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FAST PHYSICS-CHEMICAL CHARACTERIZATION OF FLY ASH

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This paper analyzes the effect of fly ash chemical character on early Portland cement hydration and the possible adverse effects generated by the addition of gypsum. Behaviour was analyzed for pure Portland cements with varying mineralogical compositions and two types of fly ash, likewise differing in chemical composition, which were previously characterized under sulphate attack as: silicic–ferric–aluminic or aluminic–silicic ash in chemical character, irrespective if they are in nature, siliceous or siliceous and aluminous materials according to the ASTM C 618-94a.

The experimental results showed that water demand for paste with a normal consistency increased with the replacement ratio in fly ash with a more aluminic than silicic chemical character, whereas it declined when silicic-ferric-aluminic ash was used. On the other hand, the differences between the total heat of hydration released at the first valley and the second peak also clearly differentiated the two types of ash. While the relative differences increased in the more aluminic than silicic ash, they declined in the more silicic than aluminic. In another vein, the findings indicate that within a comparable Blaine fineness range, the reactive alumina ($Al_2O_3^{r-}$) content in pozzolanic additions has a greater effect on mortar strength than the reactive silica (SiO_2^{r-}) content, at least in early ages up to 28 days. Finally, the adverse effect generated in the presence of excess gypsum is due primarily to the chemical interaction between the gypsum and the C_3A in the Portland cement and the reactive alumina ($Al_2O_3^{r-}$) in the fly ash.

Keywords: *conduction calorimetry, fly ash, gypsum, heat of hydration, Portland cement*

Introduction

Extensive research [1] has been conducted to identify the benefits afforded by adding fly ash to Portland cement in terms of the performance of the resulting concrete when exposed to different aggressive media such as sulphates, chlorides, seawater, carbonation, alkali-aggregate reaction and so forth.

The chemical character of natural and artificial pozzolans is determined by their behaviour when exposed to such aggressive media. Whereas those with a primarily aluminic chemical character are more chloride resistant [2–4], more silicic pozzolans perform better in sulphate environments [5, 6] (with the exception of silica fume and nanosilica which perform well in both types of aggressive media due to their small particle size and very active early age pozzolanicity [7, 8]), and vice-versa [2–6].

Fly ash normally exhibits a random mix of the two above extreme types of chemical character. Regardless of fly ash origin [9], the silicic or the aluminic chemical character may prevail, which is not incompatible in some of them, whether natural or artificial, with the existence of a substantial ferric content [5, 10].

The wide range of chemical character types defining fly ash translates into an equally broad range of fly ash behaviour, with some providing effective chloride protection for the resulting concrete and reinforcing steel [2–4], while others aggravate these adverse effects [2–4]. The contrary behaviour has been observed with respect to sulphate attack, however [5, 6].

Fly ash chemical character is determined with more or less accelerated durability tests, which call for a response time of at least 28 days (depending on fly ash reactivity and media aggressiveness) [11]. The age at which results are obtained also depends on the type of Portland cement chosen for the trials. That notwithstanding, recent research has been conducted in which ash chemical character has been determined in a much shorter period of time: only 2 days [7].

In the wake of such research, the present paper reports the early age differences and similarities in the behaviour of fly ash with differing chemical character when added to Portland cement. The same experiments were conducted in the presence of excess gypsum to highlight the behavioural differences.

Objectives

The objectives of the present study were, then, to show the similarities and differences in the behaviour of Portland cement containing fly ash with different chemical character types, using standardized, inexpensive and short-term analysis and tests, and to identify the effect of an excess of gypsum on the results.

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A second purpose was to show the suitability of some of such analysis and tests for the speedy characterization of fly ash and other natural or artificial pozzalanic additions to be able to determine their chemical character in a very short period of time (≤2 days) and thereby deduce the most likely behaviour of the resulting concrete and/or cement mortar when exposed to an aggressive (sulphates, chlorides, seawater and so on) or undefined environment.

Experimental

Materials and methods

Two fly ashes with different chemical character types (determined a priori via accelerated tests involving sulphate [11] and chloride [2–4] attacks on cement pastes and mortars) were selected for the study: a silicic–ferric–aluminic ash (FA_{SFA}) with a density of 2.43 and a Blaine specific surface (BSS) value of 399 $m^2 kg^{-1}$ and an aluminic–silicic ash (FA_{AS}) with a density of 2.09 and a BSS of 401 $m^2 kg^{-1}$. The chemical composition of the two ashes is given in Table 1.

The two Portland cements with which each of the ashes was mixed were: PC1, with a high C_3A content, 51% C_3S, 16.5% C_2S, 4% C_3A and 5.5% C_4AF, a density of 3.07 and a BSS of 319 $m^2 kg^{-1}$ and the other, PC2, with a minimum C_3A content, high C_3S content, 79.5% C_3S, 2.5% C_2S, ≈0% C_3A and 10% C_4AF, a density of 3.21 and a BSS of 329 $m^2 kg^{-1}$.

The materials were chosen to cover the wide range of mineralogical compositions found in Portland cement and the variations in the possible chemical character of fly ash. The replacement ratios used were 20 and 40% ash. These blended cements were also analyzed with and without excess gypsum with up to 7.0% SO_3 to enhance the behavioural differences between the two ashes. A 100/00 ratio denotes the two pure Portland cements used as a control, while 8 POZC with 80/20 and 60/40 (%PC–*n*/%FA–*n* ratios, respectively) were prepared with 2 Portland cements (PC) – PC1 and PC2 – and two fly ashes – FA_{SFA} and FA_{AS}. Finally, DRX analysis of pastes with metakaolin, M, gypsum and PC1, were needed as well (Fig. 1).

The pre-tests used for the (48-h) early age determinations were: water demand for normal consistency paste and setting times [12]; conduction calorimetry during the first 48 h of hydration, X-ray diffraction analysis to identify the hydrated and/or most characteristics anhydrous compounds present by that age (ettringite, gypsum and C_3A) and final test age conduction calorimetry. The XRD analyses were semi-quantitative or comparative and were

Table 1 Chemical compositions of the two fly ashes and of the metakaolin

Pozzolanic additions	LOI/%	IR/%	SiO_2/%	Al_2O_3/%	Fe_2O_3/%	CaO/%	MgO/%	Na_2O/%	K_2O/%	SO_3/%
FA_{SFA}	0.83	0.12	45.47	30.09	16.12	4.03	1.12	0.20	2.80	1.92
FA_{AS}	6.92	0.18	49.29	29.63	4.22	2.86	1.81	0.50	3.60	0.94
M	0.40	–	57.48	41.55	0.50	0.01	0.00	–	–	0.00

LOI – Lost of ignition, IR – Insoluble residue

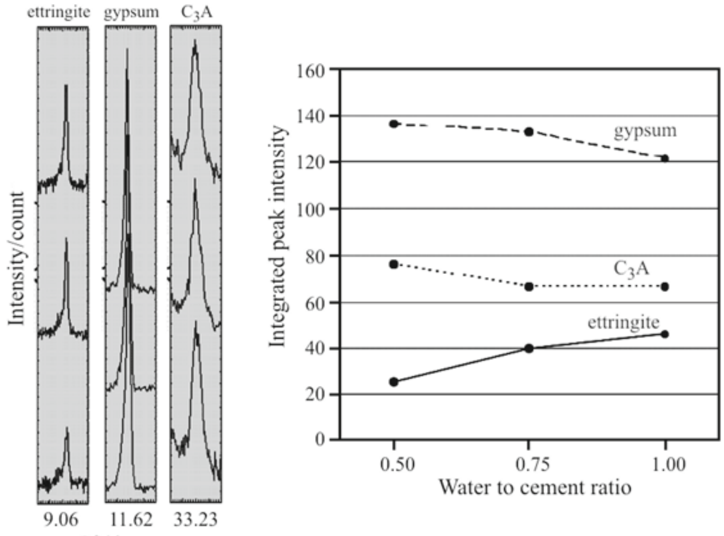

Fig. 1 Semi-quantitative analysis of ettringite, gypsum and C_3A in blended cement PC1/M 60/40 with water to cement ratio 0.5, 0.75 and 1.0

conducted on three pastes made with PC1/M 60/40 blended cement containing 7.0% SO_3 (≈15.05% gypsum, $CaSO_4{\cdot}2H_2O$) and having water/binder ratios (w/b) of 0.50, 0.75 and 1.00, because the analyses of the two fly ash pastes themselves were not sufficiently significant [10] to verify the explanation for the disparate behaviour they exhibited in some of the trials, as discussed below. The above PC1/M 60/40 blended cement was prepared with high grade (≥98%) metakaolin, M having a density of 2.52, a BSS of 333 $m^2\ kg^{-1}$ and the chemical composition shown in Table 1. This compound was, then, what Talero defines to be an artificial, clearly aluminic pozzolan [5, 11, 13], which had also been analyzed and characterized previously via heat of hydration, with and without excess (15.05%) gypsum [7, 14–16].

Lastly, the 2-, 7- and 28-day Frattini [17] pozzolanicity test results for the two ashes are given in [10] and the hydraulicity (=pozzolanic activity index (PAI) [18]) data in Table 3.

Results and discussion

Table 2 gives the setting times for the two Portland cements, with and without fly ash. Table 2 shows that in general, initial and final setting times for both PC1 and PC2 blended cements lengthened with increasing replacement ratios; this trend was also observed for blended cements containing 7.0% SO_3. These findings revealed no differences in the chemical character of the two fly ashes. This information could be deduced, however, from the water demand for normal consistency pastes, shown as the water/binder ratio in Table 2. Indeed, in both Portland cements, with or without 7.0% SO_3, the water demand declined with increasing replacement ratios of silicic–ferric–aluminic ash and rose in all cases with higher aluminic–silicic ash ratios.

This latter behaviour therefore constitutes clear evidence of the very substantial difference in the chemical character of the two ashes, inasmuch as their reactive alumina content, $Al_2O_3^{r-}$ (determined using the Florentino method [19]) also varied significantly. In FA_{AS} it was 13.5% and in FA_{SFA} 5.0% (i.e., 2.7 times greater in FA_{AS} than in FA_{SFA}), while their reactive silica content, SiO_2^{r-} [20] was similar (38.0 and 37.0%, respectively). Consequently, the different $Al_2O_3^{r-}$ content in the two fly ashes, FA, is not only largely responsible for their different chemical character, but also and in particular for their different behaviour in terms of the water demand required to make normal consistency cement paste. The reaction between reactive alumina, $Al_2O_3^{r-}$ and portlandite yields hydrated calcium aluminates as well, but in the presence of gypsum, of hydrated phases of AFt or ettringite ($C_3A{\cdot}3CaSO_4{\cdot}31H_2O$) and AFm ($C_3A{\cdot}CaSO_4{\cdot}12H_2O$) [10] (Fig. 1), via a process known to require 31 and 12 water molecules, respectively. Logically, then, while the water/binder ratio for the FA_{AS} blended cement family increased, it declined for the respective FA_{SFA} family, as Table 2 shows.

Verification of the above explanation is given in Fig. 1, which shows that the amount of ettringite formed in the 48-h pastes increased with the quantity of mixing water used in cement mix P1/M 60/40 (M had an $Al_2O_3^{r-}$ content of 29.0% and a SiO_2^{r-} content of 48.0%), while the amounts of the chemical reagents from which it formed, gypsum and the C_3A in PC1 (among others), declined.

Much the same can be said of the PAI [18] for the two fly ashes, FA_{SFA} and FA_{AS}, and the metakaolin, M. From the values obtained, it may be safely asserted that the PAI appears to be much more closely related to the $Al_2O_3^{r-}$ than to the SiO_2^{r-} content (Table 3). This is hardly surprising, for the molecular volume of both the hydrated calcium aluminates and/or sulphoaluminates, of pozzolanic origin in this case, is high or even very high, and always higher than the volume of the respective CSH gels [21].

Table 2 Times of setting

Cement	Without 7.0% SO_3			With 7.0% SO_3		
	Time of setting (h:m)		water/binder ratio	Time of setting (h:m)		water/binder ratio
	initial	final		initial	final	
PC1 100/00	3:20	5:10	0.310	3:55	6:15	0.320
PC1/FA_{SFA} 80/20	2:45	4:45	0.300	2:45	4:30	0.305
PC1/FA_{SFA} 60/40	3:10	5:10	0.275	3:10	5:00	0.280
PC1/FA_{AS} 80/20	2:55	4:40	0.330	3:10	4:40	0.340
PC1/FA_{AS} 60/40	3:05	4:55	0.345	3:15	4:45	0.350
PC2 100/00	4:30	6:15	0.280	4:15	6:10	0.280
PC2/FA_{SFA} 80/20	3:10	7:30	0.285	4:20	8:05	0.280
PC2/FA_{SFA} 60/40	3:40	7:50	0.275	5:00	7:50	0.270
PC2/FA_{AS} 80/20	3:35	8:00	0.320	5:30	7:45	0.300
PC2/FA_{AS} 60/40	6:05	8:20	0.340	6:00	8:18	0.320

Table 3 Pozzolanic activity index, PAI

Pozzolanic addition	PAI*/%	Reactive alumina/%	Reactive silica/%
FA_{SFA}	75.0	5.0	37.0
FA_{AS}	87.3	13.5	38.0
M	110.0	29.0	48.0

*According to ASTM C 618-94 [6], this PAI has to be ≥75% at 28 days-old for what the mineral addition tested can be regarded as being pozzolanic

The conduction calorimetry test, in turn, was run on both the pure Portland cements and each fly ash blend at a temperature of 25°C on pastes made in all cases with a water/binder ratio of 0.50. The characteristic points on the rate of heat release curves (Figs 2a and b) are the first, second and third peaks (maximum) and the first, second and third valleys (minimum) for PC1 and its blends and the first and second peaks and first and second valleys for PC2 and its blends. Given the features of the calorimetry test, the data for the first peak were disregarded. For the intents and purposes of standardization, therefore, the analysis covered only the data for the first valley and the second peak, which were present in all the cements analyzed.

Table 4 gives the ages and intensities of these characteristic points for PC1, PC2 and their respective blends, with and without 7.0% SO_3, while Fig. 2 provides a more detailed view of the points (peaks and valleys) for the two Portland cements.

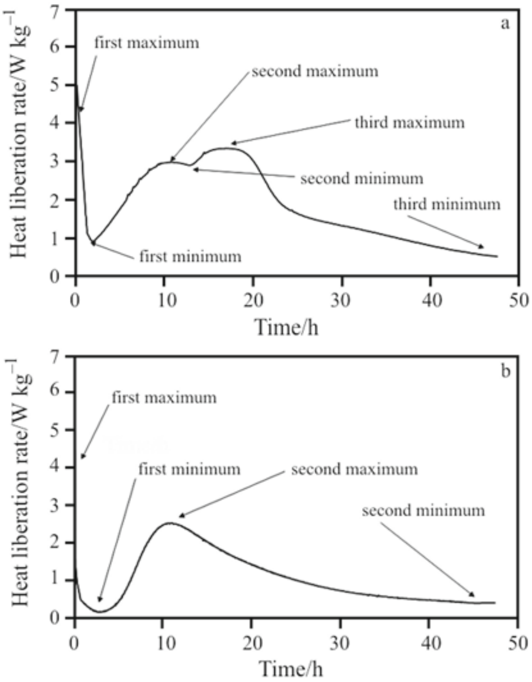

Fig. 2 Calorimetric curves of both plain Portland cements and their blends with each FA: a – PC1, b – PC2 (further details in Table 4)

The times at which the first valley and second peak were recorded increased with the replacement ratio for both Portland cements, with and without 7.0% SO_3. In other words, the behaviour observed followed a pattern similar to the setting times dis-

Table 4 Calorimetric parameters

Cement without 7.0% SO_3	First minimum		Second maximum	
	h:m	W kg^{-1}	h:m	W kg^{-1}
PC1 100/00	2:00	0.91	11:12	2.97
PC1/FA_{SFA} 80/20	1:45	0.64	9:43	2.66
PC1/FA_{SFA} 60/40	2:18	0.51	11:16	1.90
PC1/FA_{AS} 80/20	1:45	0.53	9:41	2.74
PC1/FA_{AS} 60/40	2:18	0.16	11:05	0.62
PC2 100/00	1:37	0.64	7:21	3.25
PC2/FA_{SFA} 80/20	2:14	0.27	9:26	2.28
PC2/FA_{SFA} 60/40	2:20	0.10	13:58	1.19
PC2/FA_{AS} 80/20	2:34	0.16	10:38	2.28
PC2/FA_{AS} 60/40	3:26	0.12	17:10	0.49
Cement with 7.0% SO_3	First minimum		Second maximum	
PC1 100/00	1:44	0.59	10:54	2.15
PC1/FA_{SFA} 80/20	1:32	0.58	9:51	2.53
PC1/FA_{SFA} 60/40	1:41	0.46	10:00	1.89
PC1/FA_{AS} 80/20	1:37	0.53	9:51	2.57
PC1/FA_{AS} 60/40	2:10	0.32	11:20	1.54
PC2 100/00	3:14	0.17	11:12	2.51
PC2/FA_{SFA} 80/20	3:18	0.10	12:28	1.88
PC2/FA_{SFA} 60/40	3:40	0.06	14:22	1.10
PC2/FA_{AS} 80/20	3:35	0.07	12:59	2.00
PC2/FA_{AS} 60/40	3:38	0.04	13:10	0.81

Table 5 Heat released between first minimum and second maximum

Cement	Cement without 7.0% SO_3		Cement with 7.0% SO_3	
	heat/kJ kg^{-1}	% between cements	heat/kJ kg^{-1}	% between cements
PC1 100/0	71	–	44	–
PC1/FA_{SFA} 80/20	53	83.0	51	76.5
PC1/FA_{SFA} 60/40	44		39	
PC1/FA_{AS} 80/20	52	26.9	51	68.6
PC1/FA_{AS} 60/40	14		35	
PC2 100/0	54	–	40	–
PC2/FA_{SFA} 80/20	34	82.4	32	75.0
PC2/FA_{SFA} 60/40	28		24	
PC2/FA_{AS} 80/20	38	42.1	33	42.4
PC2/FA_{AS} 60/40	16		14	

cussed above, although in the case of conduction calorimetry, the water/binder ratio was maintained at a constant 0.5 for all the cements.

Table 4 also shows that the intensity of the first valley declined with increasing replacement rates in the blended cements with and without 7.0% SO_3, and all the values were lower than found for the respective controls. While the general pattern for the second peak was a decline in intensity with increasing replacement rates, in the PC1 samples containing 7.0% SO_3, the blends with lower replacement ratios exhibited higher values than the respective control.

That general pattern, i.e., declining heat release rate with rising replacement rate, is primarily an effect of the physical dilution taking place when Portland cement is replaced by fly ash [10]. The effect of dilution is countered, however, by the stimulation effect of the hydration to the PC fraction, by the fly ash with which it is mixed, but by 'direct way' or 'through way' or simply 'direct stimulation' [7, 22], promoted by the initial adsorbed water of each fly ash (from the mixing water), that is to say, by means of physical influence only, but not chemical [14–16]. As a result of these two calorimetrically conflicting effects, the PC1 blends containing a 20% fly ash replacement rate and 7.0% SO_3 reached higher second peak intensity values than the control.

Note that the above calorimetric parameters revealed no chemical character differences between the two fly ashes.

Another calorimetric parameter, the total heat released by a given age, can be found by integrating the respective rate of heat release curve. In this study, only the difference in the total heat released at the second peak and first valley (equivalent to the energy released in that period of time) was considered. Table 5 shows the differences, in absolute values and percentage, for all the cements tested.

In blended cements containing silicic–ferric–aluminic fly ash, the difference ranged from 83.0 to 82.4%, regardless of the Portland cement used, and declined (from 76.5 to 75.0%) in the presence of 7.0% SO_3. For aluminic–silicic cement blends, in turn, the difference ranged from 26.9 to 42.1% for the two Portland cements and from 68.6 to 42.4% for the cements containing 7.0% SO_3.

These findings constitute another sign of the chemical character difference between the two fly ashes, which is also visible in the absence of 7.0% SO_3: the decline observed in blended cements containing FA_{SFA} and the increase recorded for cements containing FA_{AS} must be attributed to the lower and higher reactive alumina ($Al_2O_3^{r-}$) content (5.0 and 13.5%, respectively) in the ashes.

When 7.0% SO_3 was added, the pattern observed in the FA_{SFA} blend without gypsum was maintained, whereas in the FA_{AS} blend cements, the pattern reversed. This finding, which provides further support for the chemical character difference in the two fly ashes, is due primarily to the gypsum-mediated stimulation of Portland cement hydration in general, and the synergies between the C_3A in PC1 and the reactive alumina, $Al_2O_3^{r-}$, especially in FA_{AS}, as reported in previous studies [23].

Conclusions

The conclusions that can be drawn from the tests conducted are as follows:

- The silicic–ferric–aluminic or aluminic–silicic chemical character of the fly ash was determined in a short period of time (≤ 2 days) based on the quantity of mixing water needed to produce paste with a normal consistency. The reactive alumina ($Al_2O_3^{r-}$) fraction in the fly ash reacts rapidly (and more so in the presence of gypsum) with the portlandite released during the hydration of the Portland cement with which it is mixed, requiring water in any event to form the different hydration products. This is the main reason why the water demand increased with rising replacement rates of FA_{AS} and declined with rising replacement rates of FA_{SFA}.

- The qualitative conduction calorimetry findings for cements with and without 7.0% SO_3 failed to distinguish the chemical character of the two types of fly ash. Quantitatively speaking, however, the differences found in the total heat of hydration released at the first valley and second peak, clearly differentiated the very different chemical character type of both fly ashes, and moreover, with the presence and absence of 7.0% SO_3 in their respective blend cements. This other conduction calorimetry analysis may, then, be used to characterize fly ash in a short period of time: 2 days at the most.
- Indeed, in the absence of 7.0% SO_3, silicic–aluminic fly ash exhibited an increase in relative differences and in silicic–ferric–aluminic fly ash these differences narrowed, whereas when 7.0% of SO_3 was added, the opposite behaviour was observed: i.e., the differences in FA_{AS} dropped and the FA_{SFA} differences rose.
- Within a comparable Blaine specific surface range, the findings indicate that the reactive alumina ($Al_2O_3^{r-}$) content in pozzolanic additions has a greater effect on mortar mechanical strengths (Table 3) than their reactive silica SiO_2^{r-} content, at least in early ages up to 28 days.
- Finally, the adverse effect generated in the presence of excess gypsum is due primarily to the chemical interaction between the gypsum and the C_3A in the Portland cement and the reactive alumina ($Al_2O_3^{r-}$) in the fly ash.

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