

Accelerating Early Strength Development of Concrete Using Metakaolin as an Admixture

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A study of metakaolin (MK) as an early strength accelerator for Portland cement (PC) and Portland Cement-Pulverised Fuel Ash (PFA) concrete (PC-PFA) has been carried out. This paper reports strength development, particularly at early curing ages, to evaluate the effectiveness of MK as an accelerating admixture. This is supported by analysis of the heat evolution using a calorimeter. The Portland cement concrete covering five different addition levels of MK (1 % – 5 %) to binder and with four water to cement ratios of 0.3, 0.4, 0.5 and 0.6 (0.4 and 0.5 for PC 60 % – PFA 40 % concrete) were cured in water up to 28 days. The compressive strengths were evaluated at 1, 3, 7, 14 and 28 days. The compressive strength development of the concrete at various curing times is compared with control concrete (PC only). It is found that MK contributes significantly to early strength development as an accelerating admixture for PC and PC-PFA concrete.

Keywords: Metakaolin, fly ash, strength development, accelerating admixture, heat evolution, pozzolan.

INTRODUCTION

Concrete will certainly be the main material for Mankind to create the future built environment. With the significant growth of concrete industries over the past 40 years, customer demand has changed [1]. High strength at an early age is desirable in construction, especially in cold weather, to reduce the length of time in which temporary protection is required [2 – 4]. In the meantime, the desire to reduce production energy costs and improve Portland cement properties and the concern over environmental issues have led to the world-wide utilisation of by-product supplementary cementing materials as partial cement replacements for concrete production. These mainly pozzolanic materials include PFA [5], MK [6 – 9], condensed silica fume (CSF) [10, 11], plus latently hydraulic materials such as ground granulated blast-furnace slag (GGBS) [5]. However to satisfy increasing customer expectations for durable environmentally friendly materials, concrete mixes with even better and higher performance are needed.

Modern concrete is more than simply a mixture of mineral components having very specific characteristics that give specific properties to concrete, but it also can contain chemical admixtures that have even more specific effects. Modern concrete is a very complex chemical material where mineral products and amorphous products (not solely ground clinker and calcium sulphate); interact with organic molecules or polymers. These organic molecules are specially developed to highlight certain characteristics of concrete or correct certain deficiencies of current cements because current cements can present some deficiencies in some of their applications [12].

This paper discusses and presents results of research performed in the School of Technology at the University of

Glamorgan to investigate the use of MK as an accelerating admixture for production of high early strength concrete.

MATERIALS, MIXTURES AND TEST METHODS

Materials and mixtures

Portland cement obtained from Blue Circle and complying with BS 12: 1991 was used. Metakaolin was supplied by Imerys. Table 1 gives the chemical compositions of MK, Portland Cement (PC), and the Pulverised-Fuel Ash (PFA) used in this study. The fine aggregate was natural sea-dredged sand of grading M-F complying with BS 882: 1983. The coarse aggregates employed were 10 mm and 20 mm crushed limestone.

Table 1. Chemical composition of Portland Cement (PC), Metakaolin (MK) and Pulverised Fuel Ash (PFA)

	PC	MK	PFA
	Specific surface (m ² /kg)		
	380	12000	500
	Composition (%)		
SiO ₂	20.1	52.0	51.7
Fe ₂ O ₃	2.3	4.6	10.4
Al ₂ O ₃	4.4	41.0	27.1
CaO	63.4	0.1	2.6
MgO	2.3	0.2	1.6
SO ₃	3.2	–	0.8
Na ₂ O	0.14	0.1	1.2
K ₂ O	0.67	0.6	3.1
TiO ₂	–	0.81	–
LOI	2.81	0.6	4.0

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A polymeric sulphonated naphthalene based dark liquid superplasticizer, Cormix SP1, was added to the mixtures PC-MK with $w/b = 0.3$ at a dosage of 0.7 % of the weight of the total binder. This was done in order to restore the workability lost by reduction in the water content below that for the concretes with $w/b = 0.4$, and thus enable investigation of the effect on the workability caused by adding small amounts of MK as an admixture in concrete mixes.

Trial mixes were carried out to achieve the required workability range for the different mixture compositions. Trial mixes were made for the control and for the mixes with 5 % of MK admixture and control slump tests were carried out. After this the final mix mixtures were designed for both types of mixes (PC and PC-PFA) and are shown in Table 2. The PC-PFA mixes all consisted of 60 % PC plus 40 % PFA plus the various levels of MK admixture.

Table 2. Mixtures

Mix	w/b	Binder content, kg/m ³	MK addition (%)
PC	0.3	600	0; 1; 2; 3; 4; 5
	0.4	450	0; 1; 2; 3; 4; 5
	0.5	360	0; 1; 2; 3; 4; 5
	0.6	300	0; 1; 2; 3; 4; 5
PC-PFA	0.4	450	0; 1; 2; 3; 4; 5
PC-PFA	0.5	360	0; 1; 2; 3; 4; 5

In total, 36 mixes, were investigated – 6 mixes for each of the four w/b ratios of 0.3, 0.4, 0.5 and 0.6 for PC-MK concrete, and 6 mixes for each of the two w/b ratios of 0.4 and 0.5 for PC-PFA-MK concrete. Five admixture levels from 1 % to 5 % by mass of PC and PC-PFA were employed in addition to the controls (PC only and PC-PFA only). As mentioned above, a superplasticizer was used for mixtures PC-PFA with $w/b = 0.3$ in order to achieve an adequate level of workability. The PC, PFA and MK were thoroughly blended together by hand until a uniform colour was achieved.

Specimen preparation and strength testing

Concrete was cast in 100×100×100 mm steel moulds and compacted by vibration. After casting, cubes were covered with cling film to prevent water loss and after 24 hours the specimens were demoulded and placed in a curing tank filled with water. The temperature of the water was 20 °C ±1 °C. Test cubes were made from fresh concrete in compliance with BS 1881-108:1983 [13].

Specimens were tested for compressive strength after 1, 3, 7, 14 and 28 days of curing. All cubes were tested according to BS 1881 [14]. The cubes were tested under a continuously increasing load at a nominal rate of 0.3 N/(mm²/s). Three cubes were tested for each curing time and composition and the average strength determined.

Calorimetry test

Heat of hydration can be measured under adiabatic or isothermal conditions [7]. Measurement of heat of hydration in this study was carried out under isothermal

conditions (25 ±0.1 °C). The calorimeter used was a thermoelectric conduction type, interfaced to a computer by a data acquisition system.

Each test was conducted on a 5 g sample of pure PC (and MK as an admixture added in the range 1 % to 5 %) placed in the calorimeter test tube. The test tube was placed in the calorimeter, which was then closed. The sample was held for approximately 1 hour to attain temperature equilibrium. After equilibrium was reached 2.5 g of water was injected into the test tube (giving a w/c ratio of 0.5). The data acquisition system was initiated at the same time to record the output voltage from which the heat flow in the system can be measured.

Each heat of hydration determination was carried out over a period of 72 hours.

RESULTS AND DISCUSSION

Strength results for PC mixes

From the results obtained during this part of the study, it is apparent that MK admixture generally produces a beneficial effect on compressive strength at all ages and w/b ratios. Table 3 gives the results of the average compressive strengths obtained from testing three samples (cubes) at various curing periods for PC concrete mixes with various levels of MK admixture.

Table 3. Compressive strength (MPa) for PC mixes

W/b ratio	MK addition (%)	Curing ages (days)				
		1	3	7	14	28
0.3	Control	27.8	54.5	64.0	70.5	79.7
	1	29.6	55.1	65.4	70.6	79.8
	2	30.9	55.9	65.8	71.0	79.0
	3	32.0	58.1	67.7	74.6	84.1
	4	32.2	57.9	73.7	76.8	85.7
	5	35.1	57.9	74.1	81.9	88.2
0.4	Control	13.6	33.6	44.7	54.3	60.5
	1	14.7	36.6	48.1	58.4	63.3
	2	16.9	37.8	48.7	60.5	65.2
	3	20.7	39.3	51.3	61.3	67.7
	4	24.4	44.9	55.4	70.2	73.3
	5	26.1	45.2	58.7	70.8	73.6
0.5	Control	8.3	24.8	34.0	41.0	46.9
	1	8.8	26.3	34.0	41.1	47.3
	2	9.8	27.0	34.8	44.8	48.0
	3	10.8	27.9	36.8	45.5	50.4
	4	11.5	28.4	37.0	46.2	50.9
	5	12.1	29.1	39.6	50.2	53.5
0.6	Control	4.6	13.9	19.1	23.3	26.9
	1	5.3	15.2	21.3	25.6	30.7
	2	5.9	16.5	22.6	28.0	32.1
	3	6.9	18.6	24.9	30.0	33.7
	4	7.4	20.1	27.8	34.4	37.6
	5	7.8	21.1	29.9	37.2	41.5

It has been suggested by Bai et al [15] that the acceleration of early cement hydration for concrete incorporating MK is a result of the high pozzolanic activity of MK, which both accelerates early cement hydration and rapidly consumes CH so as to form cementitious products, thus contributing substantially to cementation at a very early stage. However, the very small levels of MK addition in the current work would not be expected to produce the levels of strength enhancement achieved after 1 day solely as a result of pozzolanic activity.

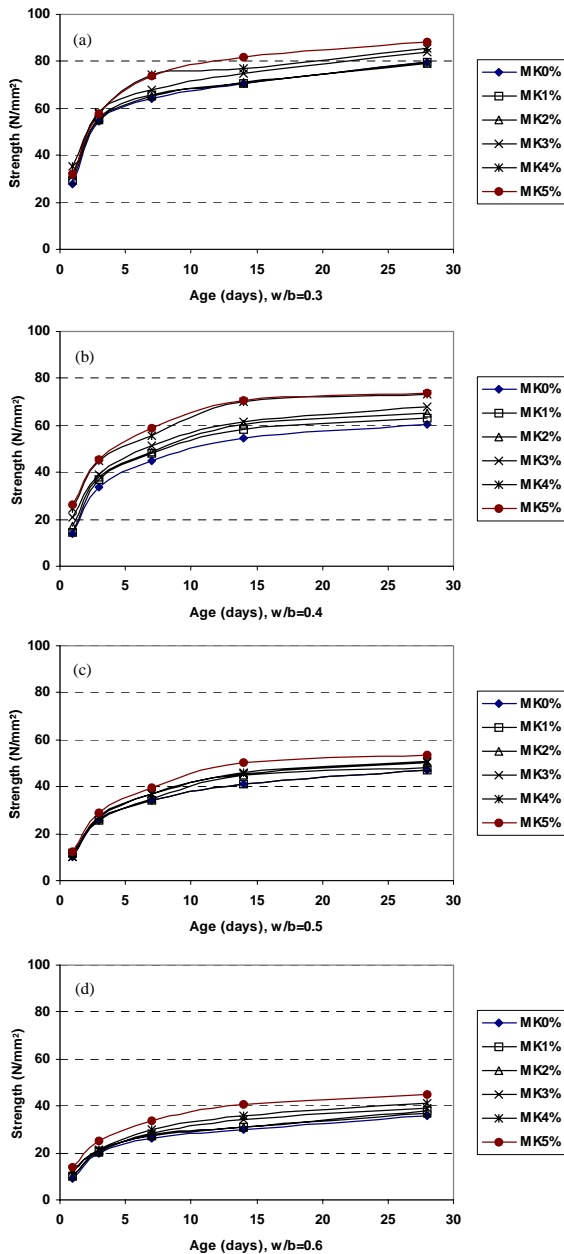


Fig. 1. Strength development for PC mixes with different water binder ratios: a – $w/b = 0.3$; b – $w/b = 0.4$; c – $w/b = 0.5$; d – $w/b = 0.6$

When pozzolanic materials are added $\text{Ca}(\text{OH})_2$ reacts with them to produce additional C-S-H gel. This forms a basis for improvement of both strength and durability of concrete, but the amounts of additional gel formed in the current work will be relatively small. The compressive strength test results for all mixes are presented in Fig. 1.

The test results show that all mixes were able to develop higher strength than the control mixes (with 0 % MK).

Fig. 1 (a, b, c, d) show the influence of the level of MK admixture on the compressive strength of the concrete at curing ages 1, 3, 7, 14 and 28 days for the four w/b ratios employed. The strength of concrete increases systematically with an increase in MK addition level at all four w/b ratios.

It is apparent from Fig. 2 (a, b, c, d), which plots relative strength versus MK content at different curing periods, that MK contributes to early strength development very effectively as an accelerating admixture for Portland cement, especially during the first few days of curing. Table 4 shows that in the early stages of curing, mixes with 5 % of MK admixture exceed the control mix strength by up to 92 %.

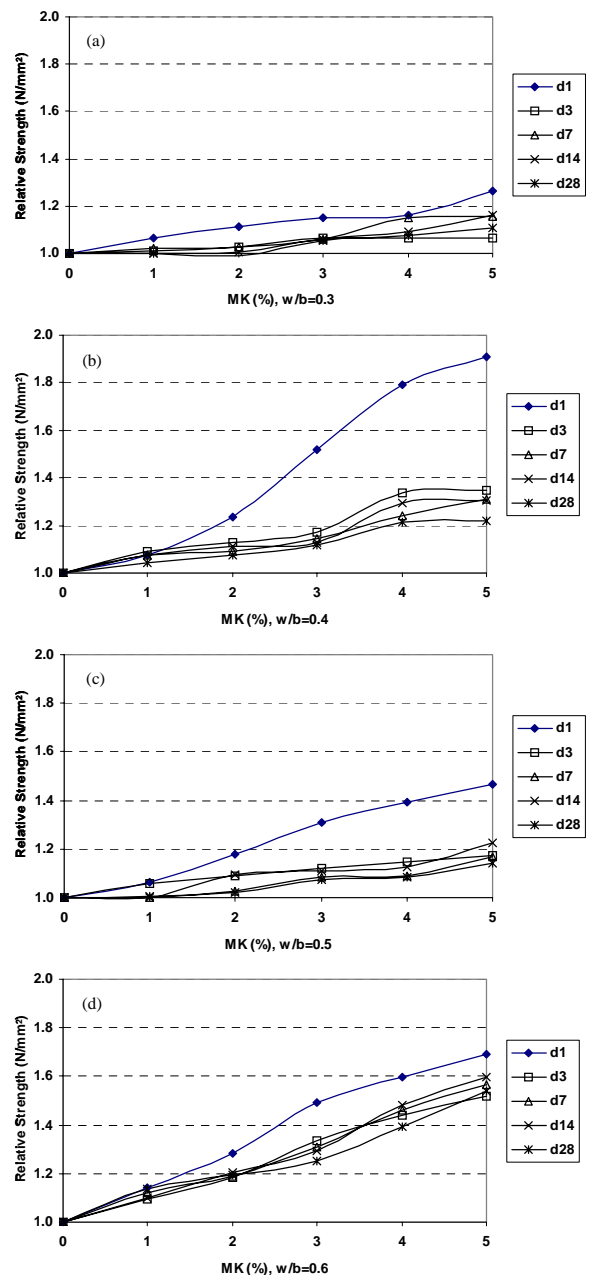


Fig. 2. Relative strength results for PC mixes: a – $w/b = 0.3$; b – $w/b = 0.4$; c – $w/b = 0.5$; d – $w/b = 0.6$

After the first few days of curing the level of strength enhancement declines, but as seen from Table 4, at between 7 and 14 days there is a further small increase in the level of strength enhancement, again followed by a decline.

For the moderately low w/b ratios of 0.3, 0.4 and 0.5 there is a decline in the enhancement of compressive strength after 7 days of curing. This decline in relative strength at longer curing periods has been suggested by Sabir et al. [16], to be due to an elementary change in the MK-CH reaction at later curing periods.

Table 4. Relative to control mixes strength results for PC mixes

W/b ratio	MK (%)	Relative strength to control at various ages (days, %)				
		1	3	7	14	28
0.3	0	100	100	100	100	100
	1	106	101	102	100	100
	2	111	102	102	100	100
	3	115	106	105	105	105
	4	115	106	115	108	107
	5	126	106	115	116	110
0.4	0	100	100	100	100	100
	1	108	109	107	107	104
	2	124	112	108	111	107
	3	152	117	114	112	111
	4	179	133	123	129	121
	5	191	134	131	130	121
0.5	0	100	100	100	100	100
	1	106	105	100	100	100
	2	118	108	102	109	102
	3	130	112	108	110	107
	4	138	114	108	112	108
	5	145	117	116	122	114
0.6	0	100	100	100	100	100
	1	115	109	112	110	113
	2	128	118	118	120	119
	3	150	133	130	129	124
	4	160	143	146	147	139
	5	169	151	156	159	153

It is found in general that the higher the water/binder ratio the higher is the influence of MK on strength development at all ages (up to 28 days). This is apparent from Fig. 2 (a) – (d), where the curves of relative strength versus MK content show an overall trend of increasing gradient with increasing w/b ratio.

This may be attributed to the high requirement of MK for free water thus inhibiting the PC hydration reaction. It should also be stressed that the MK is being applied as an admixture not as a cement replacement material. Therefore, it is not included in the calculation of w/b ratio.

The observed results confirm that metakaolin has a pronounced influence on early strength especially at high

w/b ratios where there is normally a big decline in observed compressive strength due to the high water content of the concrete.

Strength results for PC-PFA mixes

Table 5 presents the results of the average compressive strengths obtained at various curing periods for 60 % PC – 40 % PFA concrete mixes with MK levels of 1 to 5 %.

Table 5. Strength development for PC-PFA mixes

W/b ratio	MK (%)	Strength (MPa) at various curing ages (days)				
		1	3	7	14	28
0.4	0	7.1	20.5	28.3	34.8	44.2
	1	7.3	21.0	30.0	37.0	46.2
	2	8.5	21.8	32.1	40.4	48.0
	3	9.7	22.9	33.2	43.6	50.1
	4	9.9	23.0	34.3	44.2	51.3
	5	11.3	23.9	36.6	48.0	54.6
0.5	0	3.2	11.6	17.0	20.8	27.3
	1	3.2	12.5	18.0	24.2	29.9
	2	3.3	13.0	19.4	26.5	32.1
	3	4.3	13.4	20.7	29.4	34.9
	4	5.0	14.5	21.6	29.6	35.3
	5	5.5	15.2	22.2	31.0	37.1

In common with the PC mixes, the compressive strength of the PC-PFA mixes also show substantial increases with increases in metakaolin addition level, especially in the first day of curing. Table 6 presents the same data expressed as a percentage of the control concrete. After 1 day the strength of the mix with w/b ratio 0.4 increases by 60 % with a 5 % MK addition and the mix with w/b ratio 0.5 increases by 70 % with a 5 % MK addition.

Table 6. Relative to control mixes strength results for PC-PFA mixes

W/b ratio	MK (%)	Relative strength to control at various ages (days, %)				
		1	3	7	14	28
0.4	0	100	100	100	100	100
	1	102	102	105	106	104
	2	119	106	113	116	108
	3	136	111	117	125	113
	4	139	111	121	126	116
	5	159	116	128	137	123
0.5	0	100	100	100	100	100
	1	100	107	105	115	109
	2	103	111	114	127	117
	3	134	115	121	141	127
	4	156	124	126	142	129
	5	171	131	130	148	135

Thus the contrasting roles played by PFA and MK in the strength development, particularly at early ages, can be combined to produce effective blends for cements. At short curing times the MK admixture enhances the compressive strength significantly.

The relative strength of PC-PFA concrete incorporating the MK admixture shows higher strength than the control (PC 60 % – PFA 40 %) at all curing ages (up to 28 days) at both w/b ratios (0.4 and 0.5). However, in common with the PC concrete containing MK admixture, the initial strength enhancement declines after 1 day and there is then a second enhancement at about 14 days. The extension of significant strength enhancement up to at least 14 days could have useful practical advantage if MK is used as an accelerator of early strength.

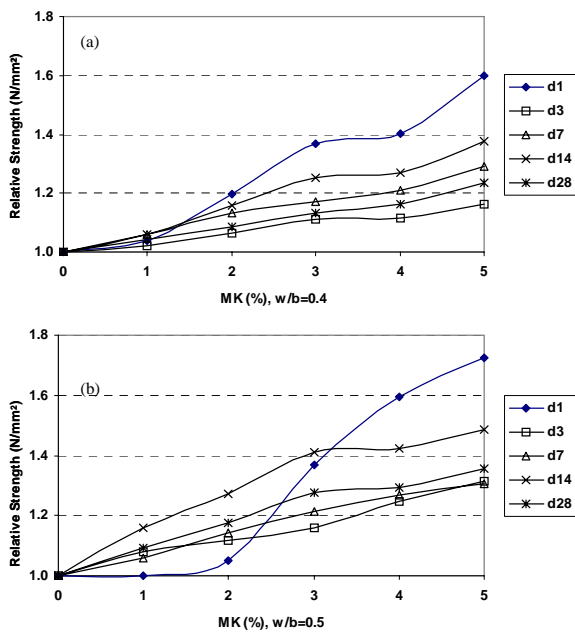


Fig. 3. Relative strength results for PC 60 % – PFA 40 %: a – $w/b = 0.4$; b – $w/b = 0.5$

Fig. 3 (a) and (b) show the change in relative strength with MK addition level. During the first day of curing there is very little strength enhancement at MK addition levels below 2 %, but significant strength enhancement above 2 % MK addition. Comparison of Fig. 3 with Fig. 2 indicates that at the same w/b ratio MK admixture provides more effective strength enhancement for MK-PFA mixes than for PC mixes. It was established for the PC mixes, that the MK is more effective at higher w/b ratio. It is therefore likely that the apparent improvement in MK effectiveness for PC-PFA mixes is a result of increased availability of free water in the PC-PFA mixes due to the reduced water requirement by the PFA.

Calorimeter results

Measurement of heat of hydration of PC pastes with MK admixture was carried out under isothermal conditions ($25 \pm 0.1^\circ\text{C}$). The calorimeter used was a thermoelectric conduction type, interfaced to a computer incorporating a data acquisition system.

From results obtained during this part of the study it is apparent that even small amounts of MK (i.e. 1 %) affect the hydration reaction heat output.

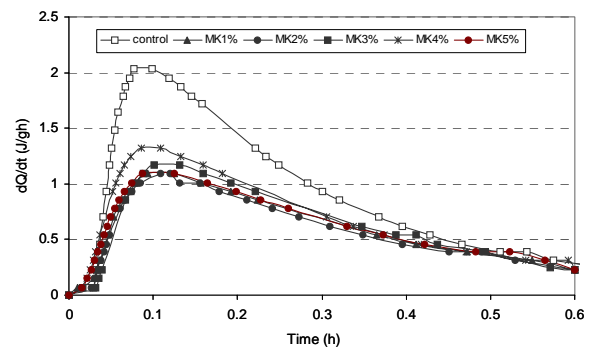


Fig. 4. First peak of hydration reaction

The first maximum in the rate of heat evolution is shown in Fig. 4 and occurs within the first 30 minutes. This immediate release of heat is attributed to exothermic surface wetting and very early reaction as the water contacts the surfaces of the cement grains [17]. This produces a gel coating and coatings of ettringite rods around C_3S and C_3A particles respectively. The current observations show that during the first hour of reaction there is a decrease in hydration heat output for mixes with MK addition. This decrease is substantial, does not appear to be systematically related to the level of MK addition and is clearly seen even for mixes with 1 % of MK. The second peak (Fig. 5) derives principally from the hydration of C_3S and formation of C-S-H gel and CH.

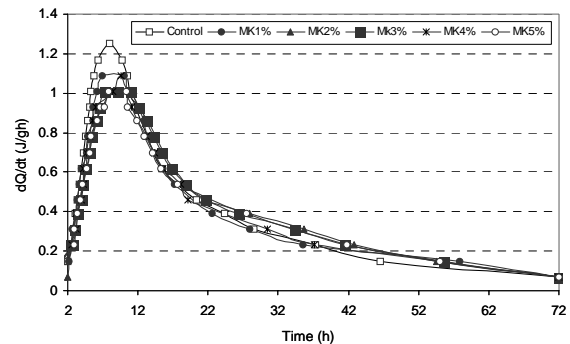


Fig. 5. Second peak of hydration reaction

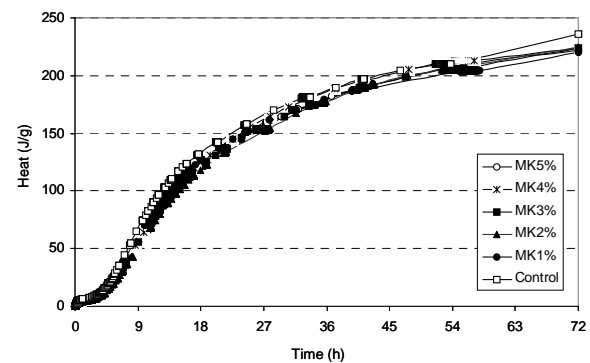


Fig. 6. Cumulative heat evolved for paste mixes with MK admixture

This peak (which starts at 2 – 3 hours after the binder was mixed with water) shows a reduction in the maximum rate of heat evolution with MK addition, although at the later stages of hydration heat evolution rates are somewhat higher with MK admixture than they are for the control.

However the cumulative heat evolved (Fig. 6) for paste mixes with MK admixture are all below that of the control within the first 24 hours and do not exceed the control up to 72 hours.

This observation is supported by previous work by Bai et al [7] who measured the temperature rise of MK-PC mortars and showed that for 5 % MK – 95 % PC mortar the temperature rise is less than that of the control. For 10 % and 15 % MK levels the temperature rise is above the temperature rise of the mortars of the control due to the greater contribution from the pozzolanic reaction of the MK.

It is of interest to note that all MK addition levels (1 % to 5 %) produce a slight increase in the rate of heat evolution relative to the control (see Fig. 5) between 15 and 55 h. Such behaviour of the MK was observed by M.Frias et al. [18], who reported that the maximum pozzolanic activity of the MK occurred between 12 and 30 h.

DISCUSSION

Clearly there is a paradox in the observed results in that strength data confirms that MK additions produce accelerated strength gain in the early stages of curing, suggesting acceleration of PC hydration, whereas calorimetry data confirm a small reduction in heat evolution suggesting that there is no acceleration of cement hydration.

Langan et al [19] studied the heat evolution of hydrating Portland cement – silica fume (PC-SF) paste at different water to binder ratios for 10% replacement of PC with SF which is much greater than the levels of MK admixture used in the current work. They reported from their observations that SF initially also acts as a retarder to the PC hydration process as a result of formation of surface layers of ultra fine SF particles on the cement grains. Water may become trapped in this layer and particularly at low w/b ratios absorption by this layer decreases the water content available for cement hydration. During the dormant period Ca ions diffuse through this layer into the pore solution but the silica fume absorbs the Ca ions and reduces the Ca ion concentration of the pore solution. At high w/b ratio this accelerates cement hydration by increasing the diffusion rate but at low w/b ratio it retards hydration due to its interference with the supply of water to the cement particle surfaces. It is suggested that MK has a similar effect which explains why the initial heat evolution peak (Fig. 4) within the first hour of hydration prior to the dormant period shows a very marked fall when MK admixture is present.

Langan et al [19] also demonstrated that at low w/b ratios silica fume prolongs the dormant period and reduces the rate of heat evolution after dormancy during the accelerating period but increases the rate after the acceleration period. This behaviour is again very similar to what is observed for MK in the current work (Fig. 5).

Between 2 and 18 hours MK reduces the rate of heat evolution (relative to the control PC paste) but between 18 and 72 hours the rate of heat evolution is greater for the paste with MK admixture relative to the PC paste.

How can the early strength of concrete containing very small additions of MK (1 – 5 %) increase substantially when there is no corresponding increase in heat evolution (in fact there is a small decrease)? Acceleration of Portland cement hydration would be expected to produce substantial increases in heat evolution to correspond with the substantial increases in early strength. Pozzolanic reaction would not be expected to produce such large increases in early strength for such low levels of MK addition. What other explanations are possible? One likely explanation is that the MK, even at these low levels of addition, has a significant influence on the chemistry and microstructure of the system.

The MK has a huge surface area and very strongly adsorbs CH. For example 2.5 % MK addition possesses the same surface area as the PC to which it is added. If the CH that is produced initially is adsorbed onto the surfaces of the MK particles it will not then nucleate at aggregate particle surfaces in the interfacial zone region, which will enable a denser initial interfacial zone to be established. There are numerous reports in the literature [20] that large crystals of CH form adjacent to aggregate grains, and that particularly large crystals or extensive deposits of CH are found in places where local bleeding has occurred. These crystals provide weak regions in the interfacial zone and tend to predominate on fracture surfaces because of their good cleavage. Also the MK reacts very rapidly with sulphate and CH to produce ettringite [21] and this may influence both the progress of cement hydration and the resulting microstructure. However, further work is needed to understand these processes.

CONCLUSIONS

The aim of this study was to assess the possibility of using MK as an accelerating admixture in PC only and PC-PFA concrete with different water/binder ratios. A series of tests on compressive strength and on hydration heat has been carried out. Comparisons have been made with the results obtained from the control mixes (MK level 0 %). Based on the results and discussion, the following conclusions can be drawn:

- MK as an admixture in PC concrete has a beneficial effect on the compressive strength at all ages and w/b ratios, but particularly at 1 day where substantial strength enhancement is obtained;
- PC-PFA concrete mixes also show significant increases in compressive strength with increase in metakaolin addition level, especially in the first day of curing. MK admixture is particularly effective in enhancing the early strength of PC-PFA concrete at w/b ratios of 0.4 and 0.5, relative to PC concrete of the same w/b ratios. This may be associated with there being a greater amount of free water available in PC-PFA concrete at early ages;
- The contrasting roles played by PFA and MK in the strength development of concrete, particularly at early ages, can be combined to produce a more effective PC-PFA blended binder by incorporating MK admixture. At short curing

times MK admixture increases compressive strength significantly and partly compensates for the reduction in strength when PC is partially replaced by PFA;

- Very small amounts of MK (even 1% of total PC) significantly reduce the cumulative hydration reaction heat output up to at least 72 hours. For the first heat evolution peak, which is mainly a result of surface wetting and initial surface reaction, there is a decrease in the rate of heat evolution and in the peak height for all mixes with MK addition. For the second heat evolution peak, which results mainly from hydration of C₃S the MK additions in the binder tend to reduce heat output in the first 12 to 15 hours, but during the later stages of hydration heat output tends to be at a rather higher level than that of the control mix. This is attributed to a contribution to the heat output from the pozzolanic activity of the MK.

Acknowledgments

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