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Effects of w/c ratio and curing conditions on strength development in BRECEM concretes

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Abstract

The early strength development of 'BRECEM' concretes, made from 50:50 mixtures of calcium aluminate cement (CAC) and ground granulated blastfurnace slag (ggbs), using 100-mm cubes at w/c ratios of 0.35 and 0.45, has been studied. BRECEM concretes showed good strength development at both w/c ratios whether water cured or air cured. In contrast, longer-term studies with 1-m cubes cured under near-adiabatic conditions have indicated that mass BRECEM concretes of similar binder composition may gain compressive strength relatively slowly. This is primarily due to the heat of hydration of the CAC component, which can lead to temperatures of up to 60°C, resulting in rapid hydration and conversion of the calcium aluminate component. However, the performance of 40 CAC/60 ggbs concrete under these near-adiabatic conditions has proved to be satisfactory over a period of 5 years, with similar strength development to that of equivalent 100-mm cubes stored under ambient conditions.

Keywords: Calcium aluminate cement; Ground granulated blastfurnace slag; Compressive strength; Hydration; Long-term performance

1. Introduction

Calcium aluminate cement (CAC) has advantages over Portland cement (PC) through its rapid early strength development and excellent chemical resistance. However, concretes made from CAC have been found to lose strength with time, particularly in hot and humid environments through 'conversion'. In the conversion process, the initial crystalline products of hydration, the metastable calcium aluminate hydrates[2] CAH_{10} and C_2AH_8 are replaced by the stable calcium aluminate hydrate C_3AH_6 (hydrogarnet) and AH_3 [1]. The conversion reactions occur extremely slowly at 5°C but become more rapid as the temperature is increased and C_3AH_6 formation is almost immediate above 50°C. Conversion is accompanied by a decrease in the solid volume and therefore an increase in porosity that can lead to a loss in strength [1,2].

Cements in which CAC is blended with latently hydraulic materials such as ground granulated blastfurnace slag (ggbs) or pozzolanic materials such as pulverised fuel ash (pfa) and metakaolin have been widely studied [3–14]. Most of the work carried out so far has concentrated on mixtures of CAC with ggbs (with cement compositions in the range 60 CAC/40 ggbs–40 CAC/60 ggbs), which BRE has patented [4] and registered under the trade name BRECEM [13]. Concretes made by using BRECEM do not exhibit the loss of compressive strength that occurs in CAC concretes when kept at 38°C under wet conditions over a prolonged period of time. BRECEM concretes also perform well on storing in sulphate solutions and in aggressive marine and acid water environments [8,9].

The absence of strength loss in BRECEM concretes is a consequence of the modified chemistry on hydration caused by the high silica content of the ggbs addition [4,15]. The increased availability of silica leads to the formation of C_2ASH_8, known as strätlingite or gehlenite hydrate. As a result, the proportion of the mix made up of the calcium aluminate hydrates is much less than in CAC and so the effects of the conversion reaction on the strength of the concrete are greatly reduced.

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[1] Deceased 1996.

[2] Cement chemistry notation: C = CaO, A = Al_2O_3, S = SiO_2 H = H_2O, c = CO_2.

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The long-term performance of BRECEM concretes cured at ambient temperatures has been shown to be excellent on exposure to a wide range of aggressive environments [5–9,13]. The current paper addresses the following areas:

1. Early age strength development at low water/cement ratios, especially in the context of the potential use of this material in semi-dry applications such as concrete pipes.
2. The effect of early age temperature rise on the long-term properties of bulk concretes.

2. Early age strength development

One of the potential uses suggested for BRECEM is the manufacture of concrete pipes. Blended PCs are currently used in this process. However, the comparatively slow rate of strength development at early ages mean that the pipes need to be kept in a controlled environment for a certain period to gain adequate strength prior to their being moved for storage.

A trial to assess the suitability of BRECEM in this application and to determine whether BRECEM would permit a significant reduction in the time during which the units needed to be kept in a controlled environment has therefore been carried out. The trial was successful showing that BRECEM has a number of advantages over PC/pfa blends in this application (despite the fact that the mix design was based on that for a PC/pfa blend and not optimised for BRECEM). Its use allowed the trial pipe units to be removed from the pallets after 8 h rather than after 16 h as required for PC/pfa concretes. The strength development and permeability of the BRECEM concrete pipes were superior to those of typical PC/pfa units.

The trial highlighted the fact that there were a number of gaps in the BRE programme aimed at assessing the properties of BRECEM concretes. In particular, tests at low w/c ratios (lower than 0.45) have not previously been carried out. The early age strength development had also not been extensively studied. This report therefore describes a small experimental programme aimed at assessing the early age strength development of BRECEM concretes at total w/c ratios of 0.45 and 0.35. It is aimed at providing additional data to support the development of applications for BRECEM.

Table 1
Chemical analyses of CAC and ggbs used in the study

Oxide (wt.%)	CAC (Ciment Fondu)	ggbs (Cemsave)
CaO	38.9	41.2
Al_2O_3	39.1	12.1
Fe_2O_3	15.6	0.77
SiO_2	3.72	34.8
MgO	0.37	8.34
Na_2O	–	0.28
K_2O	–	0.29
TiO_2	1.68	0.59
BaO	–	0.07
Mn_2O_3	–	0.23
SrO	–	0.06
V_2O_5	–	0.02
Total SO_3 (after LOI and fusion)	–	~2.1

Table 2
Concrete mix proportions

	Concrete mix proportions (kg/m^3)			
Cement type	Aggregate (5–20 mm)	Sand (<5 mm)	Cement	Total w/s ratio
50:50 CAC+ggbs	2.91	1.75	1.0	0.35
50:50 CAC+ggbs	2.91	1.75	1.0	0.45

2.1. Experimental

The chemical compositions of the CAC (Ciment Fondu, supplied by Lafarge Special cements) and ggbs (Cemsave, supplied by the Frodingham Cement) used in this study are given in Table 1. Using a 50 CAC/50 ggbs BRECEM mix with w/c ratios of 0.35 and 0.45, 100-mm concrete cubes were made. The w/c ratio of 0.35 was the lowest value that could be reasonably used to give a workable mix without the use of specialised compaction equipment. Mix proportions are summarised in Table 2. Samples for testing at 4 and 6 h were removed from the moulds immediately prior to testing. The remaining cubes were demoulded after 24 h and kept under water at 20°C or in air at 20°C for periods of up to 90 days and tested for compressive strength. Compressive strength determinations were carried out on 100-mm cubes in accordance with BS1881: Part 116 [16] with three cubes tested at each age.

The hydrated phases present in the samples were identified by X-ray diffractometry. X-ray diffractometry was carried out by means of a Siemens D500 diffractometer using Cu Kα radiation and operating at 40 kV and 30 mA.

Table 3
Compressive strength development of BRECEM concretes (average of three cubes)

	Compressive strength (MPa)			
	w/c=0.35		w/c=0.45	
Test age	Water	Air	Water	Air
4 h[a]	14±1	14±1	8±3	8±3
6 h[a]	32±2	32±2	18±3	18±3
1 day[a]	48±2	48±2	36±1	36±1
3 days	52±1	56±3	39±1	45±3
7 days	59±1	62±1	45±3	49±4
14 days	69±3	69±2	51±1	52±1
28 days	78±2	68±3	63±1	51±1
90 days	85±4	70±1	73±1	50±2

[a] Moist air.

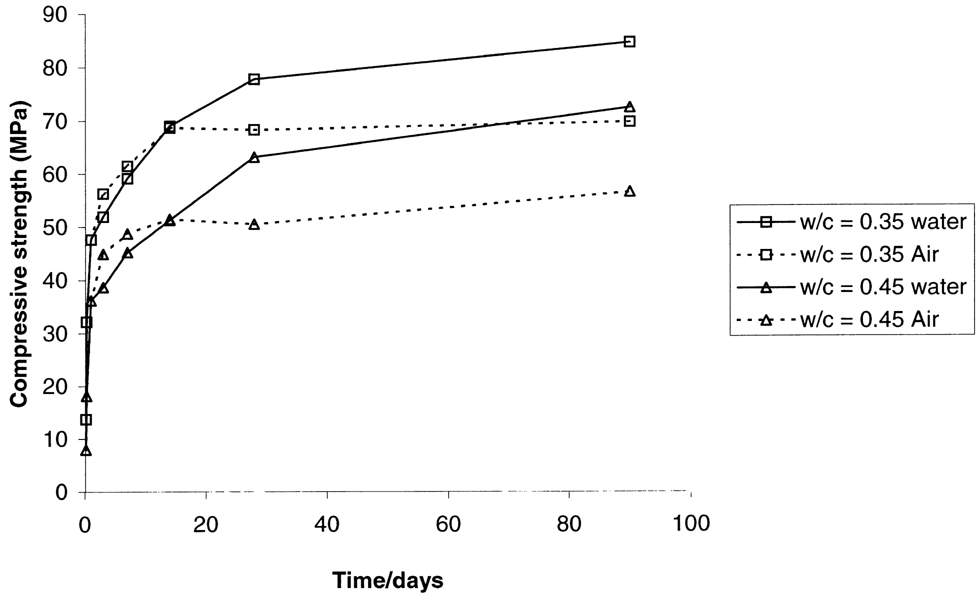

Fig. 1. Compressive strength development in BRECEM concretes with w/c ratios of 0.35 and 0.45 after storage in water and air at 20°C.

Data were accumulated over one scan of 2θ between 5° and 50°. Assignments of lines were made by comparisons with JCPDS files.

2.2. Results and discussion

The compressive strength development of BRECEM concretes made using w/c ratios of 0.35 and 0.45 is given in Table 3 and shown graphically in Fig. 1. Strength development data for CAC, PC, PC/pfa and PC/ggbs concretes (cured and stored under similar conditions to those of the BRECEM concretes), expressed as a percentage of their 28-day values, are also given in Table 4. Absolute values at each age cannot be compared and are not included here due to differences in the materials and mix designs used. The percentage data are intended to indicate, in general terms only, the rate of strength development of these materials.

The results show the following:

(1) BRECEM concretes made using a w/c ratio of 0.35 developed strength more rapidly than those made using a w/c ratio of 0.45. The 90-day strength was also higher than those made using a w/c ratio of 0.45 although, for water-stored concretes, strengths were still increasing in both cases.

(2) BRECEM concretes stored in air initially developed strength more rapidly than equivalent concretes stored in water. The initial strength development is predominantly due to the hydration of the more reactive CAC component. However, the compressive strength of air-stored concretes rapidly levelled out after 14 days and at later test ages water-

Table 4
Compressive strength development as a percentage of the 28-day strength

	Compressive strength as a percentage of the 28-day strength							
	BRECEM							
	w/c = 0.35>		w/c = 0.45		CAC	PC	70 PC/30 pfa	40 PC/60 ggbs
Test age	Water	Air	Water	Air	(w/c = 0.45, water)	(w/c = 0.54, water)	(w/c = 0.43, water)	(w/c = 0.50, water)
4 h[a]	17	20	13	16	–	–	–	–
6 h[a]	41	47	29	36	70	–	–	–
1 d[a]	61	69	57	71	82	27	22	27
3 days	66	83	61	89	96	53	52	–
7 days	76	90	72	96	101	73	73	
14 days	89	100	82	102	–	85	87	–
28 days	100	100	100	100	100	100	100	100
90 days	109	102	115	99	111	110	120	–

The CAC concrete [13] was made using a total w/c ratio of 0.45 and a cement content of 340 kg/m^3. The PC concrete [17] was prepared using a cement content of 350 kg/m^3 and a total w/c ratio of 0.54. The 70 PC/30 pfa concrete [17] was prepared using a total cementitious material content of 400 kg/m^3 and a total w/c ratio of 0.43. The 40 PC/60 ggbs concrete [18] was made using a total cementitious material content of 381 kg/m^3 and a w/c ratio of 0.50. All samples were demoulded after 24 h and stored in water until the test date.

[a] Moist air.

Table 5
Phases present in BRECEM concretes

Mix	Crystalline phases present at test ages (as indicated by XRD)					
	4 h	24 h	3 days	7 days	14 days	90 days
w/c = 0.35 (air stored)	CAH_{10} (s)	CAH_{10} (s) $C_4Ac_{0.5}H_{12}$ (tr)	CAH_{10} (s) C_2ASH_8 (tr) $C_4Ac_{0.5}H_{12}$ (tr)	CAH_{10} (s) C_2ASH_8 (tr)	CAH_{10} (s) C_2ASH_8 (tr) Cc	CAH_{10} (m) C_2ASH_8 (w) Cc, AH_3 (tr)
w/c = 0.35 (water stored)	CAH_{10} (s)	CAH_{10} (s) $C_4Ac_{0.5}H_{12}$ (tr)	CAH_{10} (s) $C_4Ac_{0.5}H_{12}$ (tr)	CAH_{10} (s) C_2ASH_8 (m) $C_4Ac_{0.5}H_{12}$ (tr)	CAH_{10} (s) C_2ASH_8 (s) Cc	CAH_{10} (w) C_2ASH_8 (s) Cc
w/c = 0.45 (air stored)	CAH_{10} (s)	CAH_{10} (s) $C_4Ac_{0.5}H_{12}$ (tr)	CAH_{10} (s) C_2ASH_8 (m) $C_4Ac_{0.5}H_{12}$ (tr)	CAH_{10} (s) C_2ASH_8 (tr)	CAH_{10} (m/w) C_2ASH_8 (s) Cc	CAH_{10} (w) C_2ASH_8 (s) H_3 (tr), Cc
w/c = 0.45 (water stored)	CAH_{10} (s)	CAH_{10} (s) $C_4Ac_{0.5}H_{12}$ (tr)	CAH_{10} (s) C_2ASH_8 (tr) $C_4Ac_{0.5}H_{12}$ (tr)	CAH_{10} (s) C_2ASH_8 (s) $C_4Ac_{0.5}H_{12}$ (tr)	CAH_{10} (tr) C_2ASH_8 (s) Cc	CAH_{10} (w) C_2ASH_8 (s) Cc, AH_3 (tr)

s = strong peak; m = medium peak; w = weak peak; tr = trace.

stored concretes had a higher compressive strength than air-stored ones. The continuing increase in compressive strength with time for water-stored samples after 14 days will have arisen due to the ongoing hydration of the ggbs and any remaining CAC.

(3) The rate of strength development of CAC concrete (see Table 4) was greater than that of BRECEM concrete during the early stages of hydration. This is due to the fact that the ggbs component in BRECEM concrete does not contribute to the hydration reactions significantly in the early stages of the hydration process. However, early strength development in BRECEM concretes is likely to be more rapid than that of PC, PC/pfa and PC/ggbs concretes [17,18].

(4) Phase development in the concretes prepared using w/c ratios of 0.35 and 0.45 are similar, with CAH_{10} forming as the main hydrate at test ages of up to 3 days. Strätlingite, C_2ASH_8, starts to form within 3 days of casting at both w/c ratios (Table 5).

(5) C_2ASH_8 was present in large amounts in water-stored concretes after 14 days and in the air-stored concrete made using a w/c ratio of 0.45. Previous studies [14] have shown that BRECEM concretes made using a w/c ratio of 0.45 are likely to show only a modest, if any, reduction in compressive strength when stored in water for up to at least 10 years. Long-term studies on concretes made using a w/c ratio of 0.35 have not been carried out. However, the fact that water-stored BRECEM concretes made using w/c ratios of 0.35 and 0.45 show comparable compressive strength and phase development up to 90 days suggests that concretes made using a w/c ratio of 0.35 may also retain their compressive strength in the long-term when stored in wet or moist conditions.

(6) C_2ASH_8 was still present only in small amounts after 90 days in the air-stored concrete made using a w/c ratio of 0.35. This may have arisen due to the limited availability of water for the ggbs component to continue to hydrate significantly after 14 days. If the ggbs component has not significantly hydrated, there may be implications for the long-term durability of the concrete. In particular, due to the large amount of CAH_{10} present, it may be susceptible to conversion as in CAC unless sufficient curing is provided.

3. Effects of early temperature rise on the long-term strength development

The adiabatic temperature rises of concretes made using a range of BRECEM mixes (60 CAC/40 ggbs, 50 CAC/50 ggbs and 40 CAC/60 ggbs) have been studied using 1-m cube moulds insulated with 100-mm-thick styrofoam. The aim of the programme was to study the effects of the early age temperature rise generated by the heat of hydration of the CAC component on the properties of the concrete. The concretes were poured in three layers and compacted by means of a vibrating poker. The temperatures at the centre and at one-quarter length from the edges were recorded using embedded sensors. The cubes were demoulded after 7 days and were stored externally in exposed conditions after 28 days. A series of 100-mm cores have been cut through each cube over a period of 5 years to allow the compressive strength and the cement hydrate composition to be assessed.

3.1. Experimental

Details of the mix designs used in the programme, together with the compressive strength and hydrated phase development, are summarised in Table 6. The 28-day compressive strengths of 100-mm cubes made using similar mix designs and cured under ambient conditions are included for comparison. Results up to test ages of 180 days have previously been published [8,13].

3.2. Results and discussion

The table shows that the rates of strength development in mass BRECEM concretes are generally slow in comparison with those of equivalent 100-mm cubes cured under

Table 6
Mix designs and properties of BRECEM metre cubes

Properties	CAC	BRECEM mixes (CAC/ggbs)			
		60:40	50:50	50:50	40:60
Cement content (kg/m^3)	390	390	390	320	390
w/c ratio	0.45	0.45	0.45	0.55	0.45
Maximum T (°C, see footnote)	91.2	67	(58.4) 63.6	58	(52.4) 61.6
Time to peak temperature	9 h	10 h	(15.5 h) 43 h	30 h	(18 h) 42 h
Compressive strength (MPa)					
8–9 days	40.8	25	31	19.5	38
29–32 days	36	26	34	21.0	41
180–190 days	36.9	47.5	36	24.5	47
1 year	41.8	32.4	45.2	33.0	53.6
5 years	–	32.1	38.0	33	55.0
28-day strength of equivalent 100-mm cube	75	58.5	47.5	No data	42
180-day strength of equivalent 100-mm cube	62	62	62	No data	51
Main phases in 100-mm cubes at 180 days (from XRD peak heights)					
C_3AH_6	Strong	Strong	Medium	Trace	Trace
C_2ASH_8	Not detected	Weak	Strong	Strong	Strong
AH_3	Strong (gibbsite)	Strong (gibbsite)	Trace	Not detected	Not detected
XRD peak height ratio (C_2ASH_8/C_3AH_6) (metre cubes 1 year)	–	0.03	2.7	2.5	7.1
XRD peak height ratio (C_2ASH_8/C_3AH_6) (metre cubes 5 years)	–	0.3	2.4	4.9	7.7

Times and temperatures given in brackets refer to a 'shoulder' in the temperature against time profile [8]. The temperature steadily increased to the second figure given.

ambient conditions. There was also little, if any, increase in compressive strength between 1 and 5 years. The results suggest that 60 CAC/40 ggbs and 50 CAC/50 ggbs BRECEM concretes may not be suitable for use in mass concretes although it should be noted that the near adiabatic conditions within the meter cubes represent a worst case. However, the strength development of a 1-m^3 concrete cube made using the 40 CAC/60 ggbs blend gained strength at a rate comparable to, although still lower than, that of 100-mm cubes made using the same concrete. The compressive strengths of cores taken from this 1-m^3 cube after 1 and 5 years were also significantly greater than those taken from otherwise equivalent 50 CAC/50 ggbs and 60 CAC/40 ggbs blends. The table also gives the ratio of XRD peak heights for C_2ASH_8 and C_3AH_6. These ratios are based on the 12.49 Å peak for C_2ASH_8 and the 5.1 Å peak for C_3AH_6 (although the actual d-spacing for this peak indicates that some Si may be included in the structure [19]). Although the analysis is not quantitative these ratios suggest that the proportion of C_2ASH_8 present at both 1 and 5 years increases with the amount of ggbs present. The trend is consistent with the strength development and suggests that the 40 CAC/60 ggbs concrete should have properties typical of 100-mm BRECEM cubes cured under ambient conditions.

The low compressive strengths of 1-m^3 BRECEM concretes in comparison with those of equivalent 100-mm cubes are probably a consequence of the rapid hydration and conversion of the CAC component as a result of the elevated temperatures attained in large volumes. At ambient or warm temperatures (up to about 40°C), CAC hydration results in the formation of the metastable hydrates CAH_{10} and/or C_2AH_8, together with AH_3. As the ggbs component hydrates, the availability of silica leads to the formation of C_2ASH_8 at the expense of at least some of the metastable hydrates. Any remaining metastable hydrate could undergo conversion to C_3AH_6, although the evidence suggests that the amount of CAH_{10} or C_2AH_8 undergoing this process is not sufficient to lead to a loss in strength.

The situation on hydrating BRECEM at higher temperatures (such as those reached during curing for mass concretes) is somewhat different from this. The CAC component hydrates rapidly in comparison with the ggbs. However, the metastable hydrates CAH_{10} and C_2AH_8 are either not formed or convert very rapidly to the stable phase C_3AH_6. This latter phase remains the main crystalline component even on cooling to ambient temperatures. It is less soluble than either of the metastable phases resulting in low concentrations of Ca^{2+}, $Al(OH)_4^-$ and OH^- in the pore solution. Experiments carried out at 60°C in the presence of excess water (and in which C_3AH_6 was the only calcium aluminate hydrate present) have shown that C_2ASH_8 forms very slowly under such conditions [19].

In a mass BRECEM concrete during the early stages of hydration the ggbs component may, therefore, be acting only as a diluent, effectively reducing the cement content and leading to the lower than expected compressive strength at early ages. If the reaction of the ggbs component and the consequent formation of C_2ASH_8 do not occur on cooling there may be adverse effects on the long-term durability of BRECEM concretes cured under these conditions. However, the phase composition and compressive strength development in 1-m^3 40 CAC/60 ggbs concrete shown in Table 6 suggest that mixes with a higher ggbs content may be suitable for use in mass concrete.

4. Conclusions

The strength development of 100-mm BRECEM concrete cubes at early ages and at low w/c ratios has been studied in the light of BRECEM's potential for use in 'semi-dry' applications such as concrete pipes. The results of the studies have shown that the early strength development of BRECEM concretes is significantly more rapid than that of PC and PC/pfa concretes although it is still appreciably slower than that of CAC. Lower w/c ratio BRECEM concrete showed good strength development and, as C_2ASH_8 was present at later ages, there appeared to be sufficient water available to allow the hydration of the ggbs component to proceed. However, the formation of C_2ASH_8 in air-stored concretes was slow at low w/c ratios, presumably due to the limited water available for ggbs hydration due to competition from the faster reacting CAC component. This could potentially affect the long-term properties of the concrete and may leave it prone to conversion if the concrete is not given sufficient curing in the early stages. Despite the potentially slow hydration of the ggbs component in air-stored concretes the results of the study together with other BRE data on the durability and chemical resistance of BRECEM concretes, show that BRECEM should be suited to use in 'semi-dry' applications.

The detailed studies that have been carried out on BRECEM concretes show that they perform extremely well in conditions other than those that may occur in mass concretes. BRECEM concretes prepared in large volumes may gain compressive strength very slowly due to temperature rises at early ages resulting in rapid hydration and conversion of the CAC component and the comparatively slow hydration of the ggbs component. However, the near-adiabatic conditions under which the concretes were cured must be regarded as a worst-case scenario and may not represent conditions that would actually occur in use even in mass concretes. The performance of 40 CAC/60 ggbs concretes even under these conditions has proved to be satisfactory, however, and has shown similar strength development to that of equivalent 100-mm cubes cured under ambient conditions.

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