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EFFECT OF TEMPERATURE ON THE HYDRATION OF THE MAIN CLINKER PHASES IN PORTLAND CEMENTS: PART II, BLENDED CEMENTS

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ABSTRACT

The hydration of three blended cements, i.e., Portland cement blended with GGBFS, PFA, and volcanic ash, based on two neat cements investigated previously, has been followed at five temperatures ranging from 10°C to 60°C. The cements were cured under water and tested at various time intervals over a period of one year. The hydration products were characterised by means of quantitative X-ray diffraction and thermogravimetry. As with the neat cements, a temperature inversion in the degree of hydration was observed, leading to a reduction in the degree of hydration at 60°C compared with that at 10°C, but the presence of the replacement materials changed the hydration patterns of the individual cement phases. There was a clear acceleration of the hydration of alite, C_3A, and the ferrite phase, although PFA did not accelerate alite hydration to the same extent as the other replacement materials. The rate of hydration of belite was retarded by PFA at higher temperatures, but accelerated by the presence of slag or volcanic ash. The depletion of CH by PFA and volcanic ash was shifted to earlier times and lower maximum values of CH content at higher temperatures. © *1998 Elsevier Science Ltd*

Introduction

The use of replacement materials in Portland cement has been gaining much attention in recent years. Given the world situation with respect to cement demand and the cost of energy, not only will blends with materials such as ground granulated blast furnace slag (GGBFS) and pulverized fuel ash (PFA) be used, but other options will also become available to satisfy these needs. In blended cements, the replacement material may take part in the hydration reactions and also contribute to the hydration products. Two groups of cement replacements are commonly recognised, pozzolanas and latently hydraulic materials (1,2). The first group reacts with the CH produced during the cement hydration, while the second has hydraulic

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characteristics itself, but its hydration needs activation, for example by the presence of hydration products (e.g., CH) or other additives (3). The successful use of a replacement material for Portland cement relies on satisfying three main interests, namely economic advantage, environmental benefit, and technological improvement. When waste materials are used, the first and second considerations may act together, and they are then shared by manufacturers and ecologists, but the technological factors must not be ignored. Failure of the materials to comply with any of the three factors can reduce the attractiveness of the replacement material.

The effect of secondary materials, both cementitious and inert, on the hydration of a cement paste has been reported by Gutteridge and Dalziel (4,5), who noted that the presence of a second component enhanced the hydration of Portland cement in cement substituted with PFA or slag. For PFA and slag blended cements, the finer the particle size, the greater the enhancement. Heterogeneous nucleation of hydration products can operate in substituted cements, which have a lower activation energy than homogeneous nucleation, favouring in this way the deposition of hydration products. Increased hydration of alite in the presence of PFA (6,7,8), volcanic material (7), and slag (9) has been reported. Dalziel and Gutteridge (8) reported that for PFA blends, the hydration of the ferrite phase was increased, but that of C_3A was not significantly increased. For belite no significant difference was noted up to 14 days in the presence of PFA. There is less information in the literature about the hydration of the different anhydrous cement phases in the presence of slag and volcanic ash.

Temperature also affects the hydration or interaction of the replacement material with the cement and its environment. Halse et al. (10) reported that the maximum in the CH concentration was displaced to earlier times as the hydration temperature was increased in PFA blended cements. Luke and Glasser (11), working with PFA and slag additions, reported increased consumption of CH in PFA blends at higher temperatures. Since the CH concentration decreased only slightly for the slag containing system, Luke and Glasser (11) suggested a modest demand of CH from the slag fraction.

No information was found in the literature regarding the combined effect of temperature and a replacement material on the hydration of all the individual clinker phases. In this paper we present the results of an extensive and systematic study into the hydration of three blended cements. The parent cements are those described earlier in Part I of this series (12), which will hence be referred to as Part I, in which we described the effect of temperature on the hydration characteristics of the four main clinker phases for two neat cement pastes. Similar results are presented here for three blended cements based on those neat cements.

Experimental

Cements

Three blended cements, based on the two neat Portland cements used in Part I, were made in the following manner: 40% OPCN–60% GGBFS, 70% OPCN–30% PFA, and a cement of approximate composition of 78% OPCS–22% VA (where VA indicates volcanic ash), by weight. The first two were blended in the laboratory and the third was an industrial product, used as received. All of the materials were kindly supplied by the Mexican company Cementos Apasco, except the blast furnace slag which was provided by Frodingham Cement Company Ltd., Scunthorpe, UK. The oxide composition, determined by chemical analysis,

TABLE 1

Chemical analysis and mineralogical composition of the starting materials.

Oxide	GGBFS	PFA	V.ASH	OPCVA	PHASE	OPCN-PFA	PFA	OPCN-SLAG	SLAG	OPCVA
CaO	41.1	1.9	1.0	49.4	Alite	51.5		28.0		48.5
SiO_2	32.9	64.2	68.2	31.9	Belite	8.0		4.5		10.5
Al_2O_3	12.6	26.5	11.2	6.9	C_3A	2.5		1.5		6.0
Fe_2O_3	0.5	4.4	1.8	2.7	C_4AF	8.0		4.5		6.0
Na_2O	0.3	0.1	0.2	0.9	other	2.0		0.5		5.0
K_2O	0.7	1.1	3.4	1.5	Quartz	3.5	11.0	0.5	1.0	1.0
MgO	8.4	0.7	0.2	1.4	Mullite	5.5	18.0			
SO_3	2.2	0.3	0.01	3.2	Magnetite		1.5			
C	0.23	1.34	0.04	0.26	Melilites			0.5	1.0	
Ins Res.					TOTAL	81.0	30.5	40.0	2.0	77
LOI		1.6		2.2	Glassy fraction*	19.0	≈69.5	≈60.0	≈98.0	≈23.0
TOTAL	98.9	100.6	86.1	100.4	Surface area† cm^2/g	3075	2721	4544	6333	4600

* Obtained by difference.

† From laser diffraction particle size analyser, Malvern Instruments.

and the potential phase composition, as estimated by quantitative X-ray diffraction, for the starting materials are reported in Table 1, except those for the neat Portland cements which were given in Part I.

Curing and Hydration

The experimental details, curing, and hydration procedures were the same as those described in Part I, unless otherwise stated. A water:solids ratio of 0.5 was used for all the cements, using distilled water freshly boiled to remove CO_2 gas.

Characterisation

The composition of the blended cements, in terms of cement and replacement material contents, was verified by means of quantitative X-ray diffraction. The results are presented in Table 2. Quantitative X-ray diffraction was also used to estimate the degree of hydration of the cement phases, while thermogravimetric analysis was employed to determine the CH concentration with time. Details of the procedures and considerations about the experimental techniques were given in Part I.

TABLE 2
Composition of the blended cements as measured by quantitative X-ray diffraction.

	OPCN-30% PFA	OPCN-60% GGBFS	OPCVA
Cement	70.5	39.0	75.5
Replacement	31.0	61.0	25.5
TOTAL	101.5	100.0	101.0

Results and Discussion

Quantitative X-ray Diffraction

Alite. The rate of hydration of alite for the three composite cements is presented in Figure 1, while Figure 2 shows a comparison of the degree of hydration of alite between the blended cements and the neat cements, using data for the latter from Part I. In the blended cements, alite is not so much the dominant phase present as in the neat cements, and therefore its hydration is less representative of the total degree of hydration of the cement fraction. As in the case of the neat cements, the rate of hydration of alite was accelerated by increase in the temperature of hydration in the early stages of hydration (up to approximately 7 days), but this situation did not prevail at later ages. The temperature inversion reported in Part I was also observed here. Thus after one year, the lowest levels of hydration were those at 60°C and the highest at 10°C, except for the slag cement (Fig. 1a), where the greatest degree of hydration was observed at 20°C and 30°C.

By comparison with the neat cements, the replacement materials had clearly different effects on the hydration of alite. The hydration was generally accelerated, but the smallest enhancement was for the PFA blend, in contrast with the greater increase produced by the presence of GGBFS and VA, which was observed from very early stages, as can be seen from the data shown in Figures 2A, B and C. After 14 days, the degree of hydration of alite for the neat cement, PFA, and GGBFS blends, respectively, was approximately 69%, 74%, and 84% at 10°C; 77%, 84%, and 90% at 30°C; and 80%, 83%, and 87% at 60°C. In the blended cements, it can be clearly observed that the temperature inversion of the degree of hydration took place at shorter times than in the neat cements and at higher degrees of hydration of alite.

The reactivity of alite in OPCS, which was lower than that of OPCN in the neat cement, was dramatically increased by the presence of the volcanic ash. After 14 days the degree of hydration of alite for OPCS and OPCVA were: 63% and 87% at 10°C; 83% and 92% at 30°C; and 77% and 87% at 60°C. As in the OPCN, the temperature inversion took place in a shorter time in the blended cement. The crossover of the curves at 10°C and 60°C in the OPCVA blended cement took place after approximately 14 days (see Fig. 1C). For all of the blended cements, the lines of alite hydration at 30°C crossed those at 60°C before the curves at 10°C (in less than 7 days in the case of OPCVA).

Belite. Results for the hydration of belite are presented in Figure 3. As in the case of alite the kinetics of hydration are temperature dependent, and the patterns are similar to some extent to those of the neat cements, shown in Part I (11).

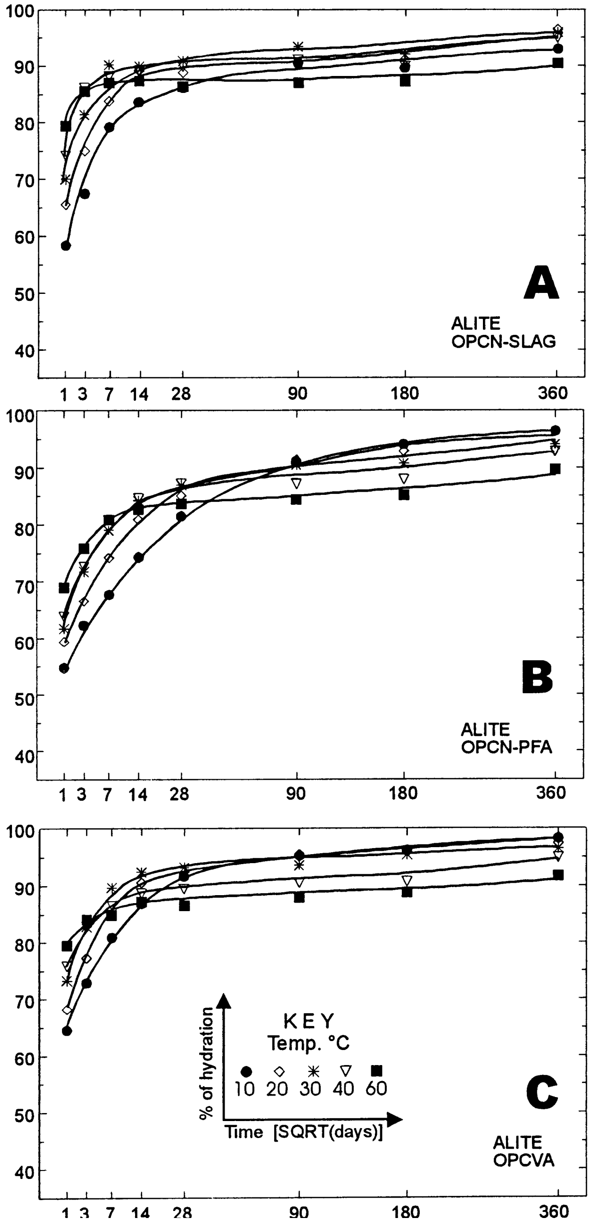

FIG. 1.

Degree of hydration of alite vs. time in the blended cements incorporating (*A*) GGBFS, (*B*) PFA, and (*C*) VA.

In the slag blend, the basic characteristics of the curves for belite hydration are very similar to those in the neat cement. The rates of hydration were higher at all temperatures but less markedly so at 10°C. At 30°C, the belite had apparently been totally consumed after 90 days, while in the neat cement it took up to a year.

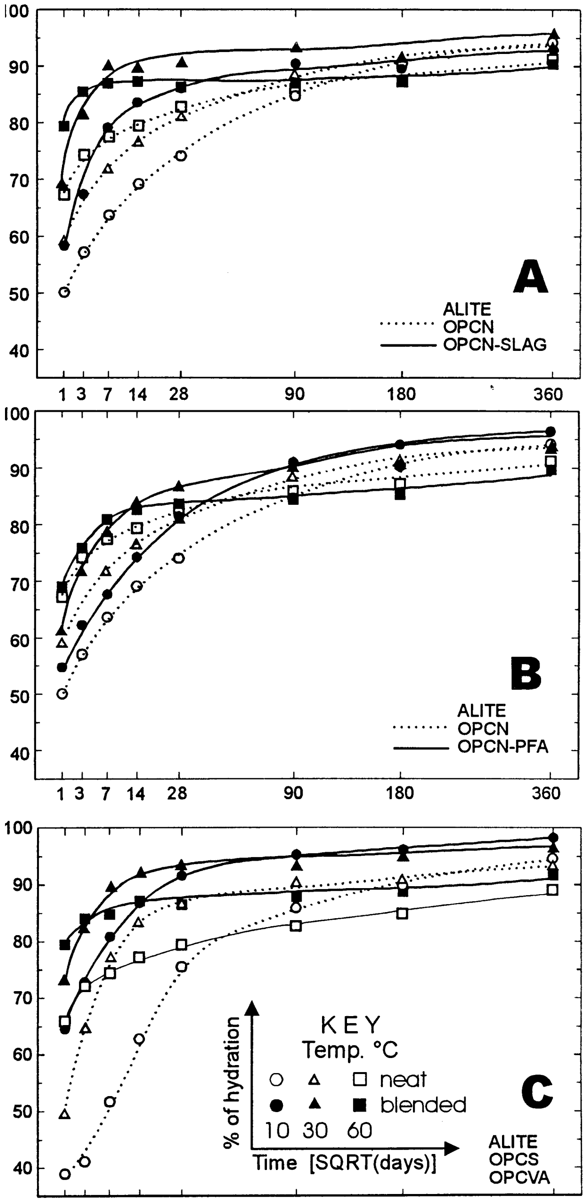

FIG. 2.

(*A*–*C*) Comparison of the degree of hydration of alite between the blended and neat cements (12).

For the PFA blended cement, after approximately 28 days, the curve at 60°C followed closely that at 40°C. Compared with the neat cement, the hydration of belite appeared to be delayed at 60°C and to some extent at 40°C. At 10, 20, and 30°C, for up to 28 days the hydration rates were fairly similar to those noted in neat OPCN, but after that there was some enhancement of the hydration.

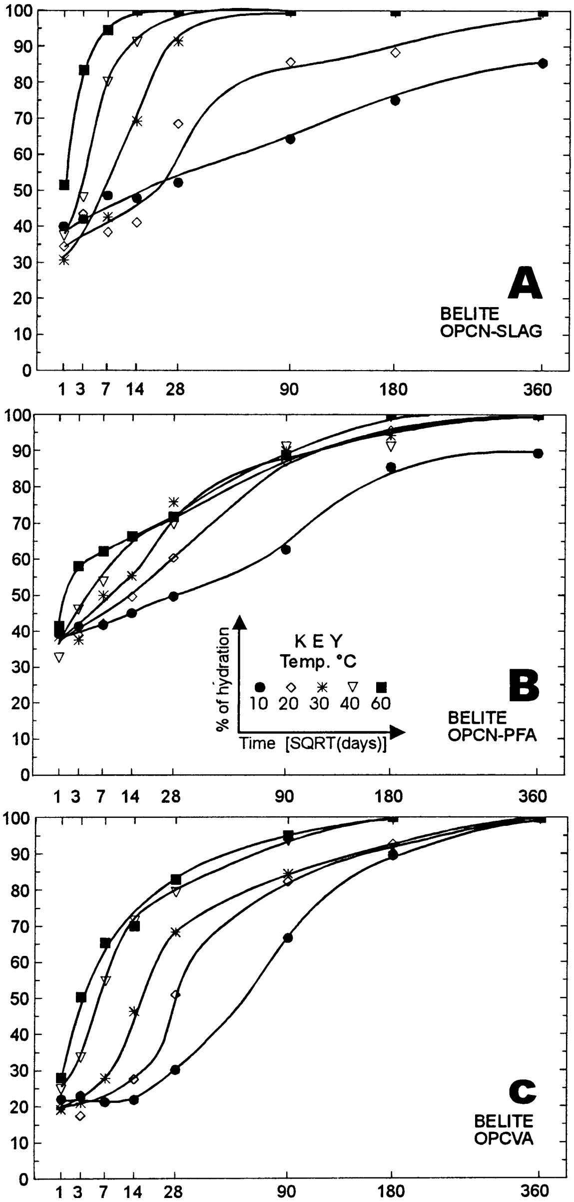

FIG. 3.

Degree of hydration of belite vx vs. time for the blended cements, incorporating (*A*) GGBFS, (*B*) PFA, and (*C*) VA.

In the case of OPCVA the plots were very similar to those of the neat cement up to 28 days at 40 and 60°C and up to 90 days at 20 and 30°C, although there was some acceleration of hydration at 10°C. After one year the belite had totally reacted in OPCVA at all the temperatures investigated. It was apparently consumed after 180 days at the two higher temperatures and between 180 and 360 days at 10, 20, and 30°C.

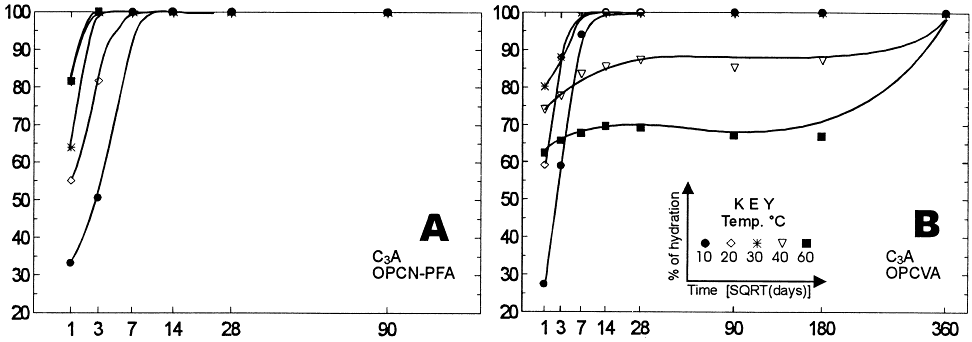

FIG. 4.
Degree of hydration of C_3A vs. time in (*A*) OPCN-PFA and (*B*) OPCS-VA.

For the three blended cements the lowest values for the degree of hydration of belite were always at 10°C. As in the case of the neat cements, the temperature inversion established for alite was not observed for belite.

Tricalcium Aluminate. Hydration curves for C_3A vs time are shown in Figure 4. Data for the slag cement are not presented; because of the very high slag load and the small amount of C_3A in the cement, the high reactivity of this phase ensured that it disappeared very quickly. For the PFA blended cement, the C_3A reacted rapidly, so that after 14 days, it was totally consumed at all temperatures.

For the OPCVA the situation was different. The results indicate that in the first few days, the rate of hydration increased with temperature in the range of 10–30°C, but there was a temperature inversion effect (also noted in the neat cement) so that the C_3A did not fully react at 40°C and 60 °C until beyond 180 days. It reacted completely at 10–30°C after only about 14 days, which was sooner than in the neat cement.

Ferrite. Data for the hydration of the ferrite phase are shown in Figure 5, and in Figure 6 there is a comparison of the new data for the blended cements with those obtained from the neat cements (11). After one year the degree of hydration reached for the blended cements was greater than for the neat cements in every case. The rate of hydration of the ferrite phase increased in all the blended cements from the early stages as shown in Figure 6, but of the three replacement materials, the greatest acceleration effect was that observed in the GGBFS blend. In this blended cement, the ferrite phase appears to have fully reacted after only 90, 140, and 180 days at 10, 30, and 60°C, respectively. The temperature inversion effect is also apparent with all three blended cements with very similar patterns to those in the neat cements. As in the case of alite, the curve for 30°C crosses that for 60°C before that for 10°C. In the two pozzolanic blended cements, the rate of hydration was strongly accelerated to relatively similar extents in both cements, and more markedly at the three lower temperatures. It is noteworthy that, as for alite, the temperature inversion in the degree of hydration took place at higher degrees of hydration and also at shorter times in the blended cements than in the neat cements.

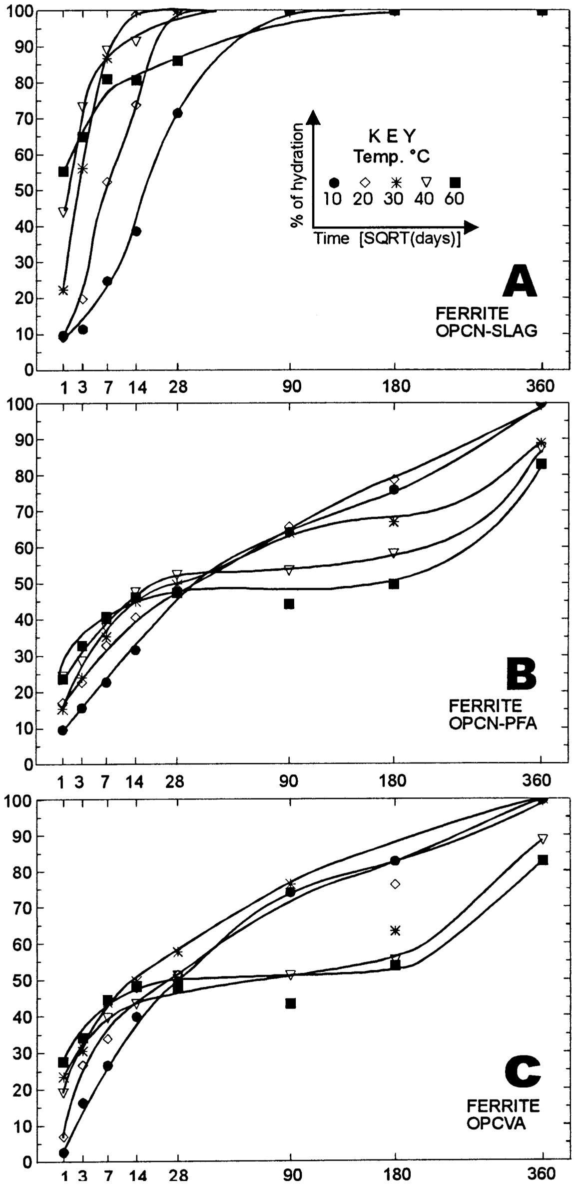

FIG. 5.
Degree of hydration of ferrite vs. time for the blended cements, incorporating (*A*) GGBFS, (*B*) PFA, and (*C*) VA.

Calcium Hydroxide Determinations

Determinations of the calcium hydroxide content of the hydrated cements by thermogravimetric analysis are shown in Figure 7 for the three blended cements, using the method that has been described previously (12). The CH contents have been normalised depending on the

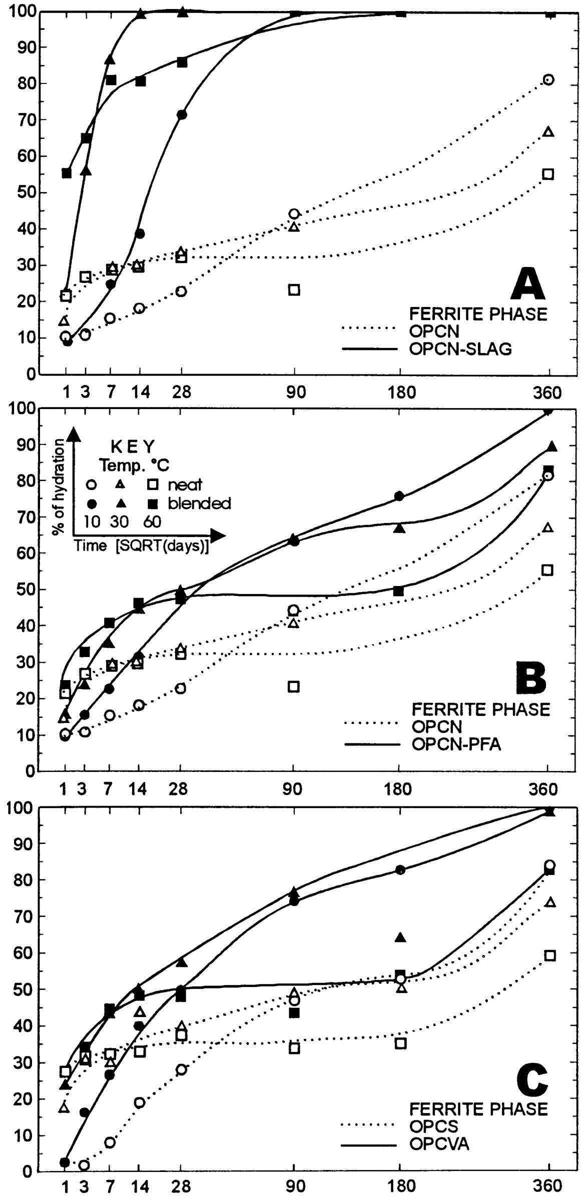

FIG. 6.

(*A–C*) Comparison of the degree of hydration of the ferrite phase between blended and neat cements (12).

replacement level in the cement, e.g. the CH content from the slag cement was divided by a factor of 0.4, in order to compare the CH values with those of the neat cements. Because the replacement materials react with the calcium hydroxide produced by the cement, the plots do not resemble those for the hydration of alite as they did in the neat cement systems.

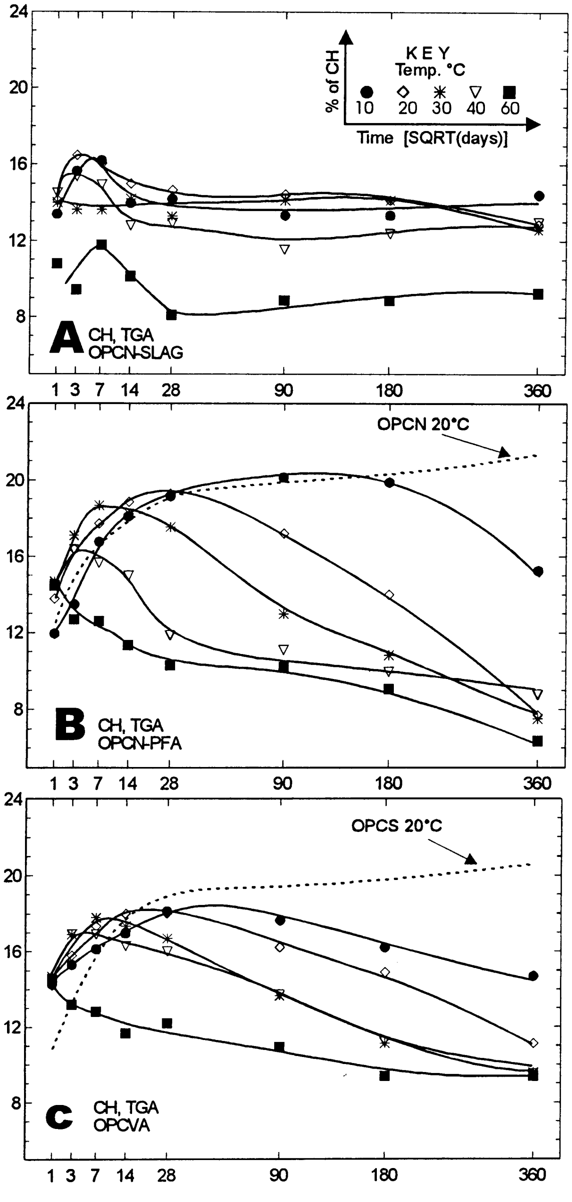

FIG. 7.

% Calcium hydroxide vs. sqrt. of time for the blended cements, (*A*) GGFS, (*B*) PFA, and (*C*) VA. Data for the neat cements at 20°C from ref (12).

It can be seen from the results shown in Figure 7C that the volcanic ash was a very effective pozzolanic addition to the Portland cement. In the early stages of hydration, its reaction with the lime liberated was slightly more than that for the PFA, although this position was reversed after longer periods of hydration, probably because of the higher level of replacement in the OPC-PFA blended cement.

A normal pattern for the CH concentration in the pozzolanic cements is that in which CH is initially produced by the cement (and perhaps consumed by the pozzolana at the same time, but at a much lower rate), and the CH concentration then reaches a maximum and subsequently declines when the rate of consumption by the pozzolana surpasses the rate of production by the cement, but the actual commencement of the pozzolanic reaction is difficult to determine from this information alone. For the PFA and VA blended cements, it can be seen from Figures 7B and C that there was a strong effect of temperature on the pozzolanic reaction, and that for both cements the maximum concentration reached by the CH was shifted to lower values and to earlier times as the hydration temperature was increased. In the early stages, the production of CH increased with respect to the neat cements (e.g. after 7 days at 20°C, the CH contents were approximately 16.5% and 18.0% for OPCN and OPCN-PFA, respectively, and 16.0% and 17.5% for OPCS and OPCVA), in line with the earlier observations of increased hydration of the silicates, reported above. For both pozzolanic cements hydrated at 60°C, it appears that after only one day the rate of consumption of CH was greater than its production by the silicates in the cement, with CH values lower than in the neat cement, and, therefore, that the pozzolanic reaction was underway.

That the pozzolanic reaction was not favoured by relatively low hydration temperatures can be noted from the data for both cements at 10°C shown in Figure 7, since the consumption of CH does not reach the same levels as at the higher temperatures. After 360 days, the concentration of CH for temperatures other than 10°C reached similar concentration levels for both of the blended cements containing pozzolanic additions and the curves at 60°C always displayed the lowest CH content in both pozzolanic cements. It would appear that the PFA cement depleted CH at a higher rate than OPCVA in the later ages, but it has to be remembered that the maximum CH values reached by the PFA blend were higher, misleading one to think that the rates were higher, when VA has consumed more CH initially. Clearly both PFA and volcanic ash acted as effective pozzolanic additions.

For the slag cement, the CH levels shown in Figure 7A appeared to increase in the very early stages of hydration with a maximum for all temperatures between 3 and 7 days. Beyond this peak there were only slight variations in the CH level with increasing curing time. The total CH concentration expected for the neat cement is about 22%, and the normalised values in the slag were below 16%. These lower CH values suggest some pozzolanic behaviour of the slag from early ages, although it did not show the typical pattern of a pozzolanic material. Contrary to what was noted with the other cements, the highest hydration temperature did not yield the highest amounts of CH at the earlier stages. It can be noted that the lowest CH levels were constantly those at 60°C, followed by those at 40°C, whereas the highest values were observed at 10, 20, and 30°C, which may suggest an increasing pozzolanic effect of the slag in the early stages with increasing temperature of hydration.

The small peak observed in the CH concentration in Figure 7A is regarded as an acceleration of cement hydration, in line with our XRD observations, and the further reduction in CH content is considered as slag reaction (13). The small variations in the CH concentration after 14–28 days would be in line with the corresponding observed reduced rates of hydration of alite in this cement, as shown in Figure 1, after an initial rapid hydration. The nearly zero rates of CH production after 28 days can be considered as an indication of a little CH consumption by the slag hydration that undoubtedly continued to hydrate during this period of time. This denotes that the slag did not consume as much CH as the pozzolanas, similar to the suggestion made by Luke and Glasser (11). Wilding and McHugh (14) observed a similar CH pattern with several slag substituted cements. To form C-S-H the slag has to take some CH (20), similarly to form AFm or other calcium-bearing phases. As the slag is more reactive with increase in the temperature, the observed reduction of CH in the relatively early stages at 60°C can provide the slag with the needed Ca^{2+} ions. After 14 days the hydration of alite progressed relatively slowly; in agreement with this, the observed CH production was also reduced and CH levels remained relatively constant. During this period the slag's needs for Ca^{2+} ions were probably satisfied from the CSH gel product. The CSH product in OPC-GGBFS systems has been reported to have a lower Ca/Si ratio compared to that in the hydration product from neat cement. (13,15).

Further Discussion

The presence of the replacement materials as well as the variation in the hydration temperature produced significant changes in the hydration of the cement phases. The rates of hydration were generally increased for the four anhydrous phases in the presence of GGBFS, PFA, and VA. However, the results from OPCVA must be interpreted with caution, as this cement was interground and had a higher specific surface area than that of the neat cement. The other two blended cements were, however, made in the laboratory so that a direct comparison is more acceptable.

One very important factor that has to be taken into account here is the water:solids ratio, which was kept constant at 0.5 regardless of the replacement level of the cements. This means that there was a "dilution effect" with respect to the cement, since there was more water to react in the slag cement than in the other cements (water/cement ratios = 1.25, 0.71, and 0.64 for GGBFS, PFA, and VA blended cements, respectively). Copeland and Kantro (16) have reported increased hydration of alite at higher w/c ratios. At w/c ratios of 0.35 and 0.63, the approximate hydration values of alite were 50% and 58% after 3 days, and 85% and 93% after 100 days, respectively. Such an effect may operate in these cements. Therefore it is possible that, in the context of this study, the effect of the additional water (relative to the cement) amounts to an effect of the presence of the replacement materials in increasing the level of hydration with respect to the systems involving only the neat cements. It was observed, however, that in spite of the additional water, PFA did not increase alite hydration to any great extent.

Most previous workers, comparing neat cements with blended cements, have reported data obtained at a constant water:solids ratio for the compared cements (5,6,7). No information was found on the effect of slags on the hydration of the individual anhydrous Portland cement phases. Furthermore, the observed reactivities for the alite in the three OPCN based cements was, OPCN $<$ OPCN-pfa blend $<$ OPCN-slag blend, which follows the same order of increasing amount of water available for reaction, i.e., water/cement ratio of 0.5, 0.71, and 1.25, respectively.

In addition to the above mentioned, another factor to consider is the reactivity of the replacement materials and whether the pozzolanic reaction requires additional water. The slag is recognised as a hydraulic material and its hydration does consume water. It was shown in Figure 7 that the pozzolanic reaction is accelerated by increasing temperature, and if this reaction consumes water, then the dilution effect is reduced from early ages. For the slag, it is well known that temperature also favours its hydration and that at low temperatures the slag has a reduced reactivity. Weiping et al. (17) observed that higher temperatures activated slag hydration, while Bland and Sharp (18) suggested an early participation of the slag in the heat evolution of blended cement hydrated at 38°C. We have noted (19) by means of estimations of non-evaporable water that for this cement, the slag hydration is accelerated at higher temperatures. How much water is consumed by the slag is not easy to determine but an augmented reactivity will consume water and reduce the "dilution effect" on the Portland cement fraction in the blended cements. Water has to be provided for the slag to hydrate, hence the dilution effect is unavoidable, and this will probably contribute to the acceleration of the hydration of the cement, and also reduce the early strength.

Addition of GGBFS certainly produced enhanced hydration of the four anhydrous phases, as did incorporation of VA. According to Uchikawa (9) the hydration of the interstitial phases is accelerated because the slag fixes Ca^{2+} and SO_4^{2-} ions; additionally the dilution effect favours the dissolution of cement grains helping the hydration of the interstitial phases. In the pozzolanic cements, in addition to heterogeneous nucleation, the increased hydration of alite can be viewed from the equilibrium standpoint; CH is a product of alite hydration and it is removed by the pozzolanic reaction, hence shifting the equilibrium to a more advanced degree of hydration of the reactants. The slag is also believed to consume CH to form C-S-H and AFm in the initially water-filled space (20), and therefore alite hydration is similarly increased. Only belite displayed some delay to its hydration in the presence of PFA.

The temperature inversion on the alite and ferrite hydration was believed to be due to a similar mechanism as in the neat cements, as described in Part I. At higher temperatures there is the deposition of more compact inner hydration product surrounding the hydrating grains, slowing down the diffusion of the reactants, hence retarding further hydration.

All these factors make a very complex picture. There is an effect of increasing temperature to bring about initial acceleration of the hydration of the cement phases, as reported in Part I (12), and in addition to that, there is the physical presence of the replacement material, the "dilution effect" and the chemical influence of the replacement material. All these factors can affect the cement hydration but with different and uncertain weights of contribution. Further research is needed to complement with information comparing the hydration of the clinker phases using a constant water:solids ratio in the neat and blended cements.

Conclusions

The hydration of the main clinker phases in three blended cements at five different temperatures has been studied using quantitative X-ray diffraction and thermogravimetric analysis. The results indicate different effects from each of the mineral admixtures in the cements. As with the neat cements, increase in temperature accelerated the early hydration of the cement phases.

The hydration of alite (Figs. 1 and 2) was greatly increased from very early stages by the presence of GGBFS and VA, but to a much lesser extent by PFA. There was a temperature inversion effect in the hydration of alite with the highest degree of hydration being observed at the lowest temperatures in the long term and vice-versa.

Belite hydration (Fig. 3) was delayed in the presence of PFA at 40°C and 60°C, but was somewhat enhanced at the lower temperatures. For the VA blend there was acceleration at 10°C and the characteristics of the plots were similar to those reported earlier for the neat cement but with some increase in the rates of hydration, reacting completely after 360 days. For the slag cement, there was a marked temperature dependence and the curves were similar to those of the neat cement, except that hydration was faster. For all three blended cements, the curve of hydration at 10°C always had the lowest hydration values and there was no evidence for a high temperature inversion.

The hydration of C_3A (Fig. 4) was accelerated in the PFA blend, reacting completely after 14 days at all temperatures. For the VA blend, hydration rates were also higher and there was a temperature inversion similar to that of the neat cement, OPCS, reported earlier (11). The hydration of the ferrite phase (Figs. 5 and 6) was dramatically accelerated by the presence of the slag, having fully reacted after only 180 days at all temperatures. VA and PFA also increased ferrite hydration but to a lesser extent. The high temperature inversion was observed for the ferrite phase in all of the blended cements.

The peak contents of CH in the pozzolanic cements shifted to shorter times and lower values as the temperature was increased, indicating that the pozzolanic reaction was favoured by the higher temperatures. The slag cement displayed a small peak after 7 days and then the CH content remained almost constant at each temperature, indicating little or no further consumption of CH. The lower absolute values of CH content suggest some early pozzolanic behaviour of the slag. The lower CH content at 60°C in the slag blend may indicate a stronger pozzolanic behaviour of the slag in the early stages of hydration.

The effect of the additional water in the blended cements may play an important role in the increase in the degree of hydration of the clinker phases (in addition to the changes in temperature) and it must be considered as an additional factor involving the physical presence of the replacement materials as well as their chemical influence. Further research is needed to help to understand this “effect of dilution” and its real weight amongst the other factors that influence the degree of hydration of the cement.

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