also help to recognize the necessary characteristics of ash products potentially improving process efficiency.

MATERIALS AND EXPERIMENTAL PROCEDURES

The forty-nine fly ashes came from different sources within the United States. The portland cement selected for the work is a Type I and meets the requirements of ASTM C 311, its chemical composition and physical properties are provided in Table 1 and 2.

Table 1. Chemical Composition of the Selected Portland Cement and Fly Ashes

	Portland cement % by mass	Fly ash % by mass
CaO	64.01	0.37 – 27.68
SiO ₂	20.13	27.88 – 59.40
Al ₂ O ₃	5.78	5.23 – 33.99
Fe ₂ O ₃	2.35	1.21 – 29.63
MgO	1.19	0.42 – 8.79
SO ₃	3.53	0.04 – 4.71
Na ₂ O	0.11	0.20 – 6.90
K ₂ O	0.77	0.64 – 6.68
TiO ₂	0.37	0.24 – 1.73
LOI	1.63	0.21 – 28.37

The calculated cement compounds are 55% C₃S, 16% C₂S, 11% C₃A and 7% C₄AF.

Table 2. Physical Properties of the Portland Cement Used

Properties	Test results
Air content, %	7
Specific surface, m ² /kg	392
Autoclave expansion, %	0.02
Compressive Strength, MPa	

1 day	18.5
3 days	29.0
7 days	35.8
28 days	43.4
Vicat Time of Setting, minutes	
Initial set	140
Final set	210
Sulfate expansion in water, % (ASTM C 1038)	0.012

All the relevant chemical and physical testing was conducted in accordance with ASTM C 311. Elemental analysis was performed by x-ray fluorescence spectrometry and presented as oxides. Specimens were fused at 1000°C with $\text{Li}_2\text{B}_4\text{O}_7/\text{LiBO}_2$. Density of the forty-nine fly ashes was determined using a Helium Pycnometer.

RESULTS AND DISCUSSION

Water requirement vs fineness and loss on ignition

It is generally recognized that the addition of fly ash has a beneficial effect on the rheological properties of cement paste and on the workability of fresh concrete. The small spherical particles of fly ash can reduce the water requirement. Owens³ reported that the major factor influencing the effects of fly ash on the workability of concrete is the proportion of coarse material ($>45\mu\text{m}$) in the ash. A comprehensive study by Minnick et al⁴ revealed that both the $45\mu\text{m}$ sieve residue and the loss on ignition (LOI) have pronounced effects on water requirements. The effect of LOI on water requirement was attributed to the absorption of water by porous carbon particles.

Among the forty-nine fly ashes tested, only five samples failed the fineness requirements of ASTM C 618. The $45\mu\text{m}$ sieve residues of the five samples were 40%, 41%, 48%, 63% and 88% with water requirements of 97%, 112%, 93%, 98% and 107%, respectively. A sixth sample not included in the graph had a fineness of 24% (passing the fineness requirements), but with a water requirement of 120%. Figure 1 shows the influence of fineness on water requirement of the mortar specimens for forty three fly ashes that meet the fineness requirement (34% retained on $45\mu\text{m}$ sieve). Results show that most of the fly ash samples (88%) can reduce the water requirement of the mortar mixtures. However, the trend between the water requirement of mortars and the fineness is not as significant or consistent as reported by Minnick et. al⁴. The data does indicate that as the fineness increases the water requirement decreases.

Figure 2 shows the influence of loss on ignition on water requirement of the mortar specimens for forty seven fly ashes. Among the forty nine fly ashes tested, only two samples had a loss on ignition value above 12%. From Figure 2, it can be seen that if the fly ash has a loss on ignition above approximately (\sim) 3%, it is likely the water requirements are higher.

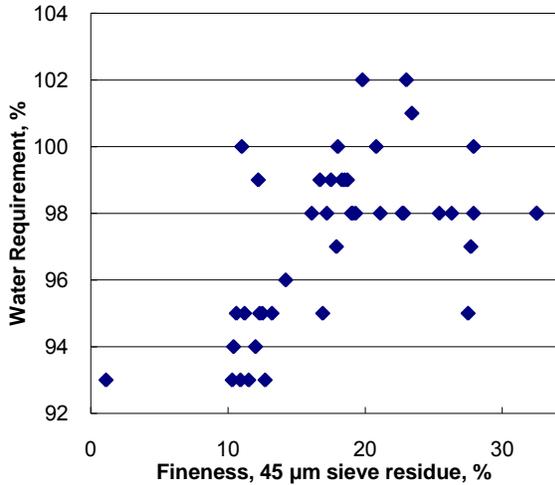


Figure 1. Water Requirement vs. Fineness

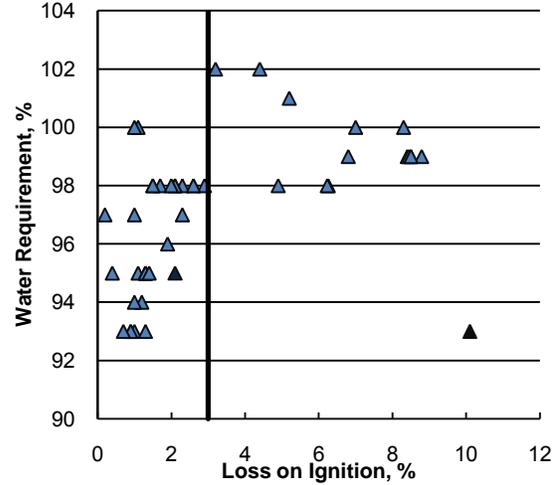


Figure 2. Water Requirement vs. LOI

An equation to describe the correlation between water requirement and fineness and LOI was reported by Helmuth⁵ as:

$$\text{Water requirement, \%} = 92.6 + 0.086 \times (\text{LOI} \times 45\mu\text{m sieve residue})$$

This equation applies when the 45µm sieve residue, loss on ignition, and their product are held within specified ranges (20% maximum 45µm sieve residue, 15% maximum LOI, and 150 maximum of the product). Figure 3 shows the correlation between the actual water requirement and the calculated water requirement obtained from the above equation. Almost two third of the fly ashes have a higher water requirement than the calculated value. The data indicates calculated values more accurately predict the actual water requirement at values below 98%.

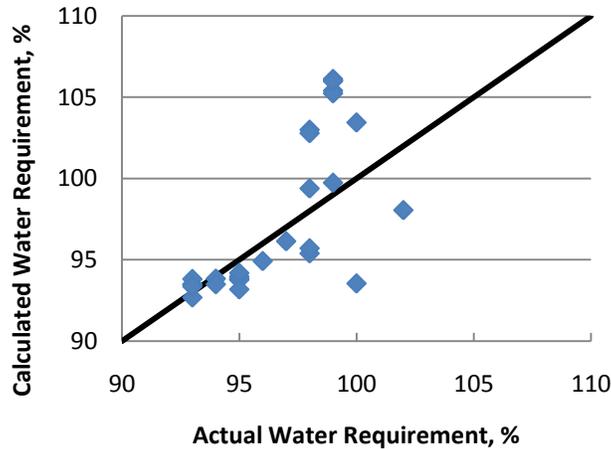


Figure 3. Actual Water Requirement vs. Calculated Water Requirement

Strength Activity Index vs. fineness, acidic oxide content, CaO content, LOI and density

It is well known that Class F fly ash has pozzolanic properties and Class C fly ash has pozzolanic properties, as well as some cementitious properties. It is also known that⁵⁻⁶ the most important factors affecting the pozzolanic reactivity of fly ashes are fineness, glass content, and acidic oxide content ($\text{SiO}_2 + \text{Al}_2\text{O}_3 + \text{Fe}_2\text{O}_3$). Other minor factors include CaO content, carbon content, and the composition of crystalline and organic substances on the particle surfaces⁵⁻⁶. In the ASTM C 618 specification, only the fineness, acidic oxide content and carbon content are included in the standard chemical and physical requirements.

Figure 4 shows the relation between 7d strength activity index (SAI) and fineness. A clear decreasing trend is observed - the coarser the fly ash particles, the lower the strength activity index.

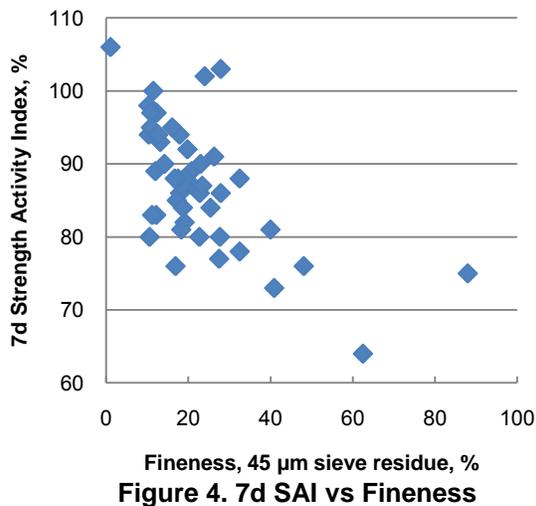


Figure 4. 7d SAI vs Fineness

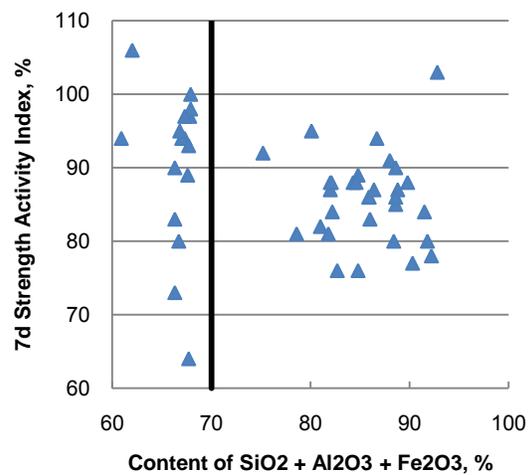


Figure 5. 7d SAI vs Acidic Oxide Content

According to the ASTM C 618 specification, the only major specification difference between the two classes of ashes is the minimum limit of $\text{SiO}_2 + \text{Al}_2\text{O}_3 + \text{Fe}_2\text{O}_3$, which is 70% for Class F fly ash and 50% for Class C. Although some researchers⁵⁻⁶ have proposed that the chemical specifications require determination of the total CaO content, the current ASTM C 618 still follows the above classification. Figure 5 shows the relation between the acidic oxide content and 7d strength activity index. It can be seen that for Class C fly ash, the acidic oxide content is not a contributing factor to determine the strength activity index; as the fly ashes within the range of 65 to 68% acidic oxide content, the strength activity index varies widely between 64 and 100%. There is no significant trend observed for Class F fly ash.

Our observations of acidic oxide content are consistent with other researchers' work⁷⁻⁸, which reported poor correlations between the compressive strength ratio and the sum of $\text{SiO}_2 + \text{Al}_2\text{O}_3 + \text{Fe}_2\text{O}_3$. However, other research⁵⁻⁶ established a good correlation between the total $\text{SiO}_2 + \text{Al}_2\text{O}_3$ content and long-term pozzolanic activity. This research suggests not only the amorphous silica and alumina contribute to pozzolanic activity,

but that compounds such as mullite and quartz may influence strength development either because they are reactive or they serve as bonded aggregate particles. Amorphous content is currently not measured.

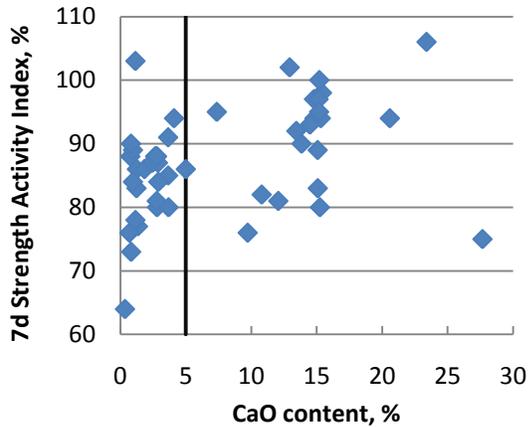


Figure 6. 7d SAI vs CaO Content

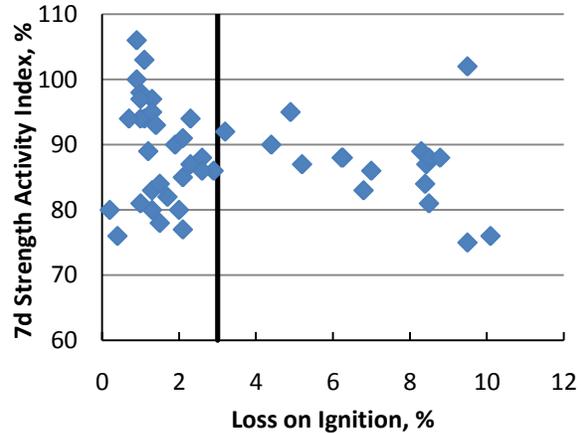


Figure 7. 7d SAI vs Loss on Ignition

Similar to the acidic oxide content observations no strong correlation between CaO content and the 7d strength activity index, as shown in Figure 6. The calcium contained in fly ash will be in different forms; such as dissolved in amorphous phases, free CaO, C₃A, CaSO₄, C₄A₃S, and other crystalline calcium compounds. These calcium forms yield different effects on the reactivity; hence, the net results obtained are mixed effects from different forms of calcium.

Figure 7 shows the relation between the loss on ignition and 7d strength activity index. When the loss on ignition is less than ~ 3%, the influence on strength activity index is not significant. When the loss on ignition is above 3%, there is a decreasing trend on the strength activity index with increasing loss on ignition. When compared to Figure 2, the water requirement generally increased with LOI above ~ 3%, thus the strength most probably decreased due to increased water.

Although within ASTM C 618 there is no requirement on density for fly ash, as a part of our evaluation the density of the forty-nine fly ashes was tested by Helium Pycnometer. The densities are distributed between 1.75 to 2.88 g/cm³. The relation between density and 7d strength activity index is shown in Figure 8. Again, no satisfactory trend can be established.

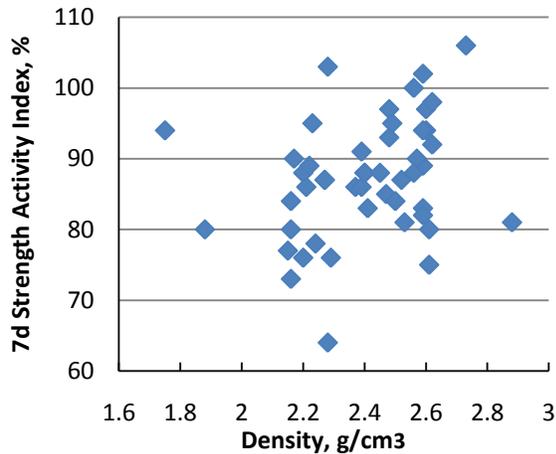


Figure 8. 7d SAI vs Density

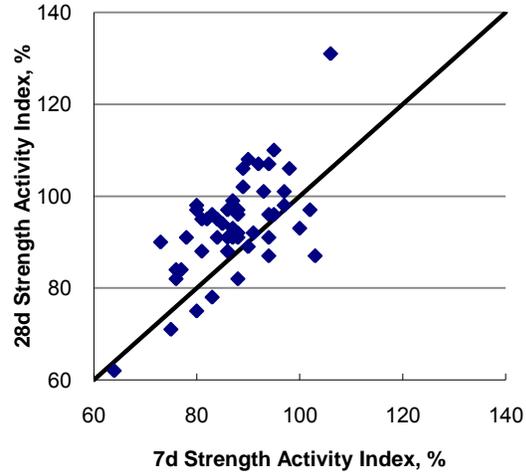


Figure 9. 7d SAI vs 28d SAI

Figure 9 shows the relation between 7d and 28d strength activity index. About 80% of the fly ashes tested have gains in SAI from 7d to 28d. As specified in ASTM C 311, the SAI is a percentage of the compressive strength of the test mixture that contains 20% (by mass) fly ash and 80% (by mass) portland cement to the compressive strength of the control mixture that contains 100% (by mass) portland cement. Hence, a decrease in the 28d strength activity index does not mean the absolute compressive strength of the fly ash mixtures is decreased at 28d. However, it should be noted that ASTM C 618 states “meeting the 7 day or 28 day SAI (both at 75%) will indicate specification compliance.” If a fly ash meets a 7d SAI, the later age SAI might still fail the 28d SAI requirement.

CONCLUSIONS

Based on the above results, the following conclusions can be drawn:

1. About 90% of the fly ashes tested can reduce the water requirement for mortar mixtures, but correlation between water requirement and fineness, or between water requirement and loss on ignition.
2. A decreasing trend between 7d SAI and fineness was observed. As the amount of material retained on 45um sieve increased the 7day SAI decreased.
3. The effects of $\text{SiO}_2 + \text{Al}_2\text{O}_3 + \text{Fe}_2\text{O}_3$ content, CaO content and density of the fly ashes on the 7d strength activity index could not be clearly defined.
4. Loss on ignition does not influence the 7d strength activity index when it is below approximately 3%; but a decreasing trend with LOI above approximately 3% is observed.
5. About 80% of tested fly ash mixtures have gains in strength activity index from 7d to 28d.

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