Evaluating Test Methods for Rapidly Assessing Fly Ash Reactivity for Use in Concrete

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ABSTRACT

In addition to various properties, including fineness and loss on ignition, used to assess fly ash, specifications covering the material as an addition in concrete normally include a measure of its pozzolanic activity. While variations exist, approaches commonly used involve testing the compressive strength of standard fly ash mortar, at different ages, and comparisons with the corresponding Portland cement control. Given the nature of the material, these can be carried out over extended periods (e.g. 90 days for Activity Index in BS EN 450-1). If the timescale was reduced, this could offer benefits with regard to fly ash testing and use. The paper will summarize various options in the literature towards achieving this and describe an investigation carried out to examine the potential of selected property measurements/techniques in the role. These include (i) fly ash characteristics: fineness, bulk chemistry and amorphous content; (ii) accelerated curing (elevated temperature) conditions for Activity Index and hydrated lime/fly ash mortars; and (iii) wet chemical tests involving lime-consumption. The methods were considered for a range of low-lime fly ashes covering different sources, seasonal variations and co-combustion, with evaluations made through comparisons against the Activity Index to BS EN 450-1. The paper will identify their relative effectiveness in assessing fly ash reactivity and explore how they relate to concrete.

INTRODUCTION

Fly ash has a long history of use in concrete, with early research investigating the material in this application dating back to the 1930s.[1] Much of the work carried out on fly ash concrete has demonstrated that it offers a range of technical benefits, covering fresh and engineering properties, and aspects of durability.[2, 3] Furthermore, it can provide environmental and economic advantages to construction.[3, 4]

Specifications for fly ash, corresponding to the period of the materials' use, have been revised over time, to reflect experience gained and developments taking place in the power industry.[5] One of the key characteristics, which can be traced to their early introduction, is a measure of fly ash reactivity (pozzolanic activity, see Table 1).[6]

Table 1. Timeline for the inclusion of key fly ash characteristics in various specifications based on Ref.[6]

Specification		Loss on Ignition, (max, %)	Fineness		Water req. (max, %)	Pozzolanic activity (min, %)
			Min. spec. surface, (m^2/kg)	45 µm sieve retention, (max, %)		
Davis *et al.* (1937)		7.0	250	12.0	103	≥ 5 N/mm^2 with lime
US Bureau of Reclamation (1951)		5.0	300	12.0	103	-
Nebraska State (1952)		10.0	-	-	-	-
ASTM C350 (1954)		12.0	280	-	-	100
BS 3892 (1965)	Zone A	7.0	125-275	-	-	-
	Zone B		275-425			
	Zone C		> 425			
ASTM C618 (1968)		12.0	650	-	105	85
Tennessee Valley Authority G-30 (1968)	Class I	6.0	650	< 12.0	-	85
	Class II	6.0	500	12.0-22.0	-	75
	Class III	6.0	-	22.0-32.0	-	75
ASTM C618 (1969)		12.0	-	12.0	105	75
ASTM C618 (1971)		12.0	-	34.0	105	75
BS 3892-1 (1982)		7.0	-	12.5	< 95	85 (7 days[a])
BS EN 450 (1995)		5.0	-	40.0	-	75 (28 days) 85 (90 days)
BS 3892-1 (1997)		7.0	-	12.0	< 95	80 (28 days)
BS EN 450-1 (2012)	Cat. A	0.0-5.0	-	< 12 (Cat. S)	< 95	75 (28 days)
	Cat. B	2.0-7.0	-	< 40 (Cat. N)	-	85 (90 days)
	Cat. C	4.0-9.0	-	-	-	
ASTM C618 (2012)	Class N	10.0	-	34.0	115	75 (7 days) or 75 (28 days)
	Class F	6.0[b]	-	34.0	105	
	Class C	6.0	-	34.0	105	

[a] *20°C in moist air for 1 day, then water curing at 20°C for 4 days, 50°C for 46 hours, and 20°C for 2 hours before testing*

[b] *up to 12.0% may be approved by the user if acceptable performance records / laboratory tests are made available*

While there are variations, methods commonly adopted for evaluating this involve compressive strength tests on standard fly ash mortar at different ages, with comparisons made against that of the Portland cement (PC) control.[7, 8] The timescale for these can, however, be up to 90 days[7] and hence properties are established for the material at relatively long periods after it is produced. If this was shortened, it could offer benefits with regard to fly ash testing and use.

The research described in the paper investigates alternative methods to the cement mortar-type Activity Index test, with a view to establishing fly ash reactivity more rapidly.

SUMMARY OF STUDIES ON FLY ASH REACTIVITY ASSESSMENT

Studies have investigated changes in the contribution of physical packing and pozzolanic reactions to Strength Activity Index with time for fly ashes of different median particle size.[9] Attempts have also been made to examine reactivity in terms of efficiency or cement equivalence factors and to identify parameters (e.g. fly ash properties, mix proportions) influencing this.[10, 11] Other research on concrete has noted strength increases with fly ash fineness.[12]

It has been suggested from work examining various materials, including fly ash, that later age pozzolanic activity is mostly dependent on chemical and mineralogical composition, and amorphous/glassy phase quantity.[13] Research examining the glass phase of fly ash indicates that differences in composition may be found with coal rank.[14] These could also be related to factors including fly ash particle density and surface area.

Investigations have considered the reactive nature of fly ash by activation through exposure to various solutions, with tests made on the resulting leachate and residue. These include fly ash/PC suspensions in water, fly ash/$Ca(OH)_2$ and fly ash/K or Na activators.[15-17]

Several techniques have been used which follow pozzolanic activity tests, but change conditions to give results more rapidly, e.g. high temperature or microwave-assisted curing[18, 19] or autoclaving.[20]

Research has used differential thermal analysis and thermogravimetric methods to determine $Ca(OH)_2$ residues in hydrated pastes.[21, 22] These have contributed to the understanding of chemical processes associated with fly ash, how these can be modified, and in determining pozzolanic reaction rates. Calorimetric methods including heat of hydration of mortar containing fly ash have been found to give good correlations with pozzolanic activity.[16]

Electrical response techniques have also been considered in this area.[23, 24] Studies have used fly ash/$Ca(OH)_2$ mixtures and PC/fly ash pastes, with measurements including conductivity and electrical resistance/resistivity carried out. This approach has proved useful for evaluating the effects of fly ash and other pozzolanic materials.

The Summary indicates that a range of approaches are possible. In this paper, options covering three specific areas were considered, as follows, (i) fly ash characteristics: fineness, bulk chemistry and amorphous content, (ii) accelerated curing (elevated temperature) conditions for Activity Index and hydrated lime/fly ash mortars and (iii) wet chemical tests involving lime-consumption. These were then evaluated against standard BS EN 450-1 Activity Index measurements.

TEST FLY ASHES AND STANDARD ACTIVITY INDEX VALUES

The research used 10 fly ashes from 6 UK sources, with at least 6 of these adopted for each of the rapid assessment tests considered. The fly ashes were all low-lime and included material of high/low fineness and loss on ignition (LOI), as well as that produced in winter/summer and by co-combustion. This therefore provided fly ashes with various reaction characteristics. Details of the range of main properties for the test materials are given in Table 2.

Table 2. Summary of main property ranges for fly ashes tested

Property	Range	Property	Range
45 µm sieve ret., %	4.8 – 36.1	CaO, %	2.6 – 6.6
		SiO_2, %	44.7 – 56.2
LOI, %	2.6 – 13.4	Al_2O_3, %	18.8 – 25.2
		Fe_2O_3, %	4.4 – 8.7
7 days AI, %	75 – 85	K_2O, %	1.4 – 3.4
28 days AI, %	80 – 94	Na_2O, %	0.7 – 1.6
90 days AI, %	93 – 110		
180 days AI, %	95 – 116	Amorphous/others, %	57.1 – 80.5

AI: Activity Index

Activity Index tests were carried out following the method described in BS EN 450-1. These used a PC of Strength Class 52.5 N to BS EN 197-1[25] and CEN standard sand as described in BS EN 196-1.[26]

The mortar in the tests had a water/cement ratio (w/c) of 0.5, a fly ash level in cement of 25% and a cement/sand ratio of 1/3. The prism samples (40 mm × 40 mm × 160 mm) were cured in water at 20°C and tested in compression at 7, 28, 90 and180 days (the middle two being the test ages given in BS EN 450-1).

The range of results obtained from the tests at these ages for the 10 fly ashes is also shown in Table 2. These follow expected behaviour with Activity Index giving continued increases with age, i.e. reflecting long-term reactivity of fly ash.

It is also apparent that the Activity Index values for the fly ashes ranged from between 80 and 94% of the PC control mortar at 28 days, to 93 and 110% at 90 days and hence all met the 75 and 85% requirements for this at these ages in BS EN 450-1.

FLY ASH CHARACTERISTICS AS INDICATORS OF REACTIVITY

Fineness

The fly ash fineness results, measured using a 45 µm sieve (wet sieving), as described in BS EN 451-2[27] are shown in Figure 1. In line with previous research,[12] the data indicate that with reductions in fineness (increasing sieve retention), there were corresponding changes in Activity Index. It is apparent that similar behaviour occurred between properties at both test ages and in terms of the correlation coefficients obtained. Given the relationships, it appears that the measurement of fly ash fineness provides a good indication of the materials' contribution in a cementitious system.

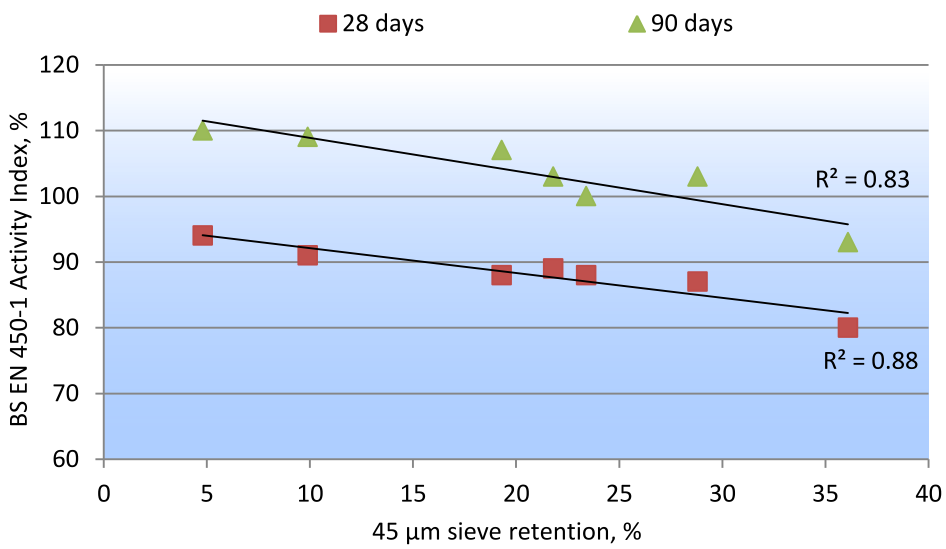

Figure 1. Relationship between 45 µm sieve retention and BS EN 450-1 Activity Index of fly ashes

To further examine this aspect of the material as a means of assessing reactivity, tests were also made using a LASER particle size analyser (Malvern Mastersizer 2000). The results in terms of the median particle size (PSD d_{50}) and sub 10 µm level (considered most reactive[28]) of the fly ashes are given in Figure 2.

These generally follow the trends noted above for the 45 µm sieve retention tests, with lower Activity Index values corresponding to increasing PSD d_{50} and reducing sub 10 µm level of fly ash. The rate of change in Activity Index with respect to these parameters was also similar for the two test ages. A comparison of correlation coefficients between the measures of fineness indicates that these were highest for PSD d_{50}.

Consideration was also given to the multiple factor, defined in ASTM C618 as the product of LOI and fineness (45 µm sieve retention). The relationship for the data from these two tests is given in Figure 3. This indicates that the parameter gave similar trends and coefficients of correlation, with respect to Activity Index, to those noted for the 45 µm sieve retention test alone.

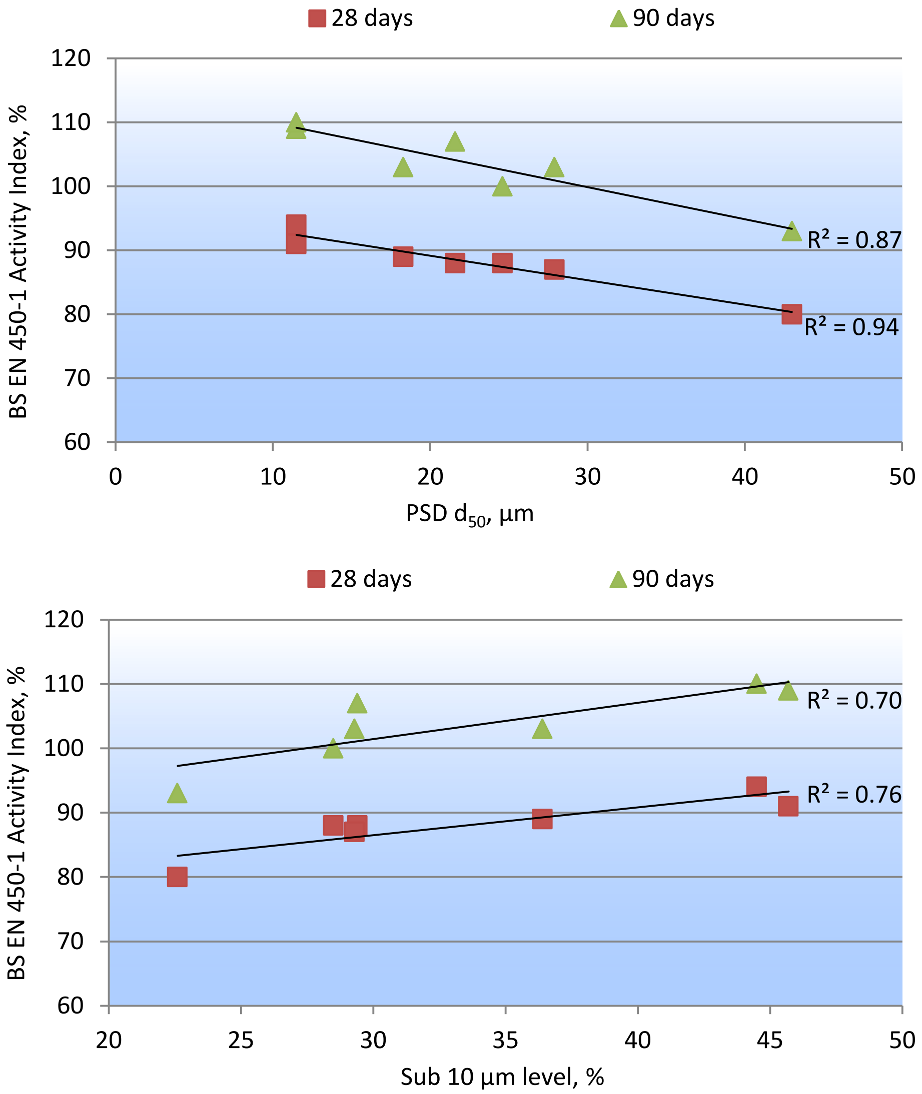

Figure 2. Relationship between PSD d_{50} (top) and sub 10 µm level (bottom), and BS EN 450-1 Activity Index of fly ashes

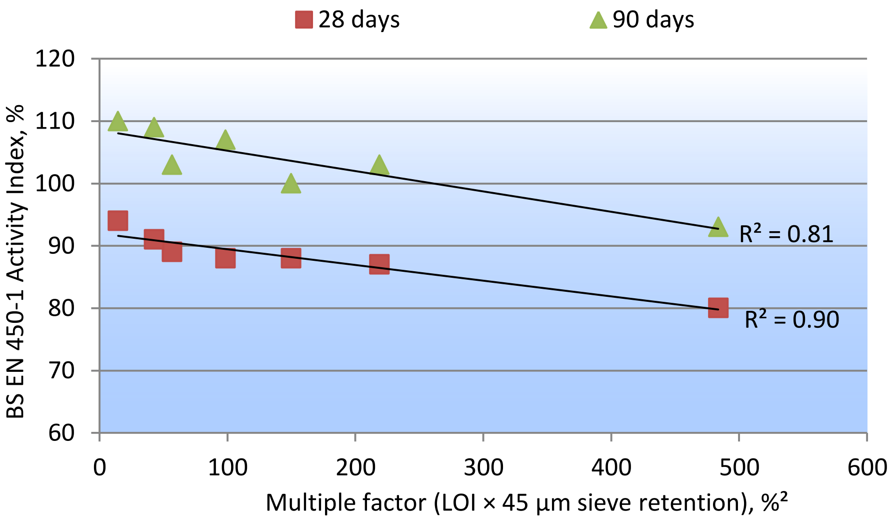

Figure 3. Relationship between multiple factor and BS EN 450-1 Activity Index of fly ashes

Chemical composition

BS EN 450-1 includes a limit for the sum of the main oxides, i.e. SiO_2, Al_2O_3 and Fe_2O_3, which should be at least 70%. A similar requirement is given in ASTM C618 for Class F fly ashes. The sum of these oxides for the test fly ashes, obtained using X-ray fluorescence spectroscopy (XRF), was therefore considered against Activity Index at 28 and 90 days.

The results are given in Figure 4 and indicate that the sum of the main oxides for the fly ashes considered, all exceeded the 70% limit referred to above and the Activity Index tended to increase with respect to this. It was also noted that finer fly ashes, from Figures 1 and 2, generally corresponded to higher values for the parameter. However, it is apparent that the correlations for the sum of the main oxides and Activity Index were poorer than those obtained with fly ash fineness, particularly at 90 days.

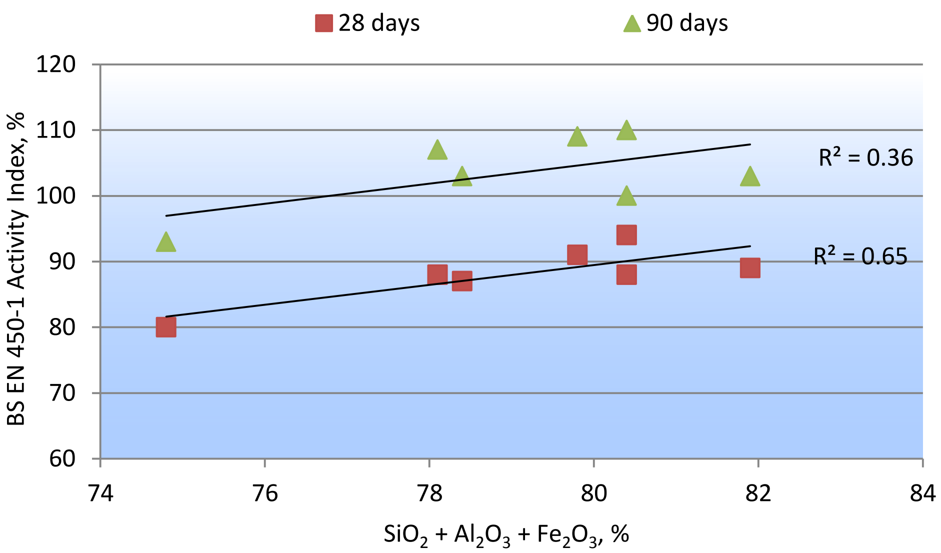

Figure 4. Relationship between sum of the main oxides and BS EN 450-1 Activity Index of fly ashes

As the reactive component of fly ash corresponds to the amorphous/glassy phase, consideration was given to quantifying the level of this in the material as a means of assessing reactivity. X-ray diffraction analysis of the fly ashes was carried out and the crystalline components quantified, and the portion other than these and the LOI (by subtracting from the total material) taken as an estimate of the reactive content (amorphous/others).

The correlation of this with Activity Index, given in Figure 5, was poor at both 28 and 90 days. The findings are similar to those of earlier work carried out in the UK,[29] which also attempted to relate the properties. Tests using alternative means of measuring the glass content[30] were considered, but did not significantly change the relationships.

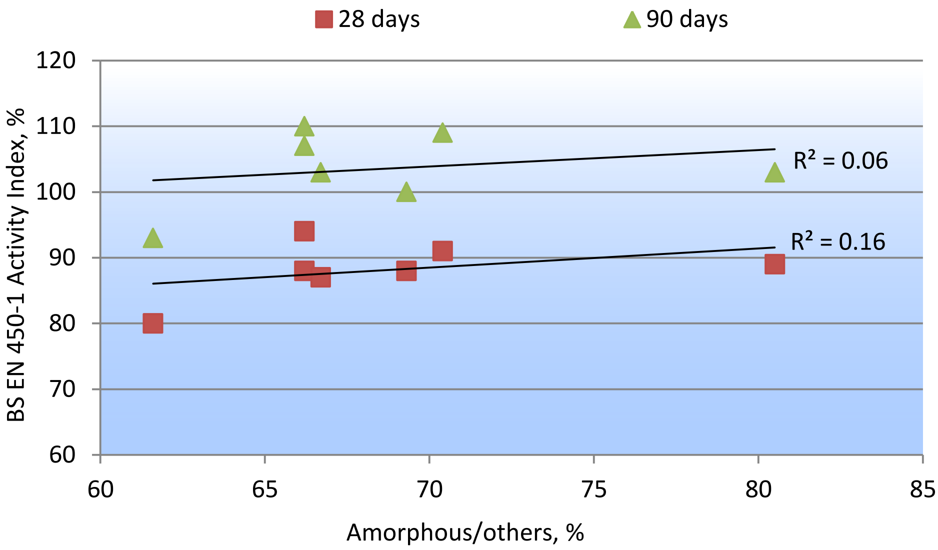

Figure 5. Relationship between amorphous / others and BS EN 450-1 Activity index of fly ashes

ACCELERATED CURING AT ELEVATED TEMPERATURE

Activity Index

The 1982 version of BS 3892-1,[31] the former UK standard for fly ash (Pulverized-Fuel Ash), included an accelerated curing method for assessing reactivity (fineness and LOI limits in the standard were 12.5% retained on a 45 µm sieve and 7.0%). The property, referred to as pozzolanic activity index (PAI), used mortar with 30% fly ash in cement and of equal flow to the control PC mortar (w/c = 0.5, against which comparisons of compressive strength were made). The curing regime followed was, 20°C in moist air for the first day, then water curing at 20°C for 4 days, followed by 50°C for 46 hours, and finally 20°C for 2 hours before testing.

The influence of elevated temperature curing on fly ash reactions in concrete has been used to good effect in precast manufacture[6] and this approach was followed in the current study. The results for Activity Index (with BS EN 450-1 mix proportions) following the curing conditions of BS 3892-1 (1982) are compared with those using BS EN 450-1 curing in Figure 6. For the range of fly ashes tested, similar results were obtained for the high LOI/coarse fly ashes, however, increases of about 10 to 15% in Activity Index were noted with accelerated curing for fine fly ashes, reflecting the effect of these conditions and material properties on reactivity.

A comparison of the relationships for the Activity Index values between the two curing conditions indicates that similar rates of change between these were obtained at both 28 and 90 days, with good correlations also noted. The results agree with other research carried out on fly ash mortar,[32] which suggests that temperatures of 40 or 60°C could be used to evaluate pozzolanic activity.

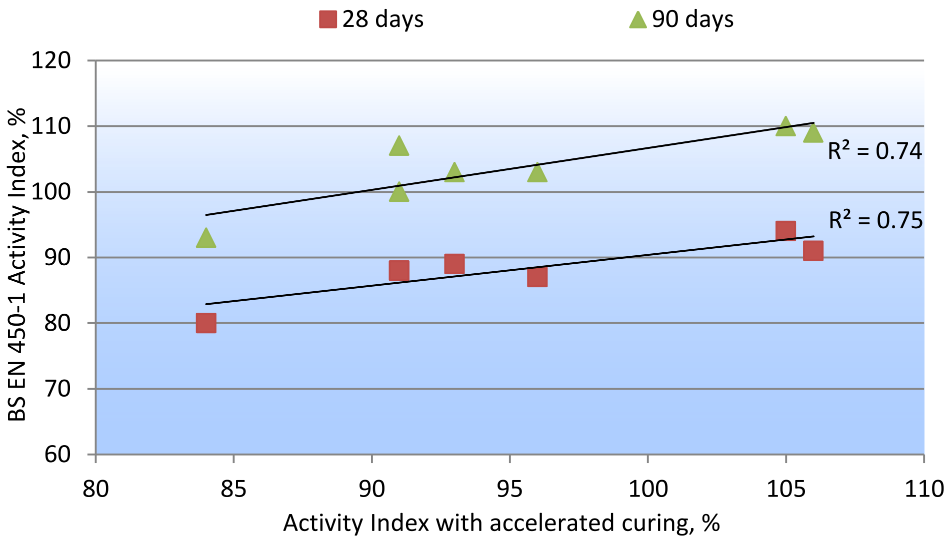

Figure 6. Relationship between Activity Index by accelerated curing and BS EN 450-1 Activity Index of fly ashes

Hydrated lime mortar strength

Given the nature of the reactions involved, attempts were made to examine the potential of using hydrated lime in the Activity Index mortar test instead of PC to assess fly ash. Following preliminary tests, based on ASTM C593[33] (used in lime-stabilization), suitable mixes were established, see Table 3. As indicated, these had their water contents adjusted to give equal flow. Following sample preparation (75 mm cubes), curing was carried out in the moulds/sealed container above water at 50°C until compressive strength tests at 14 days.

Table 3. Mix proportions of hydrated lime mortar

MATERIALS	QUANTITY	SOURCE/TYPE
Hydrated lime, g	150	Lab. supplier
Fly ash, g	300	-
Standard sand, g	1350	CEN
Free water, ml	to achieve flow of 215 ± 5 mm	(BS 4551-1)

Note: Quantity for 2 cubes of 75 mm size

A comparison of the results between the lime mortar strength and Activity Index is shown in Figure 7. This indicates that there was a slightly better correlation at the earlier test age. Clearly, the mixes are proportioned differently between tests in terms of the quantities of PC/lime used and the fixed/variable water contents. Indeed, improved correlations were noted when comparisons were made with PC/fly ash mortars (from PAI tests; variable water content/equal flow) at 7 days using BS 3892-1 (1982) accelerated curing conditions. The results suggest potential, but further work would be needed to refine the test.

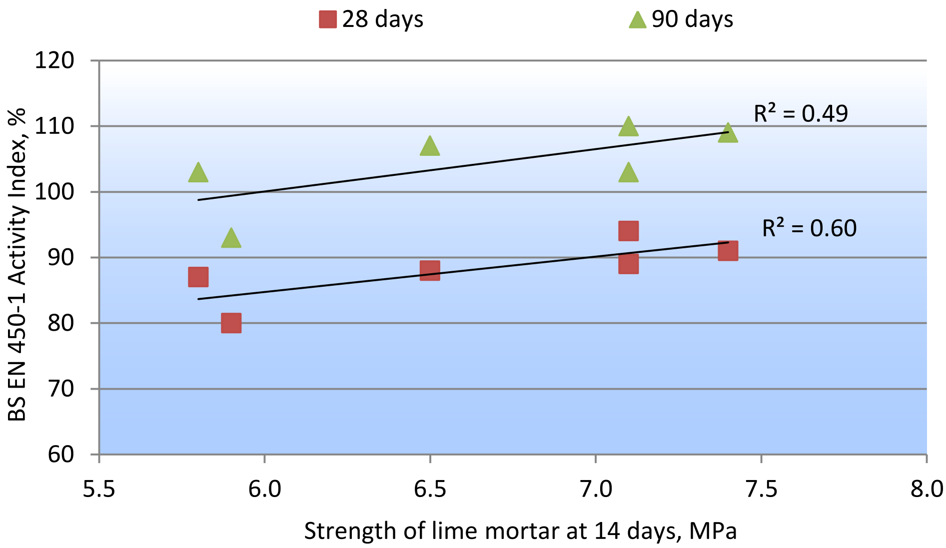

Figure 7. Relationship between strength of lime mortar and BS EN 450-1 Activity Index of fly ashes

WET CHEMICAL TESTS INVOLVING LIME-CONSUMPTION (BS EN 196-5)

A test method is described in BS EN 196-5[34] for determining the pozzolanicity of pozzolanic cements. This uses a slurry of the test (pozzolanic) cement in water, with the $Ca(OH)_2$ level in solution determined following a fixed contact period (8 or 15 days). This approach was considered in the study to assess fly ash reactivity.

To match BS EN 450-1 Activity Index test mix proportions, a fly ash level of 25% was used with PC. Details of the material quantities and the test conditions adopted are given in Table 4. Shaking of the slurry was carried out and it was stored as indicated. Following the contact period, this was filtered and the remaining CaO and OH^- concentrations measured by XRF and titration.

Table 4. Lime-consumption test details

MATERIALS/CONDITIONS	QUANTITY
PC, g	15
Fly ash, g	5
De-ionized water, ml	100
Test temperature, °C	40
Contact period, days	8

The results from the tests are given in Figure 8 and indicate that the Activity Index increased with CaO consumption after 8 days. The trends and correlations between properties were similar at both 28 and 90 days, with $R^2 > 0.7$. General agreement between fly ash fineness and lime-consumption was noted. However, differences from the relationships in Figure 8 were found for coarse/high LOI fly ash (not shown), which may relate to physical packing effects with this material in mortar (influencing activity index), that do not apply to the lime-consumption test.

Related research suggests that consumption of $Ca(OH)_2$ during the BS EN 196-5 test at a high level after 8 days indicates that there is sufficient reaction between pozzolanic materials and pore solution $Ca(OH)_2$ to form strength contributing products, as noted in 28 days Strength Activity Index tests.[15] In the current study, the test appears to be sensitive enough to detect differences between fly ashes and could provide a means of assessing their reactivity.

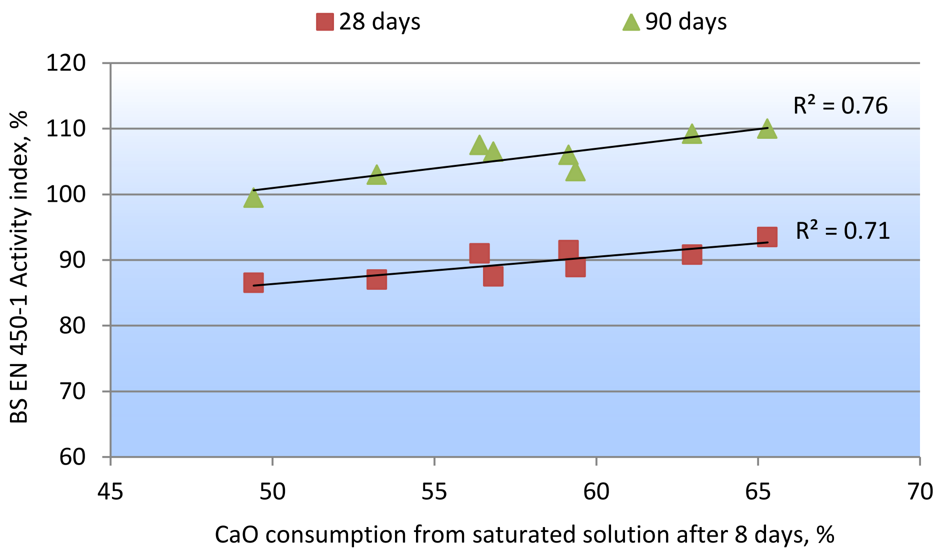

Figure 8. Relationship between CaO consumed during the BS EN 196-5 test and BS EN 450-1 Activity Index of fly ashes

POTENTIAL OF TEST METHODS FOR FLY ASH REACTIVITY ASSESSMENT

The paper considers results from tests carried out to investigate techniques for more rapidly assessing fly ash reactivity than those of the cement mortar-type Activity Index, which can take up to 90 days. Following a review of the literature, exploring test options, the research described focused on (i) fly ash characteristics, (ii) accelerated curing conditions and (iii) wet chemical lime-consumption tests, as methods of achieving this.

A summary of the results for the different tests in terms of their correlations with 90 days Activity Index and an indication of the timescales involved is shown in Table 5. This suggests that the best methods of assessing reactivity were those measuring fly ash fineness, either by wet sieving or particle size analysis.

In order to further explore the methods for fly ash reactivity assessment, compressive strength tests were made on 100 mm concrete cubes. These used the same PC and 5 to 6 fly ashes described above, a w/c ratio of 0.5, 30% fly ash in cement, and water-curing (20°C). Correlation coefficients obtained for Activity Index and concrete strength at 90 days, with each of the three methods considered, indicate general agreement between them (Figure 9).

Clearly the results reported in the paper are from a relatively small number of fly ashes. Research to broaden the database is on-going covering materials with a wider range of properties. The outcome of this will be reported in due course.

Table 5. Correlations between rapid assessment methods and BS EN 450-1 Activity Index values (90 days) and comparison of their time requirements

METHOD	PROPERTY/TEST	CORRELATION (R^2) WITH ACTIVITY INDEX	DURATION OF TEST
Fly ash characteristics	PSD d_{50}	> 0.8	5 minutes
	Sub 10 µm level	> 0.7	5 minutes
	45 µm sieve retention	> 0.8	1 day
	Multiple factor	> 0.8	1 day
	$SiO_2 + Al_2O_3 + Fe_2O_3$	< 0.4	1 hour
	Amorphous/others	< 0.1	2.5 hours
Accelerated curing	Activity Index	> 0.7	7 days
	Hydrated lime mortar strength	< 0.5	14 days
Lime-consumption	BS EN 196-5	> 0.7	8 days

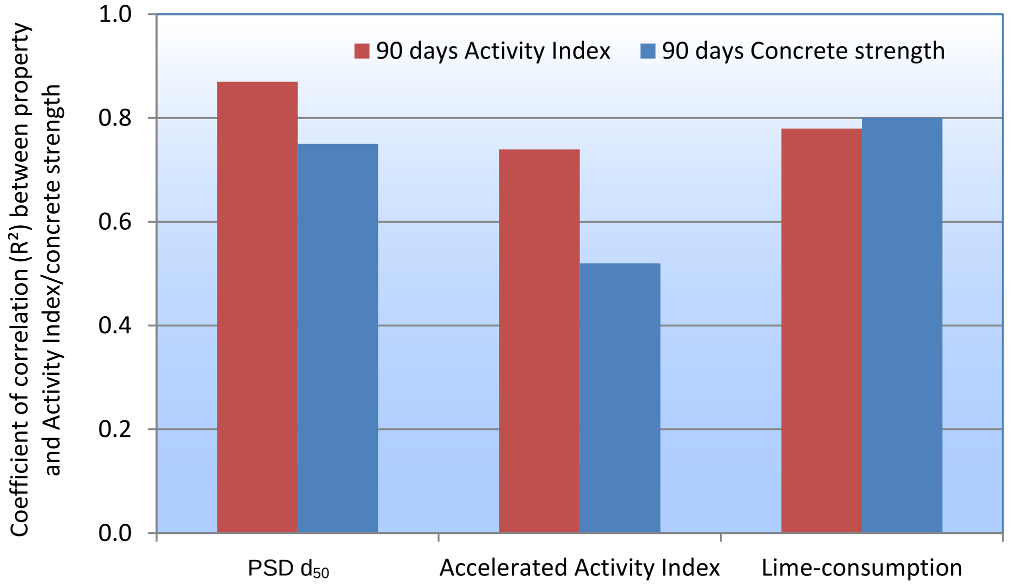

Figure 9. Comparison of correlations between rapid assessment methods and 90 days BS EN 450-1 Activity Index values/concrete compressive strengths

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