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Dale P. Bentz,[1] *Alejandro Durán-Herrera,*[2] *and Daniel Galvez-Moreno*[3]

Comparison of ASTM C311 Strength Activity Index Testing versus Testing Based on Constant Volumetric Proportions

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ABSTRACT: Currently, the (pozzolanic) strength activity indices of fly ashes and natural pozzolans are typically evaluated using the procedures outlined in ASTM C311 ["Standard Test Methods for Sampling and Testing Fly Ash or Natural Pozzolans for Use in Portland-Cement Concrete," *Annual Book of ASTM Standards*, ASTM International, West Conshohocken, PA, 2007]. In this test, the 7 and 28 d compressive strengths of mortar cubes with a 20 % mass replacement of cement by pozzolan are compared to those of a control without pozzolan, at constant flow conditions. In its current form, this procedure confounds two other properties of the pozzolan with its strength activity, namely its density and its water-reducing/increasing capabilities. In this study, the current C311 testing procedure is evaluated against an alternative in which the 20 % fly ash replacement for cement is performed on a volumetric basis and the volume fractions of water and sand are held constant, which should provide a true evaluation of the strength activity index of the pozzolan, free of these confounding influences. Class C and Class F fly ashes, a natural pozzolan, and a sugar cane ash are evaluated using both approaches, with some significant differences being noted. For a subset of the materials, the strength measurements are complemented by measurements of isothermal calorimetry on the mortars to an age of 7 d. For the constant volumetric proportions approach, a good correlation is exhibited between the cumulative heat release of the mortar at 7 d and the measured 7 d strength, suggesting the potential to evaluate 7 d pozzolanic activity via calorimetric measurements on much smaller specimens.

KEYWORDS: compressive strength, density, flow, isothermal calorimetry, pozzolan, strength activity index, water reduction

Introduction

The sustainability movement has spurred renewed interest in reducing the cement content of concrete mixtures by replacing an ever-increasing portion of the Portland cement with supplementary cementitious materials (SCMs), including fly ash, limestone powder, natural pozzolans, slag, and silica fume [1,2]. One key characteristic of fly ashes and natural pozzolans is their capability to undergo a pozzolanic reaction with the calcium hydroxide produced during conventional cement hydration to produce an increased quantity of calcium silicate hydrate gel (C–S–H), often leading to long term benefits, including increases in compressive strength and decreases in transport coefficients.[4] Naturally, one would prefer to perform a quantitative evaluation of this pozzolanic activity a priori for each SCM of interest. Although a variety of standardized test methods exist for evaluating pozzolanic activity [3], in the United States, for fly ash and natural pozzolans, this assessment is commonly based on a strength activity index outlined in ASTM C311 [4] and specified by ASTM C618 [5]. In this test method [4], the 7 and 28 d compressive strengths of a mortar prepared with a 20 % SCM substitution for cement on a mass basis are compared to those of a control mortar. Although the control mortar is prepared with a water-to-cement ratio by mass (w/c) of 0.484, the water content of the test mixture is adjusted to provide an equivalent flow to that measured for the control. The mixture with the SCM should provide 75 % of the strength of the control at 7 or 28 d, according to the ASTM C618 specification [5].

Recently, several research groups have pointed out some of the inherent limitations in the current format of the strength activity index testing of ASTM C311 [3,6,7]. In its current form, the test method

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[1] Engineering Laboratory, National Institute of Standards and Technology, Gaithersburg, MD 20899-8615.

[2] Academic Group on Concrete Technology, Universidad Autónoma de Nuevo León, Monterrey, Nuevo León, Mexico.

[3] Facultad de Ingeniería Mochis, Universidad Autónoma de Sinaloa, Los Mochis, Sinaloa, Mexico.

[4] In addition to pozzolanic activity, some fly ashes (typically Class C) are hydraulic and will react with water to produce their own space-filling hydration products.

introduces two significant confounding factors in addition to the true pozzolanic (re)activity of the SCM. First, because the replacement of cement by SCM is performed on a mass basis and because the densities of most SCMs are significantly less than that of cement, such a replacement results in a reduction in the volume fractions of water and sand in the mortar mixture. It is of course well known that a reduction in the water volume fraction of a mixture generally increases its compressive strength. Additionally, because the test mortar and the reference (cement only) mortar are prepared to provide equivalent flows, the water content (volume fraction) of the test mixture can be further modified from that of the reference. If increased water is required to produce equivalent flow, the strength of the test mixture will suffer; if a water reduction is achieved, its strength may be increased instead. Regardless, the strength activity index of the SCM is being evaluated in the presence of these confounding effects. It is interesting to note that the ASTM C595 specification for blended hydraulic cements [8], in its Annex A1, recognizes part of this problem and performs its activity index testing with a 35 % volumetric replacement of cement by pozzolan. Unfortunately, although, it still recommends testing at constant flow conditions of 100–115, as measured using the flow table [9]. In cases where a water increase is required to provide this requisite flow, an alternative that might be more consistent with current concrete practice would be to employ a water reducer at the same water content as the control mixture.

In the current study, for a limited set of cements (2), fly ashes (1 Class C and 2 Class F), and (natural) pozzolans (2), the current ASTM C311 strength activity index testing paradigm is contrasted to a proposed protocol where constant volumetric proportions are maintained. In the latter case, the test and reference mixtures have the same volume fractions of water, sand, and powders (cement and any SCM) and the change in flow induced by the replacement of cement by SCM is measured. In addition to measuring flows and compressive strengths at various ages, a subset of the mixtures are also characterized with respect to their 7 d heat of hydration, to assess the potential of quantifying pozzolanic activity through 7 d by measuring heat release (indicative of hydration and pozzolanic reactions) from much smaller specimens than those required for conventional compressive strength testing.

Materials and Experimental Methods

The testing program included two ASTM C150 Type I cements [10], one each from the United States and Mexico, three fly ashes, one natural pozzolan from Mexico, and a sugar cane ash from Sinaloa, Mexico. Densities of the various powders were provided by their manufacturer or were determined using ASTM C188 [11] (see Table 1). Each of the three fly ashes and the two pozzolans has a density that is significantly less than that of the two cements. The individual particle size distributions of the seven powders, as measured using a laser diffraction-based particle size analyzer, are shown in Fig. 1. The Class F fly ash designated as FAF2 exhibits a modal particle size greater than 100 μm and is much coarser than any of the other powders. Regarding the chemistry of the fly ashes, the Class C fly ash has CaO, SiO_2, Al_2O_3, and Fe_2O_3 contents of 23.5 %, 38.7 %, 19.2 %, and 6.5 % by mass, respectively. For the FAF1 Class F fly ash, these values are 1.2 %, 59.6 %, 28.9 %, and 3.2 %, whereas for the FAF2 Class F fly ash, they are 2.5 %, 60.4 %, 27.3 %, and 4.1 %.

The strength activity indices of the fly ashes and pozzolans were evaluated according to the ASTM C311 standard test method at both the National Institute of Standards and Technology (NIST) in the United States and the Universidad Autónoma de Nuevo León (UANL) in Mexico. First, control mortars were prepared according to the recipe provided in ASTM C311 [4]. Their flow was measured and cubes were prepared for compressive strength testing. For the mixture with the pozzolan substitution, the water in the test mixture was adjusted via trial and error to achieve a flow value that was within five units of that produced by the control mixture. Once the desired flow was produced, that batch of mortar was used to

TABLE 1—*Densities of materials investigated in the study.*

Material	Designation	Density (kg/m^3)	Mass Fraction at 20 % Volume Replacement (%)
Type I Cement—United States	Cement—United States	3.15	
Type I Cement—Mexico	Cement—Mexico	3.15	
Class C Fly Ash	FAC	2.63	17.3
Class F Fly Ash	FAF1	2.16	14.6
Class F Fly Ash	FAF2	2.10	14.3
Natural pozzolan	NP	2.40	16.0
Sugar cane ash	SCBA	2.24	15.1

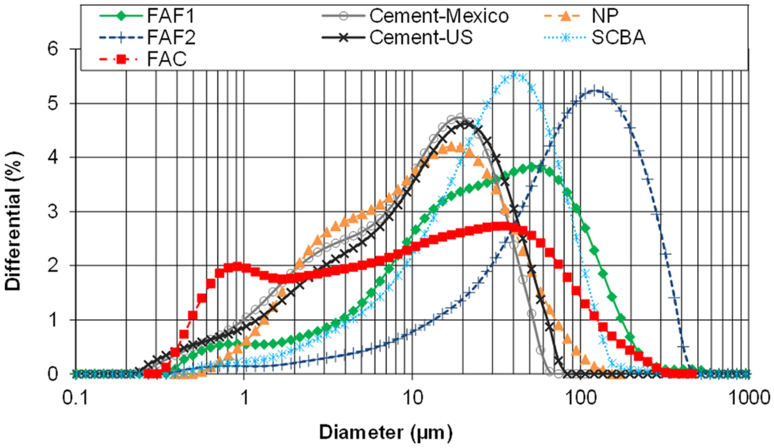

FIG. 1—*Measured particle size distributions for materials examined in the present study. The results shown are the average of six individual measurements and the error bars (1 standard deviation) would fall within the size of the shown symbols.*

prepare cubes for compressive strength testing. Finally, a second test mixture was prepared with constant water and sand volume fractions and a 20 % replacement by volume of cement by pozzolan (mass fractions provided in Table 1). To achieve a flow greater than or equal to that of the control for this mixture, if necessary, a small addition of a high range water reducing agent (HRWRA) was included. For the materials examined in this study, this HRWRA addition was only deemed necessary when employing the FAF2 fly ash or the sugar cane ash, with the latter greatly increasing the water demand of the mortar. For these two materials, a polycarboxylate or a naphthalene-based HRWRA was used with dosages of 0.03 % and 0.2 % by mass of powders, respectively. For the testing completed at NIST, cube strengths were measured at 7 and 28 d and small specimens (~7.5 g) of the mortar were also characterized by isothermal calorimetry measurements to an age of 7 d. For the testing at UANL, no calorimetry was executed, but strengths were measured at additional ages of 3 d, 14 d, 56 d, and 6 months.

Results and Discussion

The testing results, following the ASTM standards and the proposed modification to constant volumetric proportions, obtained in the United States and Mexico are summarized in Table 2 (mixture proportions

TABLE 2—*Water reductions and flows for mortar mixtures.*

Testing Protocol	Cement	Fly Ash or Pozzolan	Water in Mixture (g)	Measured Flow[a]
Control	Cement—United States	None	242	90 ± 5[b]
C311	Cement—United States	FAC	220	90
Volumetric	Cement—United States	FAC	242	124
C311	Cement—United States	FAF1	238	86
Volumetric	Cement—United States	FAF1	242	85
C311	Cement—United States	NP	241	91
Volumetric	Cement—United States	NP	242	86
C311	Cement—United States	SCBA	280	94
Volumetric	Cement—United States	SCBA	242 (HRWRA)	89.5
Control	Cement—Mexico	None	242	95 ± 5[b]
C311	Cement—Mexico	FAC	220	93
Volumetric	Cement—Mexico	FAC	242	126
C311	Cement—Mexico	FAF1	240	93
Volumetric	Cement—Mexico	FAF1	242	90
C311	Cement—Mexico	FAF2	260	96
Volumetric	Cement—Mexico	FAF2	242 (HRWRA)	108
C311	Cement—Mexico	NP	240	93
Volumetric	Cement—Mexico	NP	242	75

[a] ASTM C1437 [9].

[b] Standard deviation in flow value measured on six replicate control mixtures.

TABLE 3—*Strength activity indices for mortar mixtures.*

Testing Protocol	Cement	Fly Ash or Pozzolan	7 d Index (%)	28 d Index (%)
Control	Cement—United States	None	100 (29.7 MPa ± 1.1 MPa)[a]	100 (41.0 MPa ± 1.3 MPa)
C311	Cement—United States	FAC	100.8 (1.2 MPa)[b]	94.4 (2.7 MPa)
Volumetric	Cement—United States	FAC	87.2 (0.8 MPa)	88.9 (1.4 MPa)
C311	Cement—United States	FAF1	81.6 (0.7 MPa)	82.4 (1.6 MPa)
Volumetric	Cement—United States	FAF1	**72.3** (1.4 MPa)	**73.4** (1.2 MPa)
C311	Cement—United States	NP	85.9 (0.3 MPa)	86 (1.3 MPa)
Volumetric	Cement—United States	NP	78 (0.6 MPa)	78.2 (1.8 MPa)
C311	Cement—United States	SCBA	**56.2** (0.4 MPa)	**61.5** (1.1 MPa)
Volumetric	Cement—United States	SCBA	76.7 (2.1 MPa)	79.1 (0.4 MPa)
Control	Cement—Mexico	None	100 (33.7 MPa ± 2.5 MPa)[a]	100 (40.2 MPa ± 1.5 MPa)
C311	Cement—Mexico	FAC	97.1 (1.6 MPa)	112.6 (1.1 MPa)
Volumetric	Cement—Mexico	FAC	108.4 (0.7 MPa)	115.7 (1.0 MPa)
C311	Cement—Mexico	FAF1	83.6 (1.4 MPa)	89.9 (0.4 MPa)
Volumetric	Cement—Mexico	FAF1	77.7 (1.0 MPa)	79.7 (0.2 MPa)
C311	Cement—Mexico	FAF2	76.6 (1.0 MPa)	**73.6** (1.3 MPa)
Volumetric	Cement—Mexico	FAF2	90.5 (0.4 MPa)	78.9 (0.4 MPa)
C311	Cement—Mexico	NP	89.1 (0.5 MPa)	101.3 (0.9 MPa)
Volumetric	Cement—Mexico	NP	83.7 (0.7 MPa)	85.8 (1.2 MPa)

[a] Mean compressive strength and standard deviation measured on three cubes.
[b] Standard deviation measured on three cubes.

and flows) and Table 3 (strength activity indices). In Table 3, the mixtures not meeting the performance criteria established in the current ASTM C618 specification (e.g., greater than or equal to 75 % of the compressive strength of the control at the same age) are highlighted in bold. The performance attributes of each pozzolan, under the two testing paradigms, will be discussed separately.

The performance of the Class C fly ash (FAC) is quite similar for the two cements investigated in the present study. This C ash is a fairly active material, capable of quickly reacting on its own with water to form a set mass within 30 min. Its beneficial influence on rheological properties permitted a water reduction of almost 10 % based on the current testing protocols. Such a large water reduction will obviously be one contributor to increased strength development. In fact, under both testing paradigms, the 7 and 28 d strength results were well above the 75 % level prescribed in ASTM C618. The C ash appears to be particularly active in combination with the cement from Mexico as, in general, the strength activity indices for this combination in Table 3 (and Table 4) are well above 100 %.

FAF1, the first of the two Class F fly ashes, only provided for a minor water reduction of 4 g (1.7 %) and 2 g (0.8 %) for the U.S. and Mexican cements, respectively. However, its lower specific gravity of 2.16 significantly decreased the water (and sand) volume fractions in the ASTM C311 mixture. Thus, the strength activity indices obtained via the C311 testing were significantly higher than those obtained with the proposed constant volumetric proportions protocol. For evaluation with the U.S. cement, in fact, the measured 7 and 28 d strength activity indices were below the 75 % criteria currently prescribed in ASTM C618. Of course, for constant volumetric proportion testing, the actual prescribed level of performance would need to be established as part of a consensus process (and might be set at 70 % for instance).

The density of the FAF2 fly ash was the lowest within the studied materials; because it is significantly less than that of the cement, the water demand to achieve the target flow on the ASTM C311 protocol was higher than that of the reference by 18 g (7.4 %). Because of this material's tendency to increase water demand, for the constant volumetric proportions protocol, the use of a polycarboxylate-based HRWRA was necessary to increase the flow from 56 to a more reasonable value of 108. From Table 3, the increase in the strength activity indices obtained by the constant volumetric proportions protocol is mainly attributed to its lower water content relative to the constant flow mixture, where the extra 18 g of water necessary to achieve the target flow would naturally produce reduced strengths (at all ages). According to the ASTM C618 specification, the strength activity index for the constant flow mixture is below the requisite 75 % at 28 d, although its 7 d value would meet the requirements of the specification.

TABLE 4—*Longer term strength activity indices for mortar mixtures.*

Testing Protocol	Cement	Fly Ash or Pozzolan	56 d Index (%)	6 m Index (%)
Control	Cement—Mexico	None	100 (42.9 MPa ± 2.9 MPa)[a]	100 (44.6 MPa ± 0.2 MPa)
C311	Cement—Mexico	FAC	113.6 (1.2 MPa)[b]	113.0 (1.2 MPa)
Volumetric	Cement—Mexico	FAC	108.5 (0.4 MPa)	122.9 (1.2 MPa)
C311	Cement—Mexico	FAF1	90.9 (1.3 MPa)	117.4 (1.1 MPa)
Volumetric	Cement—Mexico	FAF1	92.9 (2.6 MPa)	96.4 (1.3 MPa)
C311	Cement—Mexico	FAF2	77.0 (0.8 MPa)	91.0 (1.2 MPa)
Volumetric	Cement—Mexico	FAF2	89.3 (1.1 MPa)	Not measured
C311	Cement—Mexico	NP	104.6 (1.0 MPa)	119.8 (0.5 MPa)
Volumetric	Cement—Mexico	NP	99.4 (2.5 MPa)	98.0 (1.9 MPa)

[a] Mean compressive strength and standard deviation measured on three cubes.
[b] Standard deviation measured on three cubes.

The natural pozzolan provided almost no water reduction under the C311 testing protocol; however, its density of 2.4 is significantly less than that of Portland cement, so that a reduction in water volume fraction is still achieved. Thus, some reductions in strength activity indices were observed for both cements when applying the proposed constant volume protocol relative to the C311 procedures. In general, all indices were well above the 75 % level prescribed in ASTM C618.

The sugar cane ash employed in the present study was unique in that it required a significant water increase (>15 %) to achieve the prescribed flow of ASTM C311. This naturally yielded reduced strengths, with its strength activity indices at 7 and 28 d being only 56.2 % and 61.5 %, respectively. However, when a small amount of HRWRA was employed along with constant volume proportions, the activity indices were significantly increased to 76.7 % and 79.1 %, both above the current ASTM C618 75 % threshold level. These results imply that the sugar cane ash may have adequate reactivity as a pozzolanic material, but that it would perhaps prematurely be eliminated from consideration as such, under the current C311 testing procedures. It should be noted that the addition of a HRWRA to concrete mixtures is now commonplace and is typically employed to avoid an increase in water content. Thus, it is suggested that the strength activity index testing should reflect this practical consideration as opposed to its current testing at constant flow conditions via water adjustments.

For the testing in the United States, the strength activity index evaluation was complemented with measurements of isothermal calorimetry to an age of 7 d (equivalently, 168 h). As shown in Fig. 2, the isothermal calorimetry provides a valuable indication of both any retardation due to the incorporation of the fly ash or pozzolan into the mixture and the longer term reactivity of the pozzolan. Specifically, in contrasting the curves for the Class C and Class F fly ashes in Fig. 2, the C ash causes an initial retardation, as exemplified by a shift of the initial rise in the hydration curve to later ages, but ultimately is more reactive

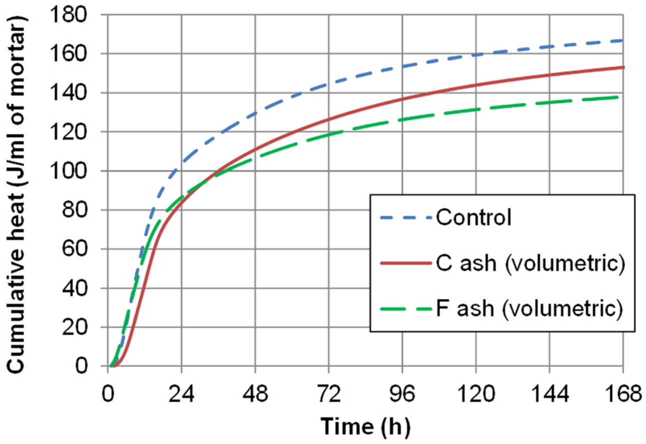

FIG. 2—*Isothermal calorimeter cumulative heat release curves to 7 d for three of the mortar mixtures.*

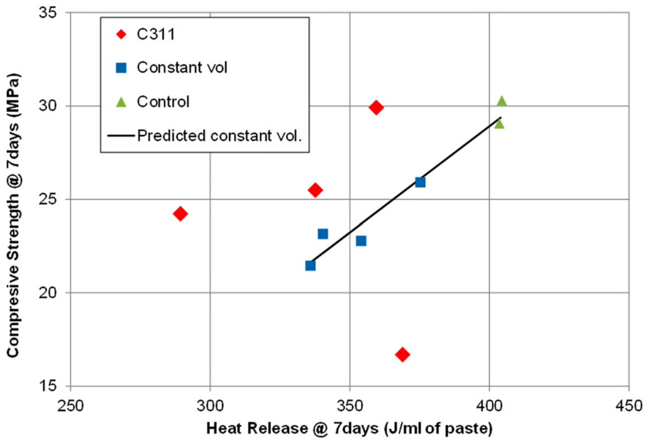

FIG. 3—*Compressive strength at 7 d versus heat release per unit volume of paste at 7 d.*

than the F ash, as it passes the F ash curve beyond an age of about 30 h. These results in Fig. 2 suggest that it might be possible to quantify fly ash and pozzolan reactivity with a given cement by monitoring the isothermal heat release of pastes with and without the pozzolan.

To further develop this hypothesis, a plot comparing the isothermal calorimetry results to the measured compressive strengths at 7 d, for both of the testing paradigms investigated in the present study is provided in Fig. 3. For the current testing protocol, basically no correlation ($R^2 = 0.04$) is observed between heat release and compressive strength, mainly due to the confounding influence of variable water volume fractions in the different mixtures. However, when the volume fraction of water (and other components) is maintained constant under the proposed testing protocol, a straight line relationship with $R^2 = 0.95$ is observed. Thus, under a constant volume fraction paradigm, the potential for evaluating pozzolanic activity via thermal as opposed to strength measurements seems quite promising and worthy of further research. The slope of the line in Fig. 3 can be used to infer the relative discrimination of the two methods. For the data shown, a span of ~28 % in compressive strength is accompanied by a span of only ~17 % in 7 d cumulative heat release. This would suggest that a 75 % acceptance criteria based on compressive strength might need to be replaced with an 80 % or even an 85 % acceptance criteria based on cumulative heat release.

A final consideration for strength activity testing is the age at which to undertake the strength evaluations. The current C618 specification passes a material that meets either the 7 and 28 d strength index criteria. For some fly ashes and natural pozzolans; however, contributions from pozzolanic reactions become significant only after a few weeks, so that perhaps longer term testing should be considered. With this in mind, the testing ages employed in Mexico were extended to 56 d and 6 months, with the results being provided in Table 4. In general, for the materials evaluated in the present study, the strength activity index increases with testing age beyond 28 d, so that materials that did not meet the 7 or 28 d acceptance criteria achieved acceptable performance at 56 d. Consistent with this observation, there has been considerable discussion within the concrete industry in recent years as to whether 28 d strength conformance testing should instead be conducted after 56 d or even 91 d for high volume fly ash concretes and other sustainable concrete mixtures with significantly reduced cement contents, to allow a sufficient period of time for their true strength characteristics to more fully develop.

Conclusions

A new constant volumetric proportioning paradigm for the evaluation of pozzolanic activity indices has been proposed. The new paradigm avoids the confounding influences of the reduced density of most pozzolans relative to that of cement and the constant flow conditions prescribed in the current ASTM C311 standardized test method. The new protocol has been contrasted against the current C311 procedures, with significant differences noted for several of the materials examined in this study. A possible additional advantage of the constant volumetric proportions approach is that it may lend itself to a simpler evaluation of 7 d pozzolanic activity via isothermal calorimetry testing.

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