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Absorption characteristics of metakaolin concrete

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Abstract

The water absorption (WA) by total immersion and by capillary rise of concrete containing metakaolin (MK) is investigated. Cement was partially replaced with up to 20% MK. The results show that the presence of MK is greatly beneficial in reducing the WA by capillary action. There is a systematic reduction in absorption by capillary action with the increase in MK content in concrete. This reduction is further supported by visual examination of specimens. The absorption by total immersion, however, tends to increase slightly with the increase in MK content. Between 14 and 28 days curing, there is a slight increase in absorption by total immersion and by capillary rise for all MK concretes. Correlation between the absorption characteristics, dynamic modulus of elasticity (E_d), strength and pore size distribution was conducted.

Keywords: Absorption; Capillary water absorption; Metakaolin; Transport properties

1. Introduction

In recent years, there has been an increasing interest in the utilisation of metakaolin (MK) in concrete as partial substitution or addition of cement. Due to its high pozzolanic activity, the inclusion of MK greatly improves the mechanical and durability properties of concrete [1–14]. Wild et al. [1] reported results on strength development of concrete, where cement was partially replaced with MK. The relative strength of concrete containing MK was found to increase with the increase in curing time up to 14 days before it begins to decline. Relative strength is the strength of concrete containing MK divided by the strength of the control concrete at the same curing period.

Bredy et al. [4] conducted work on porosity and pore size distribution of pastes containing MK. Pastes were prepared with MK content of 0–50% at different water to binder (w/b) ratios to maintain the same consistency. They concluded that when the MK content is below 20% the total porosity of paste decreases. Beyond 30%, an increase in porosity was found. Larbi and Bijen [14] reported that at 100 days of curing the pore volume of mortar and the threshold diameter decreases in the presence of MK. Khatib and Wild [3] found a slight increase in pore volume for pastes containing up to 15% MK as partial replacement of cement and at a constant water/(cement + MK), and this increase is dependent upon the MK content. The presence of MK, however, causes refinement in pore structure, in that pastes containing MK decrease the threshold diameter and increase the percentage of small pores [3,4]. The intruded pore volume of paste containing MK decreases with the increase in curing period up to 14 days. A slight increase in pore volume was observed between the age of 14 and 28 days [3]. After 28 days, the pore volume declines.

Table 1
Details of concrete mixes

Mix no.	Mix code	Mix proportions, % by mass of binder			Slump (mm)
		OPC	MK	SP[a]	
1	0% MK (control)	100	0	0	0
2	5% MK	95	5	0.6	5
3	10% MK	90	10	1.2	10
4	15% MK	85	15	1.8	25
5	20% MK	80	20	2.4	55

[a] Percent by weight of binder.

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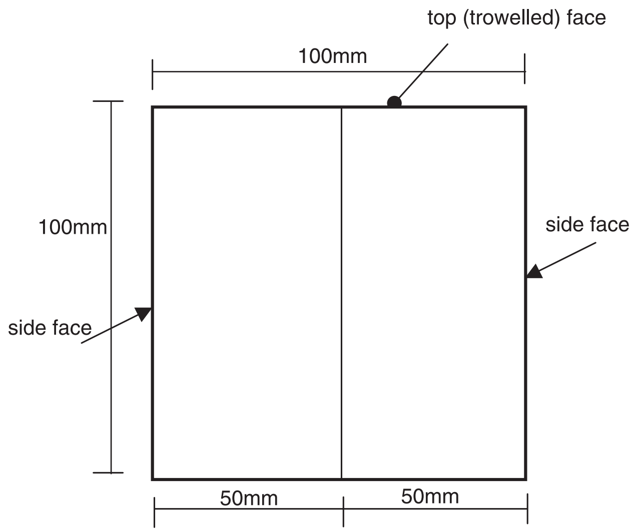

Fig. 1. Sampling of concrete specimen.

Work on calcium hydroxide consumption of MK paste and mortar, showed that the presence of MK causes reduction in the calcium hydroxide, especially during the early periods of hydration [2,3]. Sulphate resistance of mortars was found to increase when cement was partially replaced with MK up to at least a replacement level of 25% [10].

Water absorption (WA) by total (shallow) immersion is generally measured by drying a specimen to constant mass, immersing it in water for a specific time and measuring its increase in mass as a percentage of the dry mass. Capillary water absorption of concrete, however, is the phenomenon by which water is absorbed into concrete by capillary action. It involves placing a sample with one surface just in contact with water and the mass or height of water absorbed by capillary action is measured at different time intervals. The fineness of the capillary pores in concrete causes absorption of water by capillary action, hence a measure of the rate of absorption provides a useful indication of the pore structure of concrete. This phenomenon of WA by capillary action has been widely reported [14–29].

Permeability of concrete is the property that measures the flow of fluid through concrete under a pressure gradient [15], making it different from capillary WA. Permeability is of importance in water-retaining structure, whereas in structures situated aboveground such as buildings, tests on water penetration by capillary action is more appropriate to define the quality of concrete.

The use of MK in concrete in its present form is relatively a new concept. Little information is available in the literature on the absorption characteristics of concrete containing MK. Absorption by capillary action is a key durability parameter. Therefore, this study reports results on WA by capillary action and also by total immersion of concrete containing MK and the variation of absorption with curing time. The dynamic modulus of elasticity (E_d) of these concretes was also determined. Correlation between absorption, E_d and also compressive strength, pore volume and pore size distribution of previous work [1,3] was conducted.

2. Experimental

2.1. Mix constituents and materials

In order to investigate the influence of MK on absorption characteristics and dynamic modulus of elasticity, five concrete mixes were employed, details of which are given

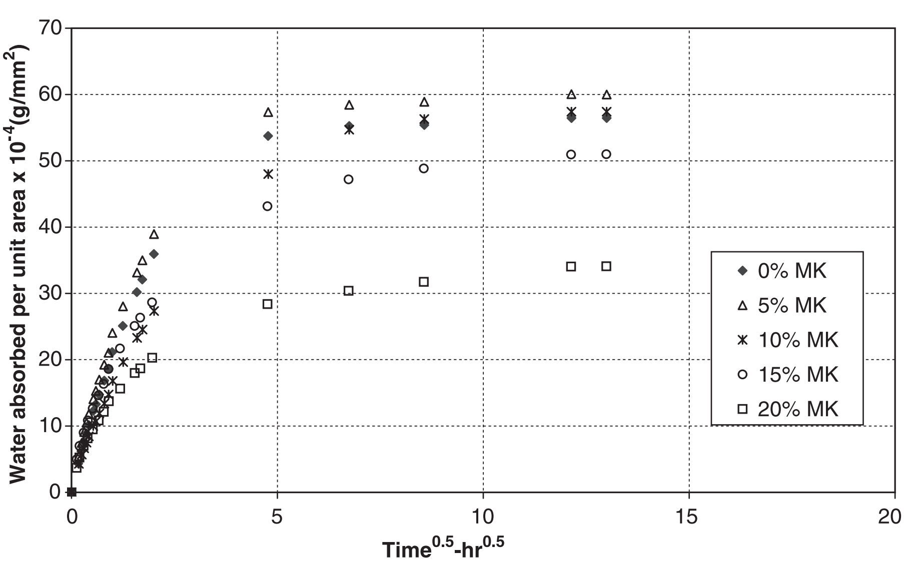

Fig. 2. Water absorbed per unit area for concrete containing 0%, 5%, 10%, 15% and 20% MK at 1 day curing.

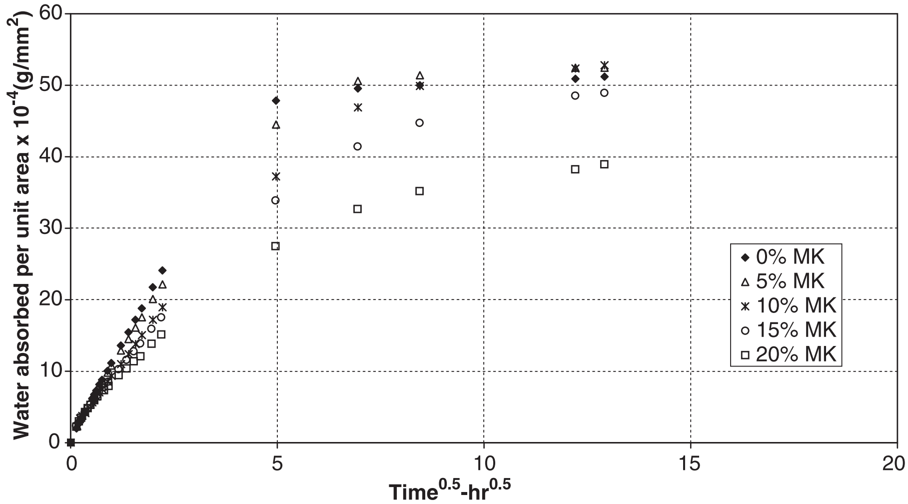

Fig. 3. Water absorbed per unit area for concrete containing 0%, 5%, 10%, 15% and 20% MK at 7 days curing.

in Table 1. The control mix 0% MK contained only Portland cement (OPC). The mix had a proportion of 1 (binder):2.3 (fine aggregate):3.4 (coarse aggregate). The binder consists of cement and MK. In the other four mixes, the OPC was partially replaced with, respectively, 5%, 10%, 15% and 20% MK (by weight). The w/b ratio was kept constant at 0.45 for all mixes. The binder consists of cement and MK. Superplasticizer (SP) with a solid/liquid ratio of 40:60 was added to mixes containing MK. The dosage of SP increased with the increase in MK content to compensate for the loss in workability. All mixes had low workability despite the presence of superplasticizer and the slump ranged from 0 to 30 mm. The compositions of the cement and the MK are given elsewhere [1,3,10]. The fine aggregate complied with Class M (medium) of BS882, 1983, and the coarse aggregate was 20-mm nominal maximum size crushed and washed gravel.

2.2. Casting and curing

Concrete cubes of 100 mm in size were cast in steel moulds for the study of absorption characteristics, whereas prisms of dimensions 100 × 100 × 500 mm were cast for the determination of the dynamic modulus of elasticity. Soon after casting, specimens were placed in a mist room at 20 ± 1 °C and about 95% relative humidity. The specimens were demoulded after 24 h from casting and then placed in water at 20 ± 1 °C until the time of testing. Testing was conducted at 1, 7, 14, 28 and 90 days from the time of casting.

2.3. Testing

2.3.1. Absorption

After the required curing period, each cube was cut into two slices as shown in Fig. 1. The area of each half section

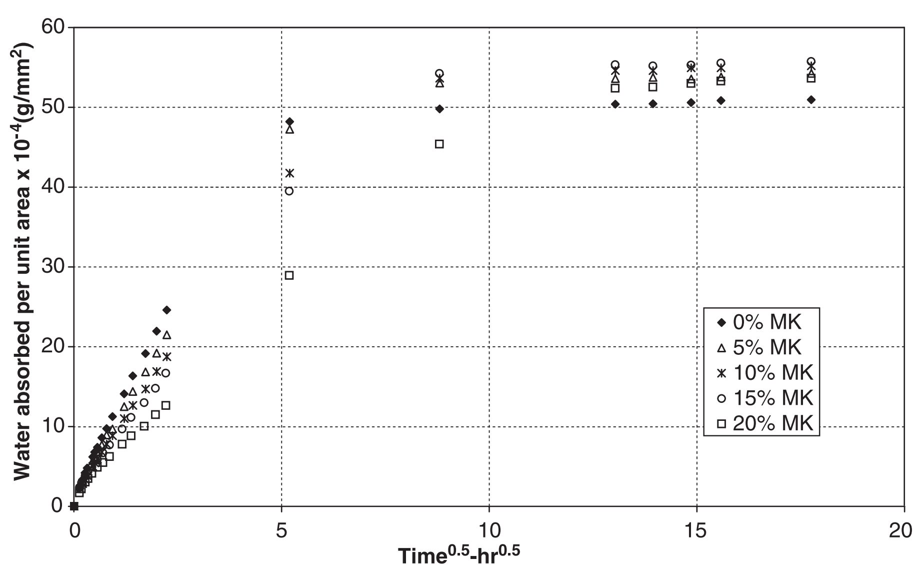

Fig. 4. Water absorbed per unit area for concrete containing 0%, 5%, 10%, 15% and 20% MK at 14 days curing.

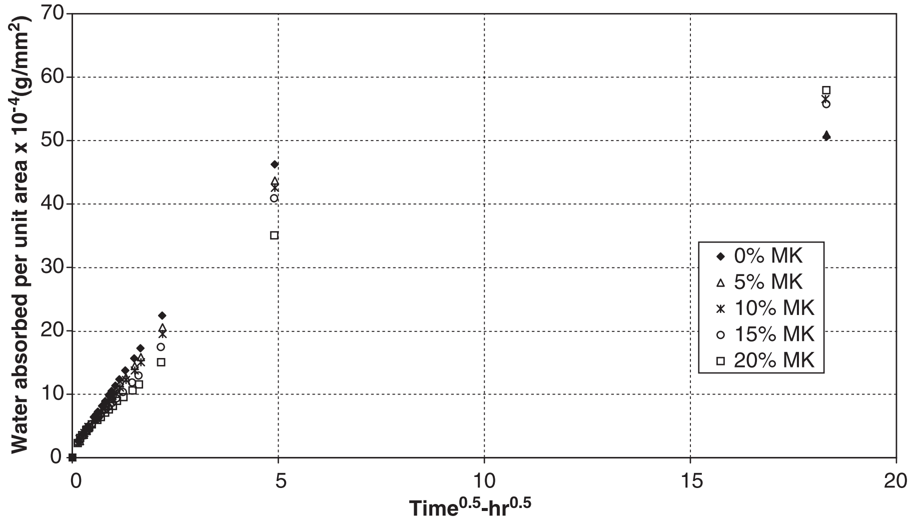

Fig. 5. Water absorbed per unit area for concrete containing 0%, 5%, 10%, 15% and 20% MK at 28 days curing.

is 100 × 100 mm and the thickness is approximately 50 mm.

After the sampling procedure, samples (i.e., slices) were dried in an oven at 100 ± 5 °C. This drying technique may lead to an overestimation of total porosity, and oven drying at 60 °C would estimate the porosity of concrete more realistically. However, the relative porosity of different concretes should not alter, provided that one drying method was adopted [29]. The total period of drying was about 48 h. The specimens were then cooled in an airtight container at 20 ± 1 °C before testing.

For the determination of WA by total immersion, the dry mass (M_d) for each sample was recorded and then totally immersed (shallow immersion) in water at 20 °C until they achieved a constant mass (M_s). M_s was taken as the saturated mass. This took up to 48 h. The WA was then calculated by the following formula:

$$\mathrm{WA}\ (\%) = 100 \times \left(\frac{M_s - M_d}{M_d}\right) \tag{1}$$

The capillary rise test was conducted at 20 ± 2 °C and 60 ± 5% RH. Dried slices were placed on supports in a shallow tray and water was added slowly until the water level was about 1.5 mm above the base of the sample in contact with water (the head of the arrow in Fig. 1 indicates the face of the slice in contact with water). The increase in sample mass was measured at regular intervals. The mass of water absorbed per unit area was plotted against the square root of time. The initial slope

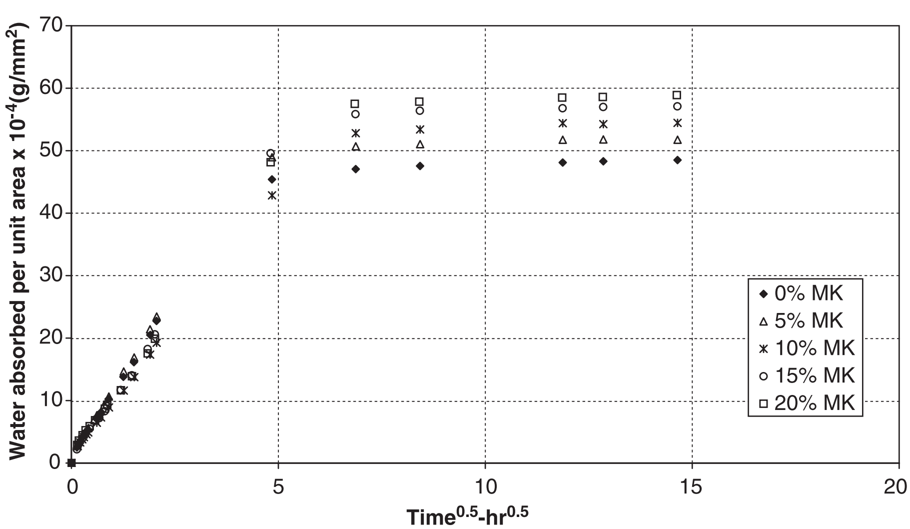

Fig. 6. Water absorbed per unit area for concrete containing 0%, 5%, 10%, 15% and 20% MK at 90 days curing.

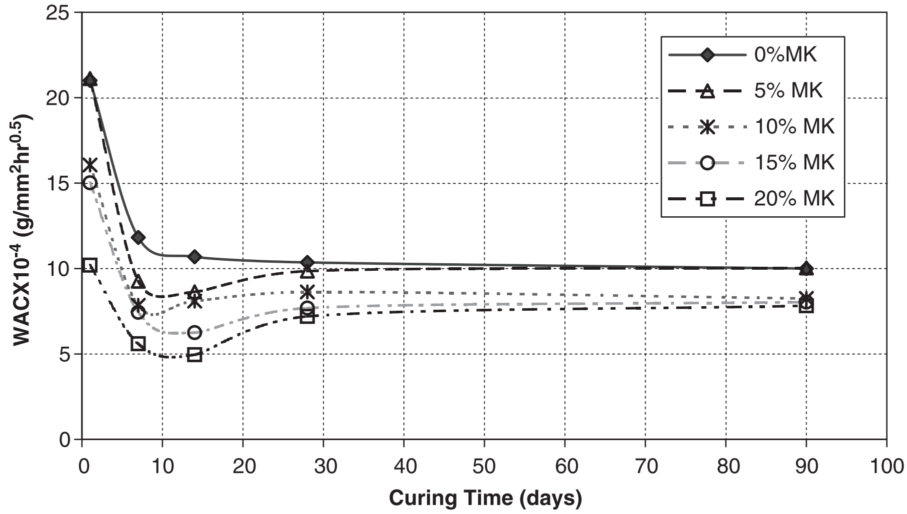

Fig. 7. Variation of WAC with curing time for concrete containing 0%, 5%, 10%, 15% and 20% MK.

of the line is taken as the water absorption coefficient (WAC). Further details about the test are reported elsewhere [25].

For each of the absorption tests (WA and WAC), two slices were tested at each age and the average was taken to represent the absorption value. The absorption values were within ±5% of the average.

2.3.2. Dynamic modulus of elasticity

The prisms were removed from water after the required curing time. Their natural frequency was measured by the use of Erudite resonant frequency apparatus and the dynamic modulus of elasticity (E_d) was determined according to BS1881, Part 77, 1977.

3. Results and discussion

3.1. Water absorption coefficient

Fig. 2 presents the water absorbed per unit area versus square root of time for all mixes employed at 1 day curing. With the exception of the 5% MK mix, there exists a systematic decrease in the slope of the linear part of the curve with the increase in MK content, indicating that the presence of MK decreases the absorption by capillary rise.

The water absorbed per unit area against square root of time for all mixes at 7 days curing is presented in Fig. 3. As the MK content in concrete increases, the slope of the linear

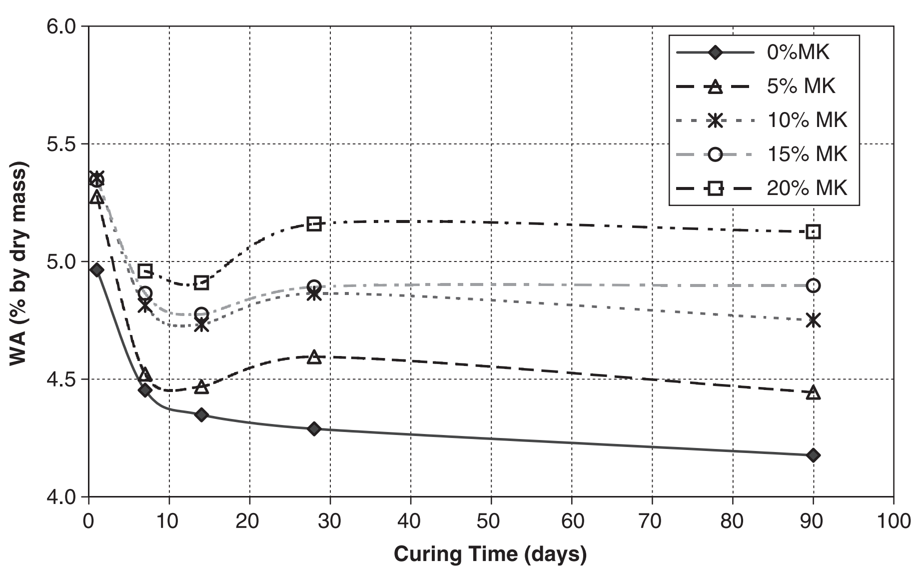

Fig. 8. Variation of WA with curing time for concrete containing 0%, 5%, 10%, 15% and 20% MK.

Table 2
Dynamic modulus of elasticity (E_d, in kN/mm^2) for concrete mixes

Mix no.	Mix code	Curing time (days)				
		1	7	14	28	90
1	0% MK	33.8	47.1	48.3	49.8	51.5
2	5% MK	28.5	46.6	48.6	50.1	51.5
3	10% MK	31.7	46.1	48.7	50.3	51.7
4	15% MK	27.2	46.9	49.3	51.0	52.2
5	20% MK	31.6	46.1	49.3	50.9	52.3

part of the curve decreases, indicating a lower rate water uptake in the presence of MK. In Figs. 4–6 the water absorbed per unit area versus square root of time is shown for all mixes at, respectively, 14, 28 and 90 days curing. The slope of the linear part of the curve generally tends to show a systematic decrease as the percentage of MK increases in the concrete mix. At 90 days curing, the difference in the slope for the various mixes is reduced. On the other hand, there is no definite trend in the nonlinear part of the curves in Figs. 2–6, caused by the presence of MK. During the early stages of hydration (e.g., 1 day curing) and when specimens nearly reached saturation, there is a large difference in the water absorbed per unit area for the different mixes. At the later stages of hydration (i.e., 90 days curing), however, this difference is greatly reduced. The nonlinear part of the curve depends on the diffusion of moisture within the concrete porous system.

The slope of the linear part of the curve in Figs. 2–6, which is termed as the WAC is plotted against the curing period for all the concrete mixes as shown in Fig. 7. As stated earlier the WAC decreases as the percentage of MK in the mix increases. In the control mix, the WAC decreases with the increase in the curing time. This decrease is sharp up to about 14 days curing. After 14 days and up to at least 90 days, the decrease in WAC in greatly reduced. The behaviour is not the same for mixes containing MK. The WAC decreases sharply with the increase in curing time up to about 14 days as for the control mix. However, an increase in WAC is observed after this curing period and up to 28 days for all MK mixes. This, and as will be discussed later, may be attributed to the change in phase composition during this period. The rate of increase is greatest at 20% MK replacement level. Despite this increase, the WAC is lower for mixes containing MK. Between 28 and 90 days curing, there is hardly any change in WAC values. At 90 days of curing, concrete containing between 10% and 20% MK still shows a reduction in WAC compared to the control mix. Larbi and Bijen [14] reported a decrease in WAC of more than 50% for concrete containing 20% MK at 100 days of curing; however, there are data available to describe the variation of WAC with time. In the present investigation this amount of decrease is only observed at 14 days of curing, whereas at 90 days there is only 20% decrease.

The beneficial effect of MK on reducing the capillary WA is apparent when the specimens are visually examined after the end of the capillary water test. The time when the test ended is the last point on the curves shown in Figs. 2–6. After the test has ended, the water can be seen on the top surface of samples for the control mix. The top surface is the surface, which is not in direct contact with water at the beginning of the test (i.e., the surface that is parallel to the side face). As the percentage of MK in the mix increases the appearance of water on the top surface is greatly reduced. In mixes containing 15% and 20% MK and after the end of the capillary WA test, no water on the top surface is observed. This suggests that there is discontinuity of pores (i.e., pore-blocking effect) when cement is partially replaced with MK.

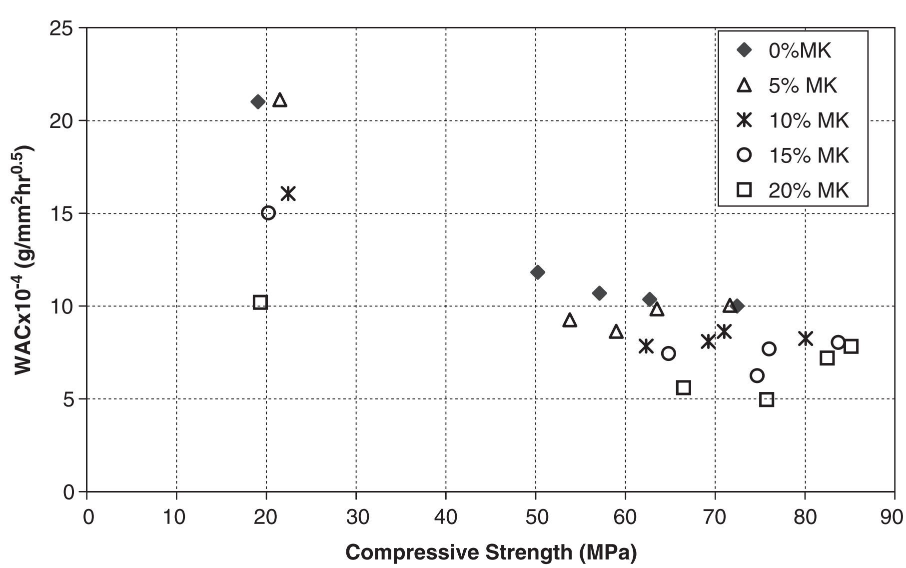

Fig. 9. Relationship between WAC and compressive strength for concrete containing 0%, 5%, 10%, 15% and 20% MK.

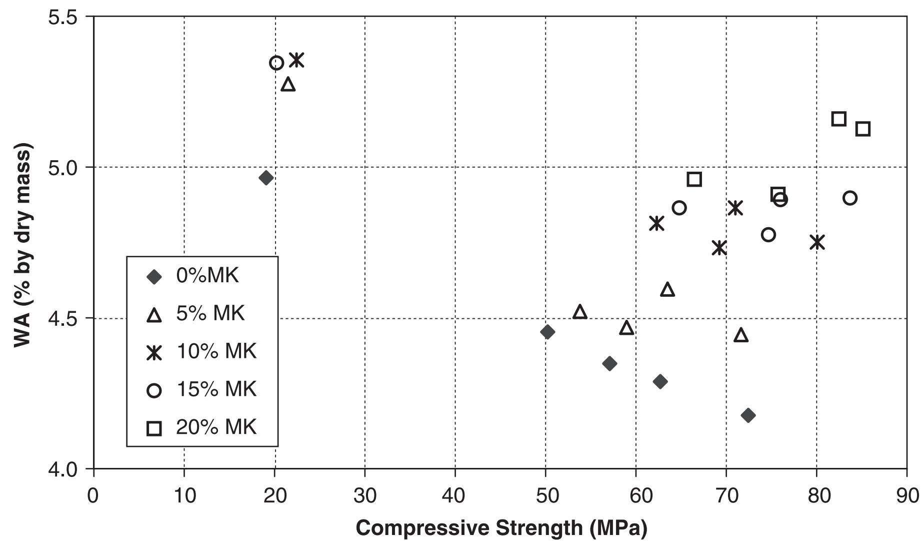

Fig. 10. Relationship between WA and compressive strength for concrete containing 0%, 5%, 10%, 15% and 20% MK.

3.2. Water absorption by total immersion

The WA by total immersion for all mixes versus curing time is presented in Fig. 8. Unlike the WAC, there tends to be a small systematic increase in WA as the percentage of MK in the mix increases at all curing times. WA values for all mixes range between 4.2% and 5.4% by dry mass.

As can be expected, the WA values for the control mix decreases with increase in curing period. The decrease in WA is noticeable during the first 7 days. After 14 days there is little decrease in WA. The variation in WA values with curing time for concrete containing MK is similar to the WAC values. A decrease in WA with curing time was obtained for all MK mixes up to about 14 days. Between 14 and 28 days curing period there is a slight increase in WA caused by the presence of MK. As will be explained later, this might be due to the formation of a denser hydration phase. After 28 days the 5% and 10% MK mixes show a slight decrease in WA, whereas at higher MK replacement levels (15% and 20%), there is little change in WA. The total pore volume of pastes containing MK is slightly higher than the control paste [3,4]. This may explain the higher absorption values of concrete containing MK. In addition, the change in WA with curing time is similar to that of pore volume [3].

3.3. Dynamic modulus of elasticity (E_d)

The values of dynamic modulus of elasticity (E_d) for all mixes at all curing times are presented in Table 2. The E_d increases rapidly with curing time up to 7 days. However,

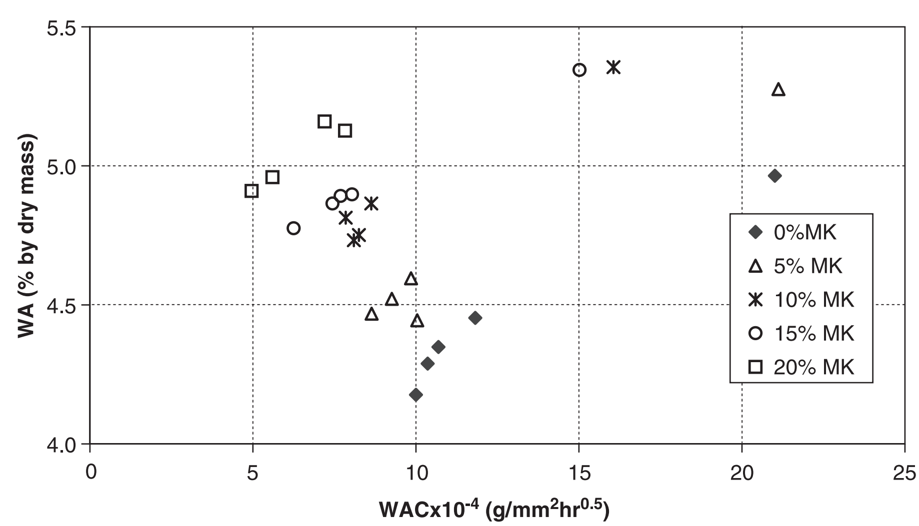

Fig. 11. Relationship between WA and WAC for concrete containing 0%, 5%, 10%, 15% and 20% MK.

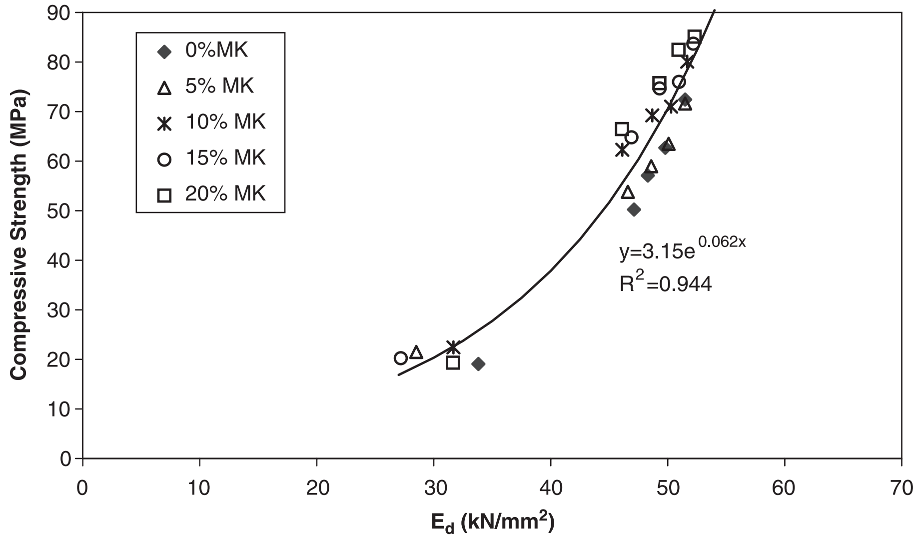

Fig. 12. Relationship between compressive strength and E_d for concrete containing 0%, 5%, 10%, 15% and 20% MK.

there is a little increase in E_d after 7 days of curing. This applies to mixes with and without MK. At 1 and 7 days curing, the values of E_d for the control mix are higher than the E_d values for the MK mixes. Beyond 14 days of curing, there is a slight increase in E_d for mixes with and without MK.

3.4. Relationship between absorption, strength and E_d

The results obtained in this work, together with results on compressive strength reported elsewhere [1], for similar mixes were used to correlate absorption, strength and E_d. Fig. 9 shows the relationship between WAC and strength, whereas Fig. 10 shows the relationship between WA and strength for all mixes considered (i.e., mixes 1–5). Generally an increase in strength is associated with a decrease in WAC when all mixes are considered (Fig. 9), whereas an increase in strength is not associated with a noticeable decrease in WA (Fig. 10). The relationship between WA and WAC is shown in Fig. 11. If all mixes are combined, there does not seem to be a good correlation between WA and WAC. However, when considering each of the mixes, it can be noticed that an increase in WAC is associated with an increase in WA. In Fig. 12, the strength versus E_d is plotted. When fitting the best exponential curve to the results, the following equation is obtained:

$$\text{Strength} = me^{nE_d} \qquad (2)$$

where $m = 3.15$ and $b = 0.062$ for the results of the current investigation with a coefficient of correlation R^2=.94, indicating that exponential relationship between strength and E_d is appropriate.

3.5. Further discussion

As with silica fume, the high fineness of MK is expected to yield denser concrete and paste matrix, in that the MK particles fill the space between cement particles [15]. The filling effect can lead to discontinuation of the capillary porosity. The large surface area of MK particles can also lead to acceleration of cement hydration [1]. It was also found that the presence of MK causes a refinement in pore structure and a slight increase in porosity of paste [3,4].

The results reported by Khatib and Wild [3] on porosity and pore size distribution are reproduced in Tables 3–5. Pastes were prepared at w/b ratio of 0.55 and cured at 20 °C. Table 3 presents the pore volume of pastes containing MK at different curing times. There is a slight increase in

Table 3
Pore volume (mm^3/g) of pastes containing MK

Age (days)	0% MK	5% MK	10% MK	15% MK
3	262.0	257.6	284.1	277.6
7	229.6	261.7	268.8	251.6
14	209.9	203.4	221.0	212.1
28	189.1	205.3	237.1	222.7
90	181.4	180.8	219.6	198.9

Table 4
Percentage of small pores (radii < 0.02 μm) of pastes containing MK

Age (days)	0% MK	5% MK	10% MK	15% MK
3	22.2	28.3	31.0	39.9
7	26.5	32.1	41.0	50.4
14	30.3	43.0	53.9	55.7
28	33.7	43.5	48.7	54.9
90	37.3	44.7	49.9	57.6

Table 5
Threshold radius (μm) of pastes containing MK

Age (days)	0% MK	5% MK	10% MK	15% MK
3	0.280	0.170	0.107	0.077
7	0.117	0.086	0.059	0.046
14	0.070	0.058	0.044	0.041
28	0.070	0.056	0.041	0.043
90	0.059	0.053	0.042	0.040

the pore volume of paste associated with the increase in MK content. This is similar to the results on WA in the presence of MK; an increase in MK content results in a slight increase in WA. It can also be noticed that pore volume decreases up to 14 days; however, a slight increase in pore volume occurs between 14 and 28 days curing due to the presence of MK. After 28 days there is a decline in pore volume. The variation of pore volume with time is similar to that of WA and WAC with time. The percentage of small pores and threshold radius of MK paste are presented in Tables 4 and 5, respectively. The evidence of pore refinement as the MK content in the paste increases is clearly indicated in both tables. The percentage of small pores (pores with radii less than 0.02 μm) increases as the replacement level of cement with MK is increased. The increase in the percentage of small pores is noticeable up to 14 days of curing relative to the control paste. Beyond 14 days, there is no further increase in the percentage of small pores. In addition, the threshold radius is noticeably reduced with the increase in MK content especially during the early period of hydration (e.g., 7 days). A smaller threshold diameter indicates pore refinement. The WA is mainly dependent on the total volume of pores, whereas WAC in addition to the pore volume is also affected by the fineness and the discontinuity of the pores. This somewhat supports the results obtained in this investigation, where a systematic decrease in absorption by capillary action (WAC) and a systematic increase in WA were found as the MK content in concrete increases.

To illustrate this dependency further, the results on porosity and pore structure in the previous investigation [3] are correlated with WA and WAC. Fig. 13 shows the relationship between pore volume of paste, WA and WAC. For the control mix (0% MK), an increase in pore volume leads to an increase in both WA and WAC. There is no trend for any of the MK mixes. However, the combined results for all mixes tend to suggest that as the pore volume of the paste increases the WA increases (Fig. 13a) whereas no obvious trend is shown between pore volume and WAC (Fig. 13b). This indicates that WA is dependent on pore volume,

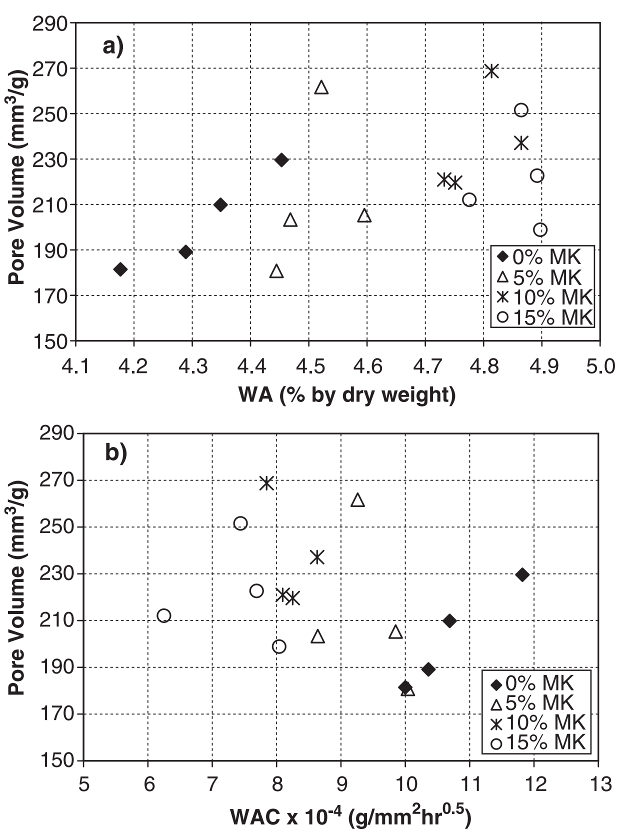

Fig. 13. Relationship between pore volume of paste and (a) WA and (b) WAC of concrete.

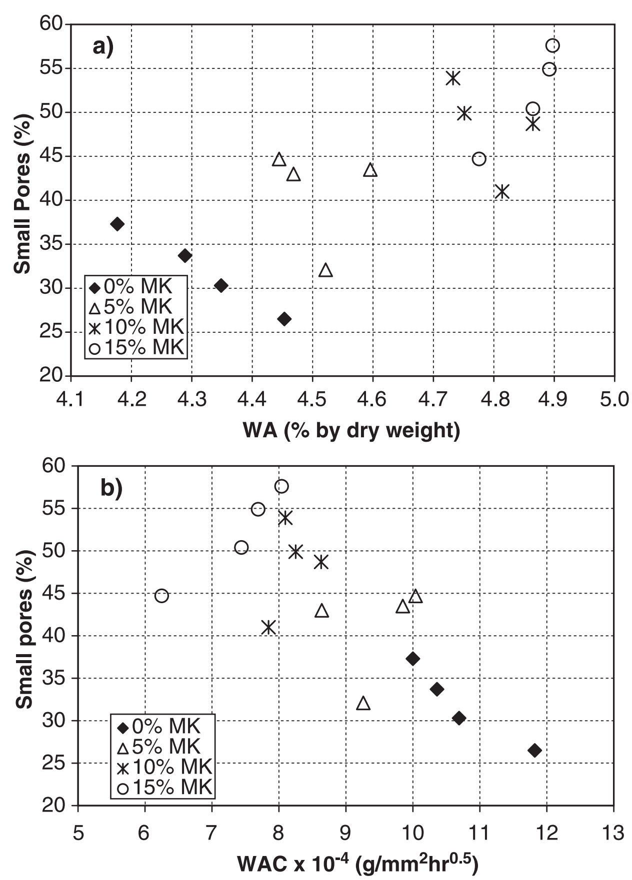

Fig. 14. Relationship between percentage of small pores and (a) WA and (b) WAC of concrete.

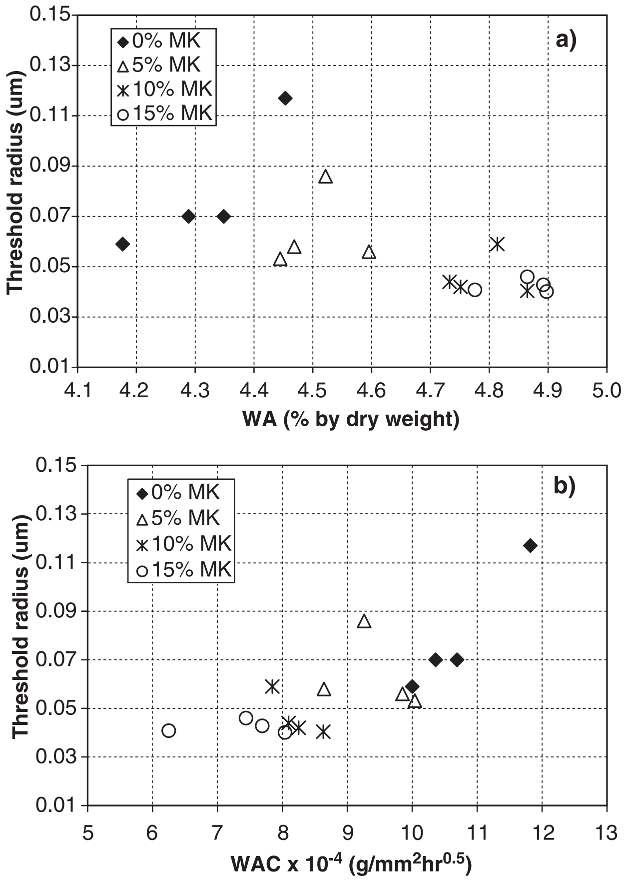

Fig. 15. Relationship between threshold radius and (a) WA and (b) WAC of concrete.

whereas this does not seem to be the case for WAC. The percentage of small pores (pores with radius less than 0.02 μm) is plotted against WA and WAC in Fig. 14 for the various mixes. When considering all mixes, Fig. 14a suggests that as WA increases the percentage of small pores increases. However, Fig. 14b indicates that an increase in the percentage of small pores leads to a reduction in WAC. The higher the percentage of small pores the finer the pore structure. The effect of pore refinement on WAC is further illustrated in Fig. 15, where the threshold radius of the paste is plotted against WA and WAC for mixes containing 0–15% MK. The threshold radius is the radius on the pore size distribution curve before which the cumulative pore volume rises sharply [3]. The combined data suggest that a smaller threshold radius leads to a lower WAC (Fig. 15b), whereas a smaller threshold radius does not necessary lead to a lower WA (Fig. 15a). A smaller threshold radius indicates a finer pore structure. It must be noted, however, that the pore volume and pore size distribution results are on paste and those for WA and WAC are on concrete. Therefore, the interfacial zone between the hydrated binder and surface of aggregates was not considered. The interfacial zone in concrete is reported to be higher than that of the pure paste [14]. A better correlation (i.e., less variation) among absorption, pore volume and pore size distribution may be obtained if the effect of the interfacial zone was taken into consideration.

The results of the present investigation and previous work [3] has also shown that the intruded pore volume of MK paste and water absorption (WA and WAC) of concrete containing MK decreases with the increase in curing period up to 14 days. Between 14 and 28 days a slight increase in pore volume and absorption is observed. This increase has mainly been attributed to the formation of a denser hydration phase during that period of curing [7]. The initial hydration products of cement and MK are C-S-H gel, C_4AH_{13} and C_2ASH_8 [4,7]. Later on hydrogarnet appears, which is denser than C_4AH_{13} and C_2ASH_8. A denser phase is associated with a decrease in solid volume and thus an increase in porosity. This may explain the increase in WA and WAC between 14 and 28 days of curing for MK concretes. With the completion of the conversion reaction the strength resumes [7] and, thereby, improvement in the microstructure. After 28 days, the porosity and absorption did not increase, indicating that the phase formation is complete.

4. Conclusions

The following conclusions are based on the results of this study:

1. The partial replacement of cement with MK reduces the water penetration into concrete by capillary action. The WA of concrete by total immersion, however, is slightly increased in concrete containing MK.
2. WA (by total immersion and by capillary rise) decreases with duration of curing for all MK concretes up to 14 days. Between 14 and 28 days of curing, there is a slight increase in WA. After 28 days of curing there is little change in absorption.
3. The absorption by shallow immersion (WA) seems to be related to the total pore volume. An increase in the total pore volume leads to an increase in WA.

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