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Investigation of the temperature change and heat evolution of mortar incorporating PFA and metakaolin

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Abstract

Temperature-time profiles of mortar cubes have been determined using a semi-adiabatic curing method for curing times of 160 h. The binders employed were metakaolin–portland cement (MK–PC), fly ash-portland cement (PFA–PC) and MK–PFA–PC blends, with a maximum PC replacement level of 40% and MK levels of up to 15%. Relative to PC mortar (and as previously reported by other workers), MK–PC mortars show an enhanced temperature-rise whereas in PFA–PC mortars the temperature-rise is depressed. In ternary mortars MK and PFA have a compensatory effect. Both MK and PFA delay the time taken to reach the peak temperature with MK producing the greatest delay. Partial substitution of MK for PC has only a minor influence on the total heat evolved by the mortar whereas partial substitution of PC with PFA produces a marked fall in the heat evolved. The temperature-rise of MK–PC mortars with respect to PC-mortar shows two distinct peaks which are attributed to a two-stage hydration effect. © 2002 Elsevier Science Ltd. All rights reserved.

Keywords: Heat evolution; Temperature rise; Mortar; Pulverised-fuel ash; Metakaolin

1. Introduction

Metakaolin (MK) has enormous potential as a pozzolanic material in the production of mortar and concrete with lower embodied energy [1], with improved performance and with enhanced durability relative to portland cement (PC) mortar and concrete. Although a large amount of published research data are available [2] on the properties of MK–PC blends, due to the high cost of MK their use at present tends to be restricted to special applications, such as suppression of efflorescence in paving slabs and coloured concrete blocks, depletion of CH in GFRC and to produce concrete of enhanced durability for applications in severe environments [3]. In addition, although MK does significantly improve the performance of concrete, there are a number of disadvantages to it being more universally applied. These include its high cost relative to PC, and its high water requirement. Recent work also indicates that MK–PC blends produce greater heats of hydration and temperature-rise in concrete and mortar than does PC alone [3–5]. For example, Zhang and Malhotra [5], in a study of thermally activated alumino-silicate pozzolans, reported a temperature-rise 7°C greater than PC concrete when 10% of the PC was replaced with MK, whereas 10% silica fume (SF) produced a temperature-rise only slightly above (0.5°C) that of the PC concrete. Ambroise et al. [3], in similar work on MK–PC mortar using semi-adiabatic calorimetry, reported temperature-rises above that of the control PC mortar by 8°C, 6°C and 1°C, at PC replacement levels by MK of, respectively, of 10%, 20% and 30%.This enhanced temperature-rise could lead to severe problems of thermal stress and cracking particularly in large volume mass concrete. Bai et al. [6,7] have demonstrated that some of the disadvantages associated with using MK to partly replace PC can be overcome by blending the MK with PFA. The current paper reports the results of research on the heat evolution and temperature-rise of MK–PC and PFA–PC binary blend mortars and MK–PFA–PC ternary blend mortars.

Sanchez de Rojas et al. [8] have demonstrated that pozzolanic materials do not necessarily cause a reduction in heat evolution in proportion to the amount of cement substituted and that some pozzolans in fact increase the heat evolved whereas others decrease it.

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Sanchez de Rojas and Frias [9] studied the relationship between pozzolanic activity (measured by lime consumption of the pozzolan in a saturated lime solution) and the heat evolved (determined using a Langavant Calorimeter) for a number of hydrating cement-pozzolan blends including fly ash and silica fume. For mortars produced using SF–PC blends and with substitution levels up to 15% the total heat liberated (0–120 h) was greater than that for PC mortar. Conversely for 30% PFA–PC mortar the total heat liberated was significantly less than that for PC mortar. The authors demonstrated a correlation between pozzolanic activity and the relative (i.e. relative to PC mortar) heat evolved.

Frias et al. [4] also compared, using similar methods, the heat evolution from PFA–PC, SF–PC and MK–PC mortars. The total heat evolved (up to 120 h hydration) was found to decrease significantly with increase in substitution of PC with PFA, showed very little change with increase in substitution of PC with MK, and showed a small increase on substitution of PC with 10% SF. The authors also measured the temperature-rise for the hydrating mortars. The MK–PC mortars showed a temperature peak significantly higher (6–7°C) than that for PC mortar, in agreement with [3,5], whereas FA–PC mortars exhibited a continual reduction in the temperature peak with increase in substitution level. For the SF–PC mortars the temperature peak was slightly higher (1.5°C) for 10% substitution and somewhat lower (3.0°C) for 30% substitution. The authors also plotted the difference in the heat evolved, between the pozzolanic mortars and the PC mortar (relative heat output), with time. For PFA–PC mortars the relative heat output fell continuously over the first 20 h and then showed a slight increase. The drop in relative heat output was attributed to the reduced PC content (dilution effect) and the negligible contribution from pozzolanic activity by the PFA. For SF–PC mortars relative heat output increased to a peak at about 8 h and then fell to an approximately constant level by 20 h. The authors attributed the peak in relative heat output to the pozzolanic reaction of the SF, which more than compensated for the reduction in PC content. Similarly the MK–PC mortars showed a peak in relative heat output at about 8 h but in addition they also showed a second peak at 18–20 h. The authors suggested that the peak at 8 h was due to the reaction of CH with SiO_2 to form C–S–H gel and that the second peak was due to the reaction of CH with Al_2O_3 (and SiO_2) to give C_4AH_{13} (and C_2ASH_8).

Clearly the total heat evolved and the rate of heat evolution from hydrating PC-pozzolan blends depend on the type of pozzolan and the PC substitution level. Partly replacing PC with a pozzolan can result in a number of different processes contributing to the heat output from the hydrating system. The reduced PC content will reduce the total heat output from PC hydration but will not necessarily reduce the initial rate of heat evolution because some pozzolans are known to accelerate PC hydration. There are a number of reports that indicate that very active pozzolans, such as SF [10], produce significant acceleration in PC hydration. It has been reported that some fly ashes can also accelerate PC hydration. For example Kokubu [11] showed that the initial rate of heat evolution of PFA–PC–water mixtures increased progressively with increasing PFA content (water washed PFA) in spite of the decreased cement contents. This was attributed not to reaction of the PFA but to acceleration of cement hydration. If the pozzolan is highly active then the heat of reaction of the pozzolan (with CH and water) will also contribute to the heat evolution in the early stages of reaction, compensating for the reduction in the cement content. Thus the combination of acceleration in cement hydration and the pozzolanic reaction can lead to enhanced rates of early heat evolution and a subsequent temperature-rise in excess of that for PC alone.

2. Experimental method

The pulverised-fuel ash (PFA) and the MK were supplied by Ash Resources and Imerys (formerly ECC International), respectively. The compositions of the PFA and MK (Meta Star 501) are given in Table 1.

Table 1
Physical and chemical properties of PC, PFA and MK

Oxide	Cement (%)[a]	PFA (%) [b]	MK (%)[c]
SiO_2	21.0	49.8	52.1
Al_2O_3	4.63	26.4	41.0
Fe_2O_3	2.26	9.3	4.32
CaO	65.6	1.4	0.07
MgO	1.18	1.4	0.19
SO_3	2.69	0.8	–
Na_2O	0.16	1.5	0.26
K_2O	0.78	3.5	0.63
TiO_2	–	1.0	0.81
Cl	0.01	0.01	–
LOI	0.99	4.9	0.6
Physical property			
Surface area (m^2/g)	0.47[d]	0.53[d]	12
Density (g/cm^3)	3.15[d]	2.34[d]	2.49

The compound composition according to the procedure given in BCA document 92/TG4/16 is 64%C_3S, 11%C_2S, 8.2%C_3A and 6.7%C_4AF.

[a] Data supplied by Rugby Cement.

[b] Data supplied by Ash Resources.

[c] Data supplied by Imerys (former ECC International).

[d] Data obtained from our own particle size analyser.

Portland cement complying with BS 12 (EN 197), 1991 and natural sea-dredged sand of grading M-F complying with BS 882, 1983 were used throughout the investigation. The tap water for the mixes was stored in the curing room overnight to allow it to equilibrate with the room temperature. A total of seventeen mortar mixes were prepared with varying levels of cement replacement. In both the binary (PFA–PC, MK–PC) and ternary (MK–PFA–PC) blends the PFA levels ranged from 0% to 40% and the MK levels from 0% to 15%. The compositions of the mixes, which were proportioned and mixed according to European Standard EN 196-1, are given in Table 2.

Each mortar mix was placed in a 150 mm plywood cube mould that was fully encased in 100 mm thick expanded polystyrene within an outer 400 mm plywood cube (Fig. 1). The temperature of the curing room was maintained at 20 ± 2°C. A thermocouple was embedded in the centre of each mortar cube, and the thermocouples were linked to a computer. Temperature readings were continually recorded at 1-min intervals immediately after casting, for a duration of seven days. The reference temperature was simultaneously measured by a second thermocouple inserted in an identical cube that contained a non-cementitious mix (the 'reference mix') of sand, PFA and water (3:1:0.5). This method is a form of semi-adiabatic curing similar to that used by Frias et al. [4] who employed a Langavant calorimeter. The difference between the 'reference mix' temperature and the test mix temperature is defined as the 'temperature-rise'. The 'heat evolved' by the mix, over the 160-h curing period, is taken as being proportional to the area on the temperature time graphs between the temperature profile of the test mix and the temperature profile of the 'reference mix'.

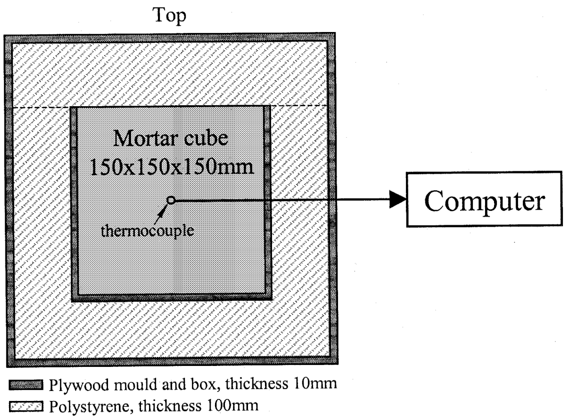

Fig. 1. Schematic diagram of experimental set-up.

3. Results and discussion

Figs. 2(a)–(f) show the temperature-rise versus time plots for the MK–PC mortars, the PFA–PC mortars, the MK–PFA–PC mortars and the PC control mortar, for total PC replacement levels of up to 40%. For MK–PC mortars (Fig. 2(a)) the rate of increase in the temperature-rise, at short curing times, increases with increase in MK content and for PFA–PC mortars (Fig. 2(b)) the rate of increase in the temperature-rise, at short curing times, decreases with increase in PFA content. Also the peak temperature-rise (see Fig. 3(a)) decreases with increase in PFA content (from 0% to

Table 2
Mix proportions

OPC (%)	MK (%)	PFA (%)	OPC (kg/m^3)	MK (kg/m^3)	PFA (kg/m^3)	Water (kg/m^3)	Sand (kg/m^3)
0	0	100	0	0	444	222	1333
100	0	0	444	0	0	222	1333
95	5	0	422	22	0	222	1333
90	10	0	400	44	0	222	1333
85	15	0	378	67	0	222	1333
0	0	10	400	0	44	222	1333
80	0	20	356	0	89	222	1333
70	0	30	311	0	133	222	1333
60	0	40	267	0	178	222	1333
90	2.5	7.5	400	11	33	222	1333
90	5	5	400	22	22	222	1333
90	7.5	2.5	400	33	11	222	1333
80	5	15	356	22	67	222	1333
80	10	10	356	44	44	222	1333
70	2.5	27.5	311	11	122	222	1333
70	5	25	311	22	111	222	1333
70	7.5	22.5	311	33	100	222	1333
60	10	30	267	44	133	222	1333

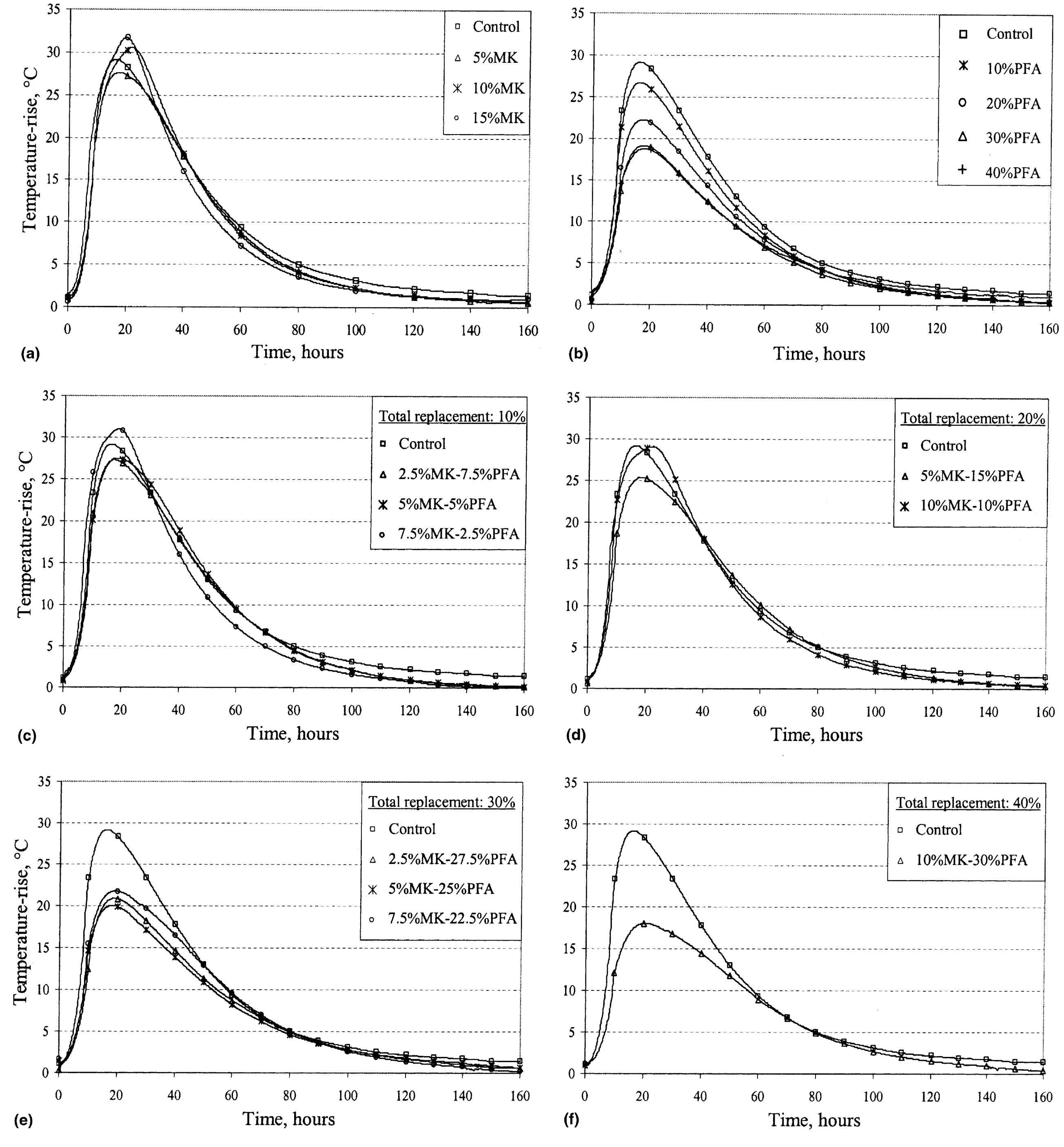

Fig. 2. Temperature-rise for binary and ternary blend mortars versus time.

40% PFA) and increases with increase in MK content (from 5% to 15% MK). This is in agreement with Ambroise et al. [3], Frias et al. [4] and Zhang and Malhotra [5] all of whom reported an increase in peak temperature-rise when MK partially substitutes for PC (for up to 30% replacement of PC by MK) in, respectively, mortar [3,4] and concrete [5]. Bajracharya et al. [12] however claim that the replacement of PC by MK (0%, 10%, 15% and 20%) does not vary the adiabatic temperature-rise of concrete. They record adiabatic temperature-rises of between 34.7°C and 36.8°C. This discrepancy in behaviour may result from differences in the purity and specific surface of the MK used by the different researchers. It may also result from the fact that experiments conducted by [12] were under fully adiabatic conditions whereas other workers [3–5],

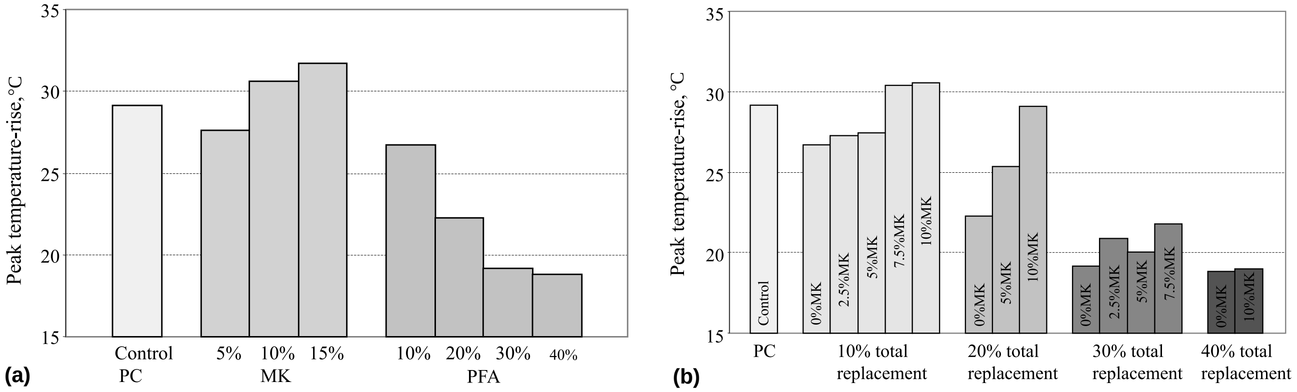

Fig. 3. (a) Peak temperature-rise for metakaolin and PFA blended mortars; (b) Peak temperature-rise for MK–PFA–PC blended mortars.

including the current authors, used semi-adiabatic conditions. In the latter case greater rates of heat generation for the same total heat output will produce a relatively enhanced temperature rise because there is a shorter time period for the heat to escape.

When both PFA and MK are blended together with PC, at total PC replacement levels of 10%, 20%, 30% and 40%, the effect that they have on the temperature-rise tends to be compensatory. Thus the initial increased rate of heat evolution provided by the MK is offset by the reduced rate provided by the PFA. Hence, for example, at 10% total replacement of PC with MK–PFA blend (Fig. 3(b)), the peak temperature-rise increases with increase in the proportion of MK to PFA in the blend, and the blends with high MK to PFA ratios have peak temperature-rise values above that of the control and the blends with low MK to PFA ratios have peak temperature-rise values below that of the control. The trend is similar for 20% total replacement but because for the mixes with 30% and 40% total replacement the proportion of MK to PFA is much smaller the influence of the PFA predominates and the MK has a much reduced influence on the peak temperature-rise.

Figs. 4(a) and (b) show the time to peak temperature for the binary and ternary blend mortars. Both PFA and MK delay the time taken to reach peak temperature. The time delay imparted by MK (Fig. 4(a)) is however greater than that imparted by PFA (at the same replacement level) and, unlike PFA, MK does not produce systematic changes in delay time with increase in replacement level. When both PFA and MK are blended together with PC (Fig. 4(b)) the delay time increases substantially with increase in the proportion of MK to PFA in the blend. At 30% and 40% total replacement the impact of the MK on the delay time is less pronounced because of the much lower MK to PFA ratios employed in these mixtures.

Fig. 5(a) shows the ratio of the heat evolved by the binary blend mortars to that evolved by the PC mortar (for a hydration period of 160 h) versus blend composition. Fig. 5(b) shows equivalent data for the ternary blend mortars. Metakaolin mortar shows a slightly declining ratio of heat evolved, with increase in MK content. This is in broad agreement with the work of Frias et al. on MK–PC cement mortars [4] and Poon et al. on MK–PC cement pastes [13], both of whom observed only small differences in heat output between MK–PC

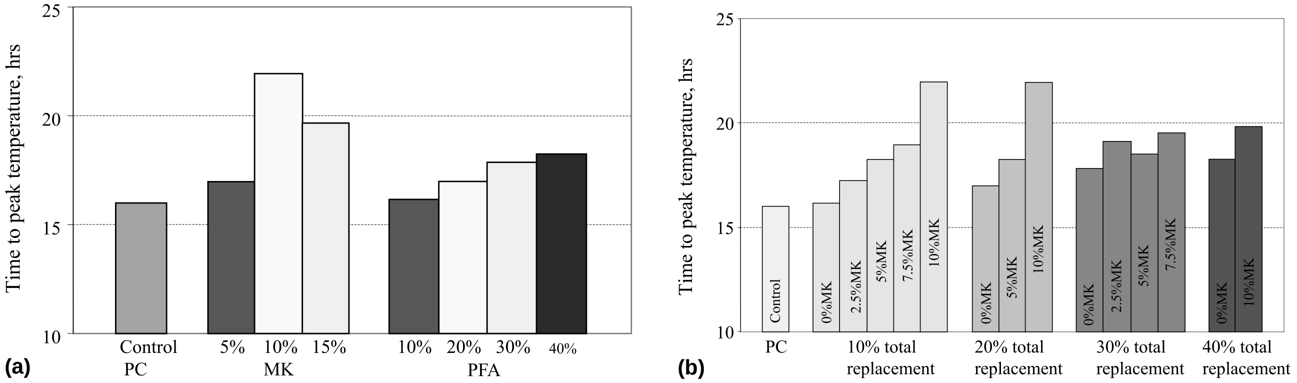

Fig. 4. (a) Time to peak temperature for metakaolin and PFA blended mortars; (b) Time to peak temperature for MK–PFA–PC blended mortars.

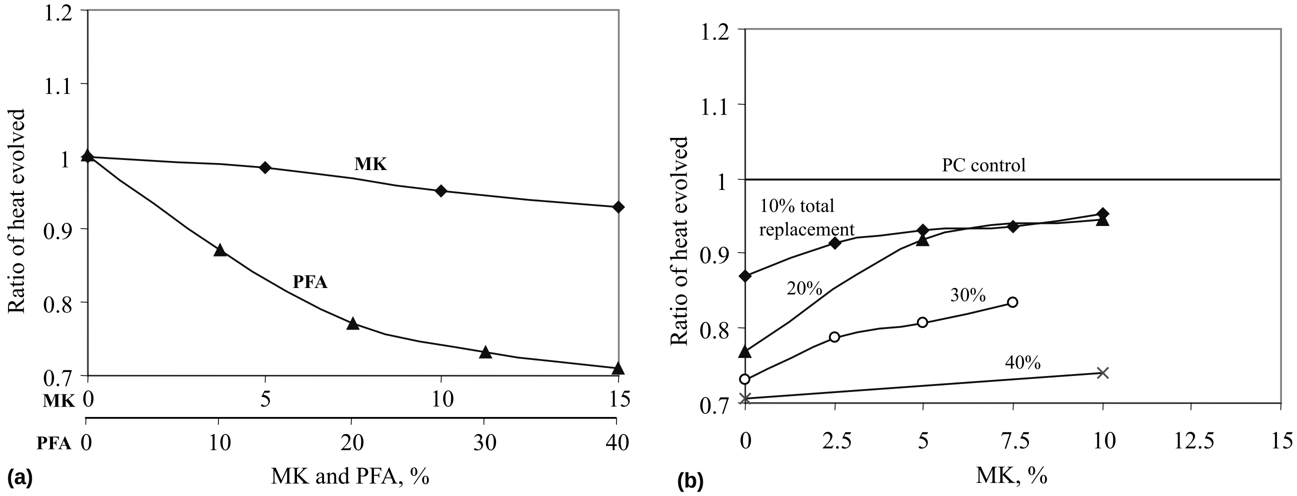

Fig. 5. (a) Ratio of heat evolved for MK and PFA blended mortars relative to that for equivalent PC mortars versus blend composition; (b) Ratio of heat evolved for MK–PFA–PC blended mortars relative to that for equivalent PC mortars versus blend composition (total replacements (MK + PFA) at 10%, 20%, 30% and 40%).

blends and the control PC. Thus although MK has only a minor influence on the 'total' heat evolved by the MK–PC mortars it has a significant effect on the temperature-rise. This is explained in terms of a significant increase in the initial rate of heat evolution when MK is blended with PC. PFA–PC mortar however shows a sharp decline in the ratio of heat evolved, which relates directly to the reduction in peak temperature-rise with increase in PFA content (see Fig. 3(a)). For the ternary blend mortars (Fig. 5(b)) the ratio of heat evolved is very sensitive to small amounts of MK particularly at low total replacement levels where the ratio of heat evolved shows substantial increases. This is attributed to the much greater pozzolanic activity of MK relative to PFA during the first 160 h and particularly in the first 24 h where additionally it accelerates PC hydration. The decline in the ability of MK to further increase the ratio of heat evolved at higher MK levels and particularly at the very high substitution levels may be a result of the reduced availability of CH. For specimens that are not perfectly insulated (i.e. non-fully adiabatic as in the current work) those that generate heat most rapidly (i.e. those with increasing amounts of MK) show the greatest temperature-rise. The advantage of using PFA with MK is that, other than for specimens with very high MK/PFA ratios and low total replacement levels, this temperature-rise is well below that of the PC control concrete. Unpublished work by the current authors on the durability of MK–PFA–PC concrete (with respect to sorptivity, carbonation and chloride ingress) shows that small amounts of MK when combined with PFA and PC as binder can also provide enhanced durability.

Figs. 6(a) and (b) show the 'relative temperature-rise' of, respectively, the binary and ternary blend mortars versus time. The relative temperature-rise is the temperature-rise of the blend mortars relative to the equivalent PC mortar, which by definition has a relative temperature-rise of zero. Fig. 6(a) shows that for all compositions there is a discontinuity between 6 and 10 h, which produces a sudden increase in relative temperature-rise. This increase is very substantial for the MK mortars and is more pronounced as the MK content increases. Thus for 15% replacement of PC with MK the complete peak (between 0 and 13 h) is above the zero line. For the PFA blend mortars the relative temperature-rise is negative from the outset and becomes increasingly negative as the PFA content increases but there is still a small increase superimposed on the background trend, between 6 and 10 h. This is the time period during which peak heat evolution occurs in hydrating PC due principally to hydration of C_3S. A similar peak at the same curing period was observed by Frias et al. [4]. It is suggested [3,4,13] that in the case of MK–PC the heat evolution deriving from the strong early pozzolanic activity, coupled with that from the acceleration in cement hydration (as a result of the rapid consumption of CH which drives the reaction forward), produces a temperature-rise in excess of that of the PC mortar. In the case of the PFA–PC mortars there is negligible initial pozzolanic activity and hence dilution of the PC results in increasingly negative relative temperature-rise values as the PFA content increases. However the PFA particles do make available a large surface on which CH can be adsorbed or be involved in an initial surface reaction. It is possible that this initial lime adsorption by the PFA is sufficient to accelerate the C_3S hydration and produce the small peak observed at between 6 and 10 h.

In addition to the peak between 6 and 10 h the MK–PC mortars also exhibit a second peak after 20 h, the

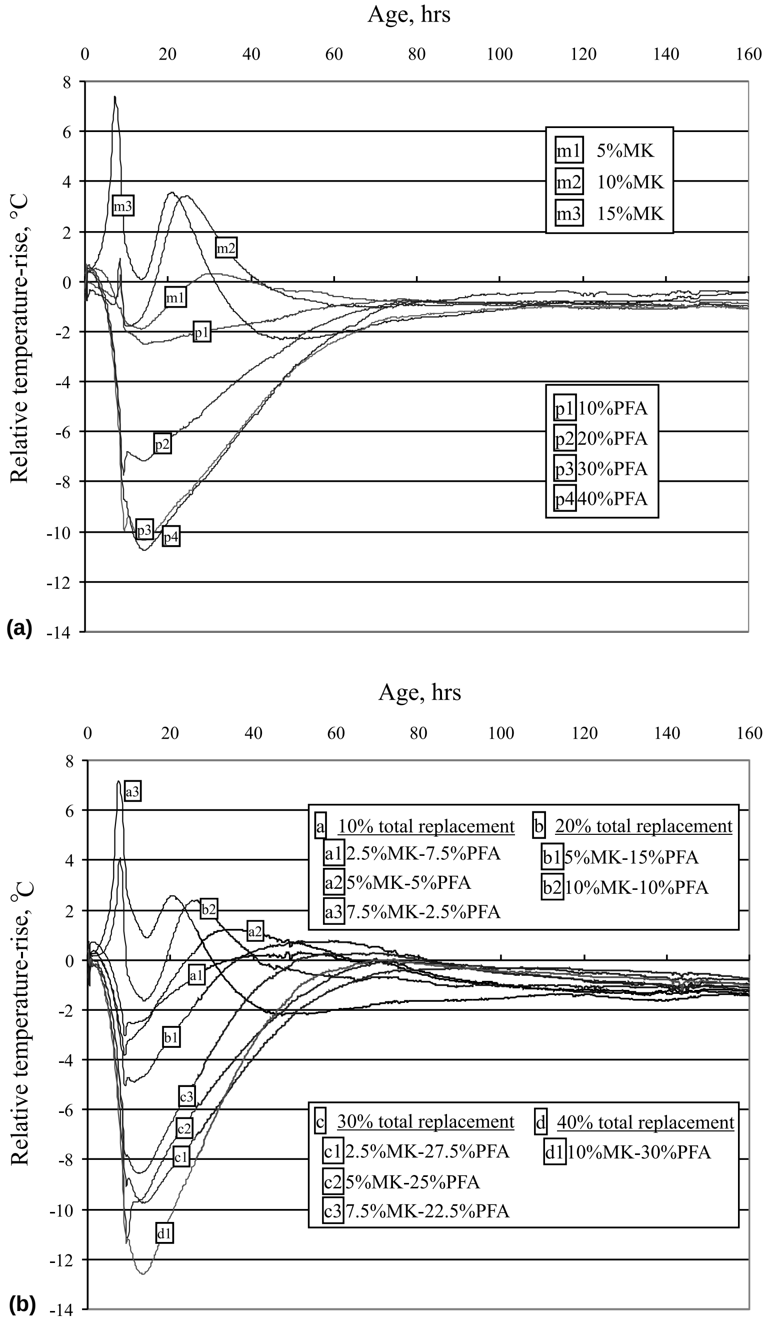

Fig. 6. (a) Relative temperature-rise of MK and PFA blended mortars (relative to 100% PC mortar = 0) versus time; (b) Relative temperature-rise of MK–PFA–PC blended mortars (relative to 100% PC mortar = 0) versus time.

time of appearance of this peak being earlier as the MK content increases (5% MK-20.8 h, 10% MK-25.8 h, 15% MK-31.7 h). Interestingly Frias et al. [4] also observed a second peak at between 18 and 24 h on plots of relative heat evolution against time for their MK (10% and 30%)-PC mortars that was not observed in the SF–PC mortars. They attributed the first peak at 6–10 h to the reaction of amorphous silica from the MK with CH (to form C–S–H gel) and the second peak at 18–24 h to the reaction of amorphous alumina from the MK with CH (to form C_2ASH_8 and C_4AH_{13}). The current authors consider this as unlikely; particularly as the MK–PC system contains sulphate, which very rapidly reacts with CH and alumina to form ettringite. This has been demonstrated by the work of de Silva and Glasser [16], who showed that the presence of sulphate in hydrating MK–CH systems significantly modifies the heat evolution profile. Without sulphate a broad heat evolution peak occurs at 6–10 h and a very broad shallow heat evolution peak occurs at between 18 and 72 h. Both peaks are attributed to hydrate formation (C–S–H and calcium aluminate hydrates). When sulphate is present a sharp heat evolution peak (attributed to ettringite) occurs at 6 h followed by a broader peak at 8–12 h (attributed to C–S–H and C_4AH_{13} formation) and a further broad peak (attributed to transformation of ettringite to monosulphate) at about 24 h. An alternative explanation of the current observations is that the initial rapid reaction of MK with CH is retarded, possibly due to formation of ettringite at the MK particle surfaces. The subsequent breakdown of this inhibiting layer at a later stage then allows further reaction of CH with MK, producing a second peak. This is a similar phenomenon to that which occurs in hydrating PC alone [14,15], where cements with a substantial C_3A content ($>\sim 12\%$) show a secondary heat evolution peak between 24 and 48 h due to breakdown of the ettringite and its transformation to monosulphate, allowing renewed hydration of C_3A. In the current work increased alumina is provided by the MK. The fact that this second peak occurs earlier, with increasing MK content, is explained by the fact that the sulphate concentration in the pore solution, necessary to maintain stability of the ettringite phase, would drop more rapidly as MK levels increased. The same phenomenon is apparent for the ternary blends (see Fig. 6(b)) where the second peak is more pronounced and occurs earlier as the MK to PFA ratio for this ternary blend increases (e.g. 10% replacement – a3, a2, a1).

4. Conclusions

The temperature-rise in MK–PC mortars (above 5% MK and up to at least 15% MK) is greater than that in equivalent PC mortar (other than at very low MK levels). This is attributed to increased rates of heat evolution during the first few hours of hydration resulting from the combined effect of pozzolanic reaction and accelerated PC hydration.

The temperature-rise in PFA–PC mortars is less than that in equivalent PC mortar. This is attributed to the dilution of the PC by the PFA coupled with the latter's negligible pozzolanic activity during the early stages of hydration, thus reducing both the rate of heat evolution and the total heat evolved.

When combined in ternary MK–PFA–PC mortars, the effects of MK and PFA on the temperature-rise tend to compensate for each other. Thus for example the temperature-rise for the 10% MK–10% PFA mortar is the same as that of the control.

The time to peak temperature increases for the blend mortars as the MK and PFA contents increase, although for a particular substitution level MK produces a greater delay than PFA.

MK has very little effect on the total (160 h) heat evolved by MK–PC mortar for MK levels up to 15%. In contrast PFA produces a marked fall in the total heat evolved by PFA–PC mortar. In MK–PFA–PC ternary mortars increasing the ratio of MK to PFA produces an increase in the total heat evolved.

The temperature-rise of MK–PC mortars with time, relative to the equivalent PC mortar, shows two well-defined peaks, a very sharp peak at 6–10 h and a much broader peak beyond 24 h. The former peak is attributed to the combined effect of pozzolanic reaction of MK plus acceleration of cement hydration. A possible explanation for the second peak, which occurs at longer times as the MK content reduces, is the breakdown of ettringite to monosulphate and renewed hydration of MK. However detailed analytical work would be required to conclusively establish this as the actual mechanism.

Although under fully adiabatic conditions there is probably little difference in the temperature-rise produced by either PC or MK–PC binders, under conditions where heat generation could give problems, employing a ternary MK–PFA–PC binder would be advantageous in reducing the temperature-rise.

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