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Effect of metakaolin on creep and shrinkage of concrete

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Abstract

The effect of metakaolin (MK) on the creep and shrinkage of concrete mixes containing 0%, 5%, 10%, and 15% MK has been investigated. The results showed that the early age autogenous shrinkage measured from the time of initial set of the concrete was reduced with the inclusion of MK, but the long-term autogenous shrinkage measured from the age of 24 h was increased. At 5% replacement level, the effect of MK was to increase the total autogenous shrinkage considered from the time of initial set. While at replacement levels of 10% and 15%, it reduced the total autogenous shrinkage. The total shrinkage (autogenous plus drying shrinkage) measured from 24 h was reduced by the use of MK, while drying shrinkage was significantly less for the MK concretes than for the control concrete. The total creep, basic creep as well as drying creep were significantly reduced particularly at higher MK replacement levels. Compared with estimated values by the CEB 90 model, total creep of all concretes was overestimated, especially in the mixes containing the higher levels of MK. For basic creep, estimates for low levels of MK were acceptable but, for the higher levels, creep was overestimated.

Keywords: Metakaolin; Autogenous shrinkage; Drying shrinkage; Total creep; Basic creep; Drying creep

1. Introduction

Mineral admixtures such as silica fume, fly ash and ground granulated blast-furnace slag improve the engineering and performance properties of concrete when they are used as a mineral additive or as partial cement replacement [1]. In recent years, there has been an increase interest in the use of metakaolin (MK) as a mineral admixture. Unlike other mineral admixtures that are mostly by-product pozzolans, which can have variable composition, purity and reactivity, the production of MK can be closely controlled and thus, a higher degree of purity and pozzolanic reactivity can be obtained [2]. Concrete containing MK has been claimed to possess enhanced engineering properties that are comparable to silica fume concrete [3]. The inclusion of MK has been reported to refine the pore structure of the cement paste matrix of concrete [4,5]. In addition, the incorporation of MK can increase resistance to acids and sulphates, reduce porosity, reduce oxygen permeability, reduce chloride ion diffusivity, prevent or minimize the risk of alkali-silica reaction and reduce the unsightly effect of efflorescence [2,6].

On the other hand, the deformation properties of concrete containing MK have not been sufficiently explored. The test results of Brooks et al. [7] indicated that the use of MK at a 15% replacement level increased the six-month autogenous shrinkage of high-strength concrete, measured from the age of one day, by about 60%. Wild et al. [8] observed an increase in chemical shrinkage of cement pastes containing between 0% and 15% MK, but at MK content of greater than 15% they observed a reduction in chemical shrinkage. Their results also showed that at 5% and 10% replacement levels, MK increased the autogenous shrinkage of cement pastes, the paste with 10% MK exhibiting the maximum autogenous shrinkage. At higher replacement levels, the autogenous shrinkage appears to be comparable to that of the control cement paste. Kinuthia et al. [9] also found that at 5% and 10% replacement levels, MK increased the autogenous shrinkage of cement pastes. While at higher replacement levels of 15% and 20%, they observed significant reduction in autogenous shrinkage. In the case of drying shrinkage, Caldarone et al. [3] found that the effect of replacing part of cement with 10% high reactivity MK was to reduce the shrinkage of concrete after 156 days of exposure to drying by 33%.

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To the knowledge of the authors, there seems to be no information available on the effect of MK on the creep of concrete.

This paper presents the results of an investigation into the effect of MK on the deformation of high-performance concrete. The elasticity, autogenous shrinkage, creep and drying shrinkage of concretes containing MK at 5%, 10% and 15% replacement levels were compared with that of control concrete having identical mix proportions but without MK.

2. Materials and mix proportions

The materials used in this investigation were ordinary Portland cement (OPC), MK, natural river sand, quartzitic gravel with a maximum size of 10 mm and a superplasticiser based on sulphonated vinyl copolymer. The chemical compositions and the physical properties of the OPC and the MK are given in Table 1. The control mix was made from OPC, whereas the other mixes were prepared by replacing part of the OPC with MK at 5%, 10% and 15% on mass-for-mass basis. A constant water/binder ratio and superplasticiser content were used for all concrete mixes. Details of the concrete mix proportions are shown in Table 2, while the workability, setting time and the 28-day compressive strength of the concretes are given in Table 3.

Table 1
Chemical composition and physical properties of cement and metakaolin

Item	OPC	Metakaolin
SiO_2	20.69	51.6
Al_2O_3	4.72	41.3
Fe_2O_3	3.06	4.64
CaO	63.76	0.09
MgO	2.08	0.16
TiO_2	–	0.83
SO_3	2.92	–
K_2O	0.61	0.62
Na_2O	0.26	0.01
LOI	0.87	–
	Fineness	
SSA (m^2/kg)	380 (Blaine)	15,000 (BET)

3. Test procedure

3.1. Autogenous shrinkage

The measurement of autogenous shrinkage was performed in two stages commencing from the time of initial set of the concrete. The setting times of the concrete as given in Table 3 were determined by the penetration resistance method [10]. For the measurement of autogenous shrinkage before demoulding, a cylindrical mould of 267×76 mm diameter with a polytetrafluoroethylene (PTFE) lining was used and the shrinkage and temperature changes were automatically recorded for approximately 24 h after casting. Details of the experimental apparatus and test procedure have been recently described by Brooks et al. [7], and the method was found to give good repeatability and comparable results to the method proposed by the Japan Concrete Institute (JCI) [11].

At the age of 24 h, the specimens were demoulded and sealed with aluminium waterproofing tape. This type of sealing was found to be very effective since the specimens showed very minimal weight loss [7]. Shrinkage was measured using a mechanical Demec gauge including the measurement of total shrinkage of companion unsealed specimens. Thus, the contribution of autogenous shrinkage to the total shrinkage of concrete could be quantified. All specimens were stored in a controlled environment of 21 ± 1°C and 65 ± 5% relative humidity throughout the duration of the tests.

3.2. Creep and shrinkage

Cylindrical concrete specimens of 267×76 mm diameter were cast for the creep tests. After being demoulded at the age of 1 day, specimens for creep and companion load-free specimens were moist cured at 20 ± 2°C for 28 days. The apparatus and method of strain measurement used for creep were similar to those described previously [12]. Creep and load-free strain were treated as additive, i.e. creep was defined as the change in deformation in excess of initial elastic strain during the application of load, corrected for strain of companion load-free specimens. At the age of 28 days, the specimens were subjected to a stress, which corresponded to an initial stress/strength ratio of 0.20 of the

Table 2
Mix proportions of the concrete mixtures

Concrete mixes	OPC (kg/m^3)	MK (kg/m^3)	Sand (kg/m^3)	Gravel (kg/m^3)	Water (kg/m^3)	SP (kg/m^3)
OPC	450	–	675	1125	126	14
MK5	427.5	22.5	675	1125	126	14
MK10	405	45	675	1125	126	14
MK15	382.5	67.5	675	1125	126	14

Table 3
Workability, setting times and 28-day compressive strength of the concrete

Concrete mixes	Workability/ slump (mm)	Initial setting time (h)	Final setting time (h)	28-days Cube strength (MPa)
OPC	100	5	7.7	87.0
MK5	30	6.42	8.82	91.5
MK10	20	6.98	9.42	104.0
MK15	5	6.45	9.31	103.5

creep cylinder strength, for a period of 200 days. The specimens for basic creep and companion cylinders were sealed with aluminium waterproofing tape prior to commencing the creep test. As in the case of autogenous shrinkage, the tests for creep were performed in a controlled environment of $21 \pm 1°C$ and $65 \pm 5\%$ relative humidity.

4. Results and discussion

4.1. Autogenous shrinkage

Figs. 1 and 2 show the results of the two stages of testing to determine the autogenous shrinkage. Fig. 1 is the early age autogenous shrinkage measured from the time of initial set to 24 h, while Fig. 2 shows the autogenous shrinkage from 24 h until the age of 200 days. Fig. 3 represents the total autogenous shrinkage, namely, the sum of shrinkages given in Figs. 1 and 2.

For the early age tests, Fig. 1 clearly demonstrates that replacement of cement by MK reduces the autogenous shrinkage as the level of replacement increases. At the end of the test period (i.e. approximately 24 h after casting), the autogenous shrinkage of the MK15 concrete was reduced by 65% compared with the OPC concrete. The reduction of autogenous shrinkage could be explained by a dilution effect which is caused by a reduction in cement content as part of the cement was replaced by MK. In addition, since the same amount of

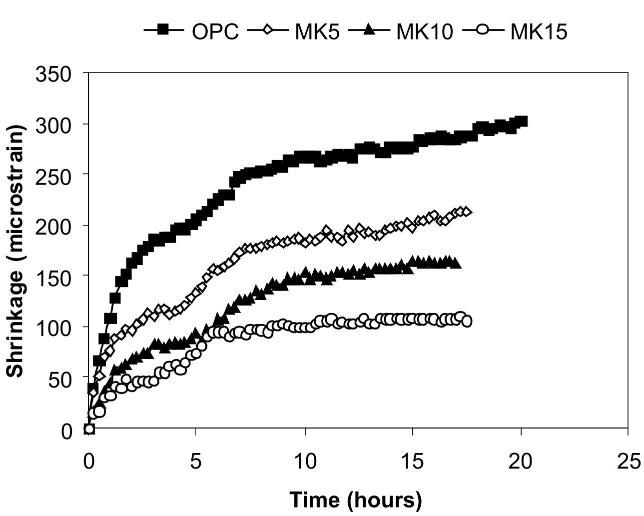

Fig. 1. Effect of metakaolin on the early age autogenous shrinkage of concrete, measured from initial set.

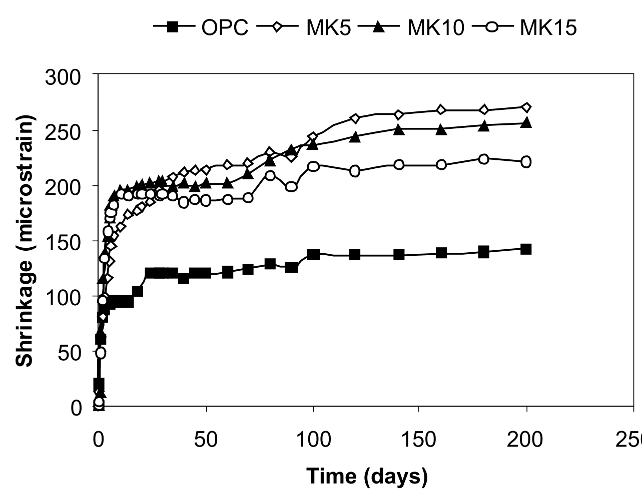

Fig. 2. Effect of metakaolin on the long-term autogenous shrinkage of concrete, measured from the age of 24 h.

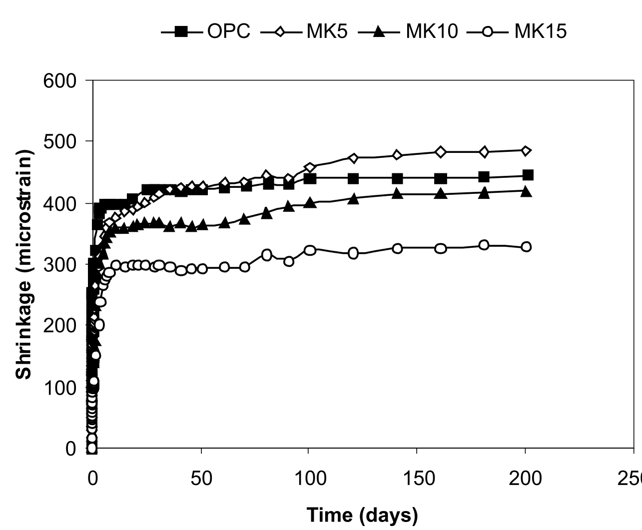

Fig. 3. Effect of metakaolin on the 200-day total autogenous shrinkage measured from the time of initial set (Fig. 1 plus Fig. 2).

superplasticiser was used in all concrete mixes, the concretes containing MK should have higher 'effective superplasticiser dosage', whose likely effect is to retard hydration reactions at early age. The test results of Kinuthia et al. [9] also indicated that, although the cement pastes containing 5% and 10% MK exhibited greater long-term autogenous shrinkage, the early age autogenous shrinkage was less than the control paste.

On the other hand, in the case of long-term autogenous shrinkage measured from the age of 24 h (Fig. 2), the effect of MK is to increase autogenous shrinkage but not as the level of replacement increases. For example, the 200-day autogenous shrinkage increases by 91%, 80% and 56%, for MK5, MK10 and MK15, respectively. Fig. 2 shows that for the MK concretes, there was a rapid increase in autogenous shrinkage from the start of the test up to the age of 14 days. This could be attributed to an acceleration in the OPC hydration and in the pozzolanic reaction of MK with calcium hydroxide. This situation has been reported for silica fume [13,14], as well as for MK [4,15]. The acceleration of the hydration process and the pozzolanic reaction will escalate self-desiccation, and due to the finer pore structure of the MK concrete [4,5], self-desiccation would induce greater autogenous shrinkage. From the age of 14 days to 4 months there was a relatively small increase in autogenous shrinkage, and after 4 months the autogenous shrinkage appears to reach a constant value. The trend of a lower autogenous shrinkage at higher contents of MK is contradictory to the previous findings of other investigators using pastes and concretes containing silica fume. It was found that at constant water/binder ratio, autogenous shrinkage of pastes and concretes increases with increasing level of silica fume [16,17]. Wild et al. [8] proposed that at higher content of MK, tetracalcium aluminate hydrate is replaced by a lower density gehlenite hydrate, producing an overall volume increase and thus reducing autogenous shrinkage.

Fig. 3 shows that the order of magnitude of the total 200-day autogenous shrinkage of the concrete measured from the time of initial set is MK5 > OPC > MK10 > MK15, with the magnitude ranging from 327 to 485×10^{-6}. In comparison with the OPC control concrete, at a MK replacement level of 5%, the 200-day autogenous shrinkage was increased by 9%. At the higher replacement levels of 10% and 15%, the autogenous shrinkage was reduced by 6% and 27%, respectively. The trend of a reduction of autogenous shrinkage at higher MK replacement levels appears to support the previous findings of Kinuthia et al. [9]. However, in their study, they found that maximum autogenous shrinkage occurred at a 10% replacement level, while at replacement levels of 15% and 20% they observed significant reduction in autogenous shrinkage. These differences could be expected because in the previous study the tests were commenced at the age of 24 h on cement pastes having a much higher water/binder ratio of 0.5 and without the use of superplasticiser.

4.2. Total and drying shrinkage

Fig. 4 shows the total shrinkage measured on the unsealed specimens from the age of 24 h. The total shrinkage consists of autogenous shrinkage from 24 h (Fig. 2) and drying shrinkage due to water loss to the outside environment. The total shrinkage trend is clear, namely, the effect of MK reduces the total shrinkage as the level of replacement increases.

The contribution of drying shrinkage to the total shrinkage can be seen in Fig. 5 which is given by Fig. 4 minus Fig. 2. The influence of MK can be seen to reduce the drying shrinkage of the OPC concrete by about 50%, the level of MK replacement having an insignificant effect.

The summary of the results for the 200-day autogenous and total shrinkage for all the concrete mixes is given in Table 4 where it is clear that the effect of MK is to reduce the total shrinkage considered either from initial set or from the age of 24 h. The reduction in the total shrinkage of the drying specimens can be partly attributed to the lower amount of evaporable water as hydration and pozzolanic reaction used up significant amount of the free water. The relatively lower percentage of water loss of the MK concretes (Table 4) seems to support this explanation. Table 4 also shows that for the MK concretes, autogenous shrinkage represents greater part of the total shrinkage and this observation does not seem to be influenced by the content of MK. Similar observation was found by Brooks et al. [17] on concrete containing up to 15% silica fume. However, at higher

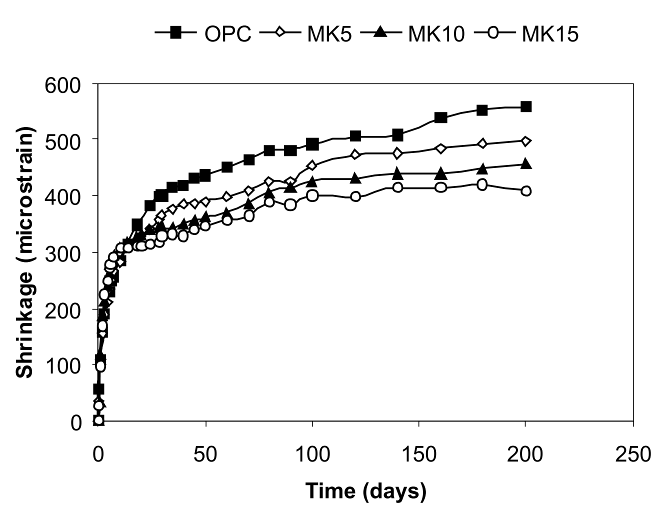

Fig. 4. Total shrinkage of drying concrete specimens from 24 h.

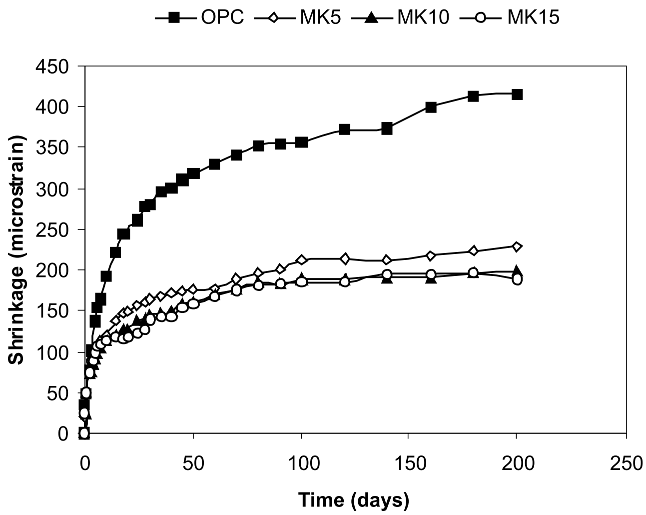

Fig. 5. Effect of metakaolin on drying shrinkage (Fig. 4 minus Fig. 2).

silica fume replacement levels, they observed that even greater part of the total shrinkage was contributed by autogenous shrinkage.

Overall, the foregoing trends lead to confirm that the MK concretes have a lower porosity and finer pore structure which encourages loss of water by self-desiccation rather than by diffusion to the outside environment.

4.3. Creep

The test results for total creep and basic creep at constant initial stress/strength ratio are given in Figs. 6 and 7, respectively, while the results for the companion load-free specimens are shown in Fig. 8. From Figs. 6 and 7, it is evident that the inclusion of MK is to reduce both the total creep and basic creep of concrete, with a greater reduction in creep at higher replacement levels, for example, for MK15 concrete the total and basic creep were reduced by 52% and 60%, respectively. The reduction in creep could be attributed to a denser pore structure, stronger paste matrix and improved paste aggregate interface of the MK concrete mixtures

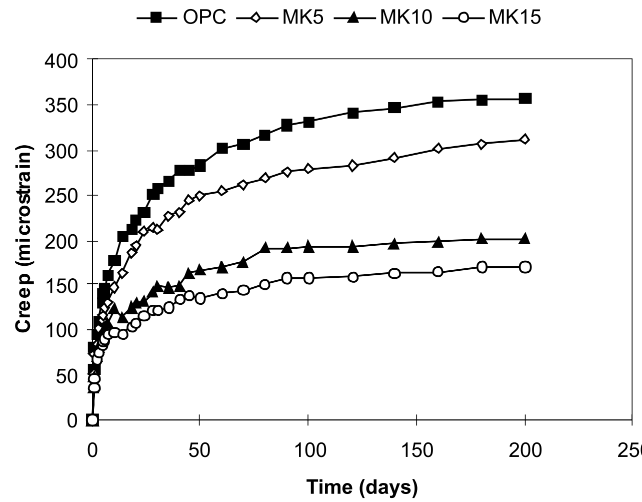

Fig. 6. Influence of metakaolin on the total creep of concrete loaded to an initial stress/strength ratio of 0.20 at the age of 28 days.

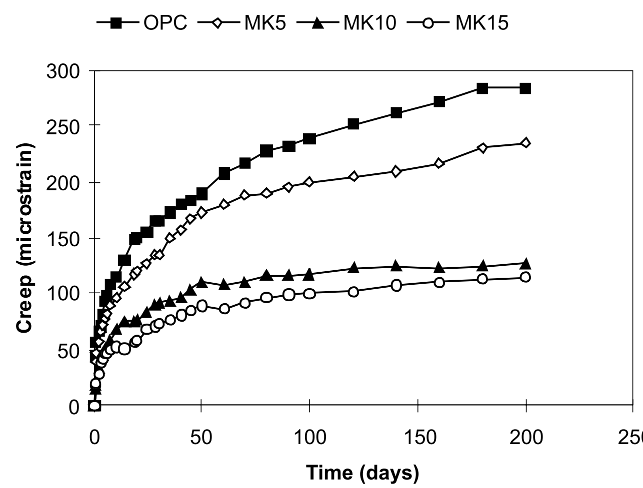

Fig. 7. Influence of metakaolin on the basic creep of concrete loaded to an initial stress/strength ratio of 0.20 at the age of 28 days.

as a result of the formation of additional hydrate phases from secondary pozzolanic reaction of MK and its filler effect. According to Wild et al. [15], the elementary factors influencing the contribution of MK to

Table 4
Summary of results for the 200-day total and autogenous shrinkage of concrete[a]

Item/concrete mixes	OPC	MK5	MK10	MK15
Total shrinkage from initial set (10^{-6})	861	713	618	516
Total shrinkage from 24 h (10^{-6})	558	499	455	410
Total autogenous shrinkage from initial set (10^{-6})	445	485	419	327
Autogenous shrinkage from 24 h (10^{-6})	142	271	256	221
Percentage of A.S.–T.S. from initial set (%)	52	68	68	63
Percentage of A.S.–T.S. from 24 h (%)	25	54	56	54
Weight loss of the exposed specimens (%)	0.85	0.67	0.73	0.76

[a] A.S. – autogenous shrinkage; T.S. – total shrinkage.

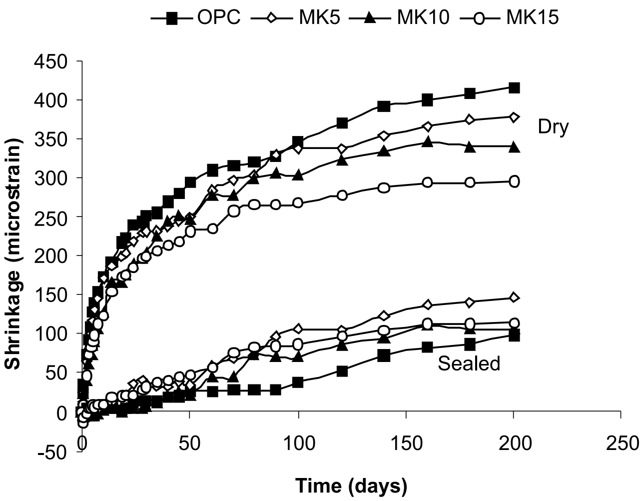

Fig. 8. Influence of metakaolin on the shrinkage of concrete under exposure to drying and under sealed conditions after 28 days of moist curing.

concrete strength are the acceleration in cement hydration, the filler effect and the pozzolanic reaction of MK with calcium hydroxide. The refined pore structure could influence the movement of water which is associated with initial creep [18]. The hydration and pozzolanic reaction of MK could have used up part of the water, as indicated by higher autogenous shrinkage, and reduce the amount of water available for movement out of the gel, thus reducing early creep. While the strength and the quality of the paste matrix as well as the transition zone could effect particle or solid movement which is associated with long-term creep [18]. In addition, as in the case of autogenous shrinkage, the formation of new hydrate phase (gehlenite hydrate) as postulated by Wild et al. [8] could also be possibly accounted for the significant reduction in creep at higher MK replacement levels.

The 200-day specific drying creep (Table 5) which is taken as the difference between the specific total creep and basic creep was also reduced for the MK concretes. For example, compared to the OPC concrete, the specific drying creep reduced by 42% for the MK15 concrete.

The effect of MK on the load-free specimens of concrete after moist curing for 28 days (Fig. 8) is similar in pattern with its effect on creep, i.e. there is a shrinkage which reduces with increasing replacement levels of MK. However, the extent of reduction in shrinkage is somewhat lower than that of the total creep and basic creep. For example, the 200-day shrinkage was reduced 28.7% for the MK15 concrete. The relatively lower amount of water loss for the MK concretes at the end of the test (Table 5) seems to support this observation.

Generally, the shrinkage measured on the load-free specimens would be termed drying shrinkage, but as can be seen from Fig. 8, there was also some autogenous shrinkage ($100–140 \times 10^{-6}$) still occurring even after 28 days of moist curing of the sealed concrete specimens. Therefore, the term total shrinkage from 28 days should be applied to describe the deformation of the load-free companion specimens for the creep tests.

It is of interest to compare the total shrinkage results of Fig. 8 with those of Fig. 4 in order to assess the effect of length of curing on subsequent shrinkage. It is clear that after 28 days of moist curing, the total shrinkage is less because of the reduced contribution of autogenous shrinkage. However, even if the true drying shrinkage is compared (Fig. 5 and the difference between the 'dry' and the 'sealed' specimens of Fig. 8), an increase in length of curing appears to decrease drying shrinkage. The influence of length of curing on shrinkage is a source of disagreement in the standard methods of prediction.

Finally, the measured 200-day creep and shrinkage values have been compared with those estimated by the CEB 90 prediction model [19], which caters for high strength concretes made from blended cements. Table 6 shows that the modulus of elasticity at the age of 28 days is estimated very accurately. Basic creep (sealed concrete) is predicted satisfactorily for OPC and MK5 concretes but overestimated for the higher levels of MK replacement. Total creep (drying concrete) is overestimated by the CEB 90 model code for all the concrete mixes. As could be expected, since the code does not allow for autogenous shrinkage, the measured total shrinkage is greater than the predicted drying shrinkage,

Table 5
Summary of the results of creep test after 200-days under load and weight loss of the drying shrinkage specimens

Concrete mix	Creep (10^{-6})		Stress (MPa)	Specific creep (10^{-6}/MPa)		Drying creep (10^{-6}/MPa)	Weight loss of companion load-free specimens (%)
	Dry	Sealed		Dry	Sealed		
OPC	358	285	13.9	25.8	20.5	5.3	0.63
MK5	312	235	16.8	18.5	14	4.5	0.60
MK10	201	126	17.7	11.3	7.2	4.1	0.59
MK15	171	115	17.9	9.5	6.4	3.1	0.53

Table 6
Comparison of measured and predicted elasticity, creep and shrinkage

Concrete mix	Modulus of elasticity (GPa)		Basic creep (10^{-6})		Total creep (10^{-6})		Total shrinkage (10^{-6})	Drying shrinkage (10^{-6})	
	Measured	Predicted	Measured	Predicted	Measured	Predicted	Measured	Measured	Predicted
OPC	41.3	41.9	285	263	358	774	415	317	271
MK5	42.5	42.6	235	187	312	552	377	231	243
MK10	43.4	44.5	128	177	201	521	340	233	163
MK15	42.9	44.4	115	196	171	577	296	181	166

but if the 'true' measured drying shrinkage is considered, Table 6 shows there is a reasonable agreement.

5. Conclusions

1. The effect of MK is to reduce the early age autogenous shrinkage of concrete measured from the time of initial set, the reduction being greater at higher replacement levels. On the other hand, the long-term autogenous shrinkage measured from 24 h increases significantly for the MK concretes with a reducing trend at higher replacement levels. The MK5 concrete exhibits a maximum in long-term autogenous shrinkage considered from the time of initial set, while the MK10 and MK15 concretes show lower autogenous shrinkage than the control concrete.
2. Compared with the control concrete, the greater part of the total shrinkage of the MK concretes is constituted by autogenous shrinkage, the smaller part being drying shrinkage. This observation does not appear to be influenced by replacement level.
3. Total creep, basic creep and drying creep of the concrete are greatly reduced as a result of MK inclusion particularly at higher replacement levels.
4. The CEB model code 90 estimates the modulus of elasticity accurately and basic creep for low MK levels. For higher levels of MK basic creep is overestimated, as is total creep. True drying shrinkage is estimated fairly accurately.

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