Autogenous Shrinkage, Pozzolanic Activity and Mechanical Properties of Metakaolin Blended Cementitious Materials

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·· Abstract

In the literature, there are some conflicting results regarding the influence of metakaolin (MK) addition on autogenous shrinkage behavior of cementitious materials. In this study, with the aim of identifying how the use of MK changes the properties of cementitious materials, cement was partially replaced by MK in different proportions (8%, 16%, and 24%) in pastes produced with variable water/binder ratios (w/b; 0.28, 0.35 and 0.42). The temperature development and calcium hydroxide consumption by thermogravimetric analysis were observed to better characterize the effects of MK on the autogenous shrinkage behavior. The mechanical properties such as compressive strength and flexural strength were experimentally investigated. The results show that the addition of MK has contrasting effects on autogenous shrinkage during early ages depending on the w/b ratio. Thermogravimetric analysis showed that the amount of stratlingite was smaller in pastes with low w/b ratios than in pastes with high w/b ratio. The calcium hydroxide consumption with the pozzolanic reaction of MK was significant even at 2 days, but consumption ratios with respect to the reference paste slowed down at 7 to 28 days.

Keywords: autogenous shrinkage, mechanical properties, metakaolin, pozzolanic activity, temperature development, thermogravimetric analysis

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1. Introduction

Industrial wastes and by-product pozzolans have variable properties, which in some cases may limit their usage. Metakaolin (MK), on the other hand, is produced in a specific way and can be produced in requested particle size, purity, and whiteness (Brooks et al., [2000](#page-6-1)). The highly reactive transition phases, named MK $(AI_2O_3.2SiO_2)$, are produced by burning the kaolinrich clay minerals $(AI_2Si_2O_5(OH)_4)$ in a specific temperature range $(500 - 600^{\circ}C)$ with altering the crystal structure. This process can be strictly controlled and, as a result, desirable properties such as purity and reactivity can be achieved. MK reacts with water and calcium hydroxide and forms calcium hydro-silicate and calcium hydro-alumina-silicate with calcium hydro-aluminate such as C_2ASH_8 -stratlingite, C_4AH_{13} and, in some rare cases, C_3ASH_6 -hydro-garnet (Murat, [1983;](#page-7-1) Khatib and Wild, [1996](#page-7-2)).

In some recent studies it has been suggested that the use of MK enhances the mechanical properties of concrete (Qian and Li, [2001](#page-7-0); Bindiganavile and Banthia, [2001](#page-6-0); Badogiannis et al., [2004](#page-6-2); Poon et al., [2006;](#page-7-3) Siddique and Klaus, [2009;](#page-7-4) Madandoust and Mousavi, [2012](#page-7-5); Dhinakaran et al., [2012;](#page-6-3) Akcay et al., [2016](#page-6-4)). Also the variations in fresh properties of concrete containing MK have been examined experimentally in some previous studies (Brooks and Megat Johari, [2001](#page-6-5); Siddique and Klaus, [2009;](#page-7-4) Akcay and Tasdemir, [2015](#page-6-6)), where fresh state behavior such as setting times of mixtures with MK was found to be different compared to that of plain Portland cement mixtures. According to Brooks et al. [\(2000](#page-6-1)), who conducted comparative tests on pastes with both MK and silica fume, the setting time is not affected by the amount of silica fume, whereas it is delayed with increasing MK content and shortened significantly at 15% of MK replacement. The authors interpreted the marked decrease especially at the initial setting time to be a result of MK addition that forms a denser binder phase, which needs more water and consequently accelerates the setting. However, they also stated that this explanation was not sufficient. Moulin et al. [\(2001](#page-7-6)) suggested that setting time of concretes with MK was shorter than the control samples with no MK at water/binder (w/b) ratio of 0.40. They also determined the yield stresses of pastes at 5th and 90th minutes with using rheometer and they found that MK addition resulted in significant increases in these values. They stated that higher water requirement of MK caused the thixotropic behavior of paste and they explained it with the acceleration of Portland cement hydration by using MK (Moulin et al., [2001\)](#page-7-6). Similarly, Li and Ding [\(2003\)](#page-7-7) stated that MK promotes Portland cement hydration and shortens the setting time. However, MK has no effect on aggregate segregation of mortar and concrete. It

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was proposed that the mixtures with MK had a higher slump and flow diameter in comparison with the mixture containing an identical amount of silica fume (Ding and Li, [2002;](#page-7-8) Akcay and Tasdemir, [2018\)](#page-6-7).

The use of pozzolan changes the microstructure and hydration properties of cementitious materials and hence results in an alteration in autogenous shrinkage behavior. The use of fly ash causes a significant reduction in water movement in the pores (e.g., Tangtermsirikul, [1999;](#page-7-9) Staquet and Espion, [2004](#page-7-10)). It was shown that with an increasing volume of fly ash the autogenous shrinkage is reduced in the paste with low w/b (Termkhajornkit et al., [2005\)](#page-7-11). On the other hand, it was demonstrated that with an increasing amount of slag the autogenous shrinkage of concrete is increased since the slag causes self-desiccation (Chan et al., [1999;](#page-6-8) Lura et al.[, 2001;](#page-7-12) Lee et al., [2006](#page-7-13)). The addition of silica fume also causes a considerable increase in autogenous shrinkage of concrete (Dela and Stang, [1997;](#page-6-9) Zhang *et al.*, [2003](#page-7-14)).

In the literature, there are some conflicting results in explaining the effects of MK addition on autogenous shrinkage. For MK blended paste the self-desiccation is expected to be more pronounced than diffusion to the external media because of the finer pore structure. In some studies the autogenous shrinkage was shown to be increased with using MK by up to 15% of replacement ratio (Wild et al., [1998](#page-7-15); Brooks and Megat Johari, [2001;](#page-7-15) Justice and Kurtis, [2007](#page-7-16)), after that there was a 65% decrease in 24 hours autogenous shrinkage (Brooks and Megat Johari, [2001](#page-6-5)). In some other studies, however, autogenous shrinkage was shown to have decreased with MK addition especially in pastes with high water to cement ratio (Gleize et al., [2007\)](#page-7-17). Autogenous shrinkage behavior of MK blended pastes has been examined by Wild et al. [\(1998\)](#page-7-15) who indicated that the use of MK by up to $10 - 15%$ replacement ratio increases autogenous shrinkage, while at higher replacement ratios it causes a sharp decrease. The maximum value of autogenous shrinkage is considered to be attained by optimizing the effects of cement hydration and the consumption of water from the pore structure due to the pozzolanic reaction.

In this work, a total of 12 paste samples were prepared. Three different reference cement pastes prepared using w/b of 0.28, 0.35 and 0.42 (by weight) were produced. Then MK blended pastes were cast by replacing 8%, 16% and 24% of cement by MK. In this study, the effect of the addition of MK on autogenous shrinkage, temperature development, thermogravimetric analysis, calcium hydroxide consumption, and mechanical strength were detailed.

2. Experimental Details

2.1 Mixtures

The mixtures were produced using CEM I 42.5R cement. The compressive strength of standard RILEM Cembureau of mortars was found as 48.1 and 60.5 MPa at 7 and 28 days, respectively. Some chemical and physical characteristics of cement used are given in [Table 1.](#page-1-0)

The MK powder (MetaMax, BASF) used in mixtures was a kaolinite mineral composed of a tetrahedral sheet of silica and an octahedral sheet of alumina with 7,2 Å inter-laminar space. Embedded water with a proportion of 1:2 between $SiO₂$ and Al_2O_3 lamina evaporated during calcinations to form MK. The compositional and physical characteristics of MK used are listed in [Table 2](#page-1-1). Consistent with ASTM C618 [\(2018](#page-6-10)), this MK is identified as the N-type mineral additive which is classified as raw or calcined natural pozzolans including some clay and shales that fulfill the application requirements for this class as suggested in the relevant standard.

In the mixtures, a polycarboxylic ether-based high-range water reducing admixture (HRWRA) was used to obtain the same workability (MasterGlenium 51, BASF). The mixture designation proportions and sample names are listed in [Table 3.](#page-2-0) The numbers subsequent to the letters P and MK in the description code indicate w/b ratios and replacement ratios of MK, respectively. Exceptions, however, are the control samples, which do not contain MK and are coded by only w/b ratios. The sample code for the paste starts with the letter P, while the letter M is used for the mortar sample. The mix proportions for paste samples can be found in Akcay and Tasdemir [\(2015](#page-6-6)). For determining the effects of MK addition on mechanical properties, the mortar phases of pastes [\(Table 3\)](#page-2-0) were produced using 20% volume of natural

Table 2. Some Chemical and Physical Characteristics of MK used in Mixtures

Oxide compositions $(\%)$										
SiO ₂	TiO ₂ MgO CaO Fe ₂ O ₃ Al_2O_3 Na ₂ O K,O									
53.0	48.8	0.43	0.02	0.03	1.70	0.19				
C_{model} C_{model} C_{model}										

Specific gravity: 2.50 g/cm³ Color: white

Particle size distribution: $d_{50} \sim 1.3 \mu m$, passed 2 μm sieve: 66% Specific surface area: $13 \text{ m}^2/\text{g}$

Specific gravity					3140 kg/m^3				Initial		183 minutes	
Blaine specific surface				Setting times $3490 \text{ cm}^2/\text{g}$ (Vicat Test)			Final			217 minutes		
Volume Stability					l mm							
C_3S			C_2S			C_3A		C_4AF				
	Compound composition $(\%)$		62.82			10.15			6.08		10.47	
						Chemical composition $(\%)$						
SiO ₂	Al_2O_3 CaO Fe ₂ O ₃		MgO		SO ₃		Insoluble residue	Loss on ignition		Free lime	Total alkalis	
20.07	4.49	3.44	64.66	1.23		2.76	0.58		2.61		1.21	0.53
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Table 1. Chemical and Physical Characteristics of Cement

Mixture	Cement,	Water,	MK,	HRWRA.	Natural sand.
code	kg	kg	kg	kg	kg
M42	1078	453	0	Ω	536
M42MK8	992	453	86	0.4	536
M42MK16	906	453	173	1.38	536
M42MK24	820	453	259	2.6	536
M35	1210	423	0	0.88	536
M35MK8	1114	423	97	1.22	536
M35MK16	1017	423	194	3.38	536
M35MK24	920	423	290	4.4	536
M28	1315	368	θ	2.96	536
M28MK8	1210	368	105	4.48	536
M28MK16	1105	368	210	7.32	536
M28MK24	1000	368	316	10	536

Table 3. Mixture Design of Mortar Samples (1 m^3)

sand aggregate with a particle size range of $0 - 4$ mm, a particle density of 2,600 kg/m³ and effective water absorptions of 2.1% as suggested in ASTM C127 [\(2015](#page-6-11)), ASTM C128 ([2015\)](#page-6-12).

2.2 Test Procedures

The autogenous shrinkage in the samples was determined volumetrically by assessing the volume variation in buoyancy during the first 48 hours from the initial setting. More information for the test methods is given in Akcay and Tasdemir [\(2008\)](#page-6-7). The change in temperature in semi-adiabatic condition was also observed at the same time. The linear autogenous shrinkage was measured by determining the displacements on the sealed samples with sizes of 40 mm \times 40 mm \times 240 mm. The measurement was performed for a gauge length of 200 mm, and the accuracy was 0.001 m/m (the details can be found in Akcay and Tasdemir, [2009\)](#page-6-13). The monitoring of calcium hydroxide content, which can be accurately measured by differential thermal analysis/ thermogravimetric analysis (DTA/TGA), is known to be a proper way for the evaluation of pozzolanic activity in cement pastes. The heating rate was 10°C/min, and experiments were carried out from 20°C up to 1,000°C. In order to prevent carbonation of the samples a 30 ml/min nitrogen flux was used. The method suggested by Marsh and Day [\(1998](#page-7-18)) was employed to calculate the calcium hydroxide content. Flexural strength of mortar [\(Table 3](#page-2-0)) was determined using beam specimens (40 mm \times 40 $mm \times 160$ mm) and the compressive strength was measured using the broken parts of the specimens.

3. Discussions on the Results

3.1 Temperature Development and Autogenous Shrinkage

in [Table 4,](#page-2-1) where the initial setting time appears to have reduced finity with using MK in the specimens with 0.42 w/b. In contrast, in the sh Vol. 23, No. 11 / November 2019 $-4729 -$ First, the data evaluation time for autogenous shrinkage measurements was determined. Since the mixtures contain different amounts of MK and HRWRA, the initial setting time was different for each series. To identify the initial and final setting times, Vicat needle test was used. The outcomes are listed with using MK in the specimens with 0.42 w/b. In contrast, in the

Table 4. Initial and Final Setting Times of Mixtures according to the Vicat Needle Test

samples with 0.28 w/b, the initial setting was delayed with the amount of MK. This is likely because of the fact that the higher amount of HRWRA (also given in [Table 4](#page-2-1)) in the low w/b ratio series has a retarding effect on the setting. The autogenous shrinkage measurements were started after the initial setting time for each series.

The variations in both autogenous shrinkage and temperature development of pastes with variable w/b are presented in [Fig. 1](#page-3-0).

The results show that the autogenous shrinkage of pastes increased remarkably with decreasing w/b ratio. Autogenous shrinkage is mainly a result of self-desiccation and water consumption in the paste. The absorption of water in pores by the hydrates and the development of fine pores is mainly governed by the chemical shrinkage. However, with ongoing hydration, self-desiccation becomes more effective (Akcay, [2018\)](#page-6-14). When w/b ratio was reduced, the total porosity and the mean pore size of pastes also decrease (Akcay et al.[, 2016](#page-6-4)). This leads to a decrease in internal relative humidity and makes the effect of self-desiccation more significant. The use of MK appears to have variable effects on autogenous shrinkage depending partly on w/ b ratios of pastes. In the specimens with w/b of 0.42, autogenous deformation reduced with the amount of MK, which can be explained by the filler effect of MK due to its denser internal structure that prevents water loss. With decreasing w/b ratio, however, recently developed hydration products interrupt the water and cause shrinkage in pores. This mechanism was operative in specimens with w/b of 0.35 and even more significant in pastes for which a higher amount of MK was used to obtain the denser internal structure. This explains the higher autogenous shrinkage values of the P35MK24 series than those of the P35MK16. In the specimens with w/b ratios of 0.28, the autogenous shrinkage diminished with increasing MK content at early ages because there was less amount of cement to undergo shrinkage. At later stages, however, the pozzolanic reaction of MK started with the formation of calcium hydroxide and this finer inner structure resulted in an increase in autogenous shrinkage. Such effects are also observed in linear autogenous

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Fig. 1. Volumetric Autogenous Shrinkage and Temperature Developments of Pastes: Figures (a), (b) and (c) Show the Variations in Volumetric Autogenous Shrinkage of Pastes, While (d), (e) and (f) Shows the Variations in Temperature Developments of Pastes with 0.42, 0.35 and 0.28w/b Ratios, Respectively

Fig. 2. Variations in Linear Autogenous Shrinkage of Pastes: (a) Pastes with 0.42 w/b Ratios, (b) Pastes with 0.35 w/b Ratios, (c) Pastes with 0.28 w/b Ratios

shrinkage measurements at later ages [\(Fig. 2\)](#page-3-1).

−calcium hydroxide, the high-temperature peak may be ascribed The temperature developments of pastes are also illustrated in [Fig. 1](#page-3-0). Each of the results represents the average of two specimens. In the pastes prepared using w/b ratio of 0.42, the peak temperatures for the series with 8 and 16% of MK are significantly higher than the reference series. Although the reaction of MK with calcium hydroxide is less likely in the first 48 hours due to the lack of to the promoting effect of MK on cement hydration in, especially

temperature became shorter with the increasing amount of MK,
which also shows that there was an increase in hydration rate.
4730 − KSCE Journal of Civil Engineering normal strength pastes. In all series, cement was replaced by MK and, as a result, the amount of cement in each paste was different. It was expected that an increase in MK content would decrease the temperature because of the lower amount of cement in the pastes. However, as is presented in [Fig. 1](#page-3-0) the change in temperature with the different amount of MK was insignificant. It was also documented that the time to reach the maximum which also shows that there was an increase in hydration rate.

In the reference mixtures, the major part of the volumetric autogenous shrinkage was completed in the first 24 hours and the curves reached an asymptotic stationary. In the samples with MK, however, there was a gradual increase in volumetric autogenous shrinkage with increasing MK content especially in samples with low w/b ratios $(Fig, 1)$. Since there was no asymptotic stationery for these series linear autogenous shrinkage measurements were conducted at later ages. The results of linear autogenous shrinkage for the paste samples at 90 days are illustrated in [Fig.](#page-3-1) 2. In the samples with w/b ratio of 0.42, the linear autogenous shrinkage was decreased with using MK in more abundance as their microstructures become denser. This effect was more obvious at later ages because of the initiation of pozzolanic activity of MK and the increasing amount of calcium hydroxide in time. This effect is observed in the series with w/b ratio of 0.35 with an exception of the paste with 24% of MK addition (P35MK24). In the pastes with 0.28 w/b ratios, the linear autogenous shrinkage increased compared to the reference series. In these series with low w/b ratio, linear autogenous shrinkage was increased with increasing MK content. These results indicate that autogenous shrinkage should be considered as an important parameter in high performance concrete containing MK.

3.2 Thermal Analysis and Calcium Hydroxide Consumption

DTA/TGA was applied for all produced pastes at 2, 7, 28 and 90 days. Two or more specimens were analyzed for each of the sample groups, and these results were subsequently used to calculate the calcium hydroxide contents. DTA curves of the

Fig. 3. DTA Results of Reference Samples with 0.42 w/b Ratios at Different Ages

Fig. 4. DTA Results of P35MK8 Samples at Different Ages

reference sample with 0.42 w/b ratios at different ages are presented in [Fig. 3.](#page-4-0) The values of calcium hydroxide abruption signals seem to have increased with time. Similar results are also seen in pastes with MK as, for instance, in the DTA curves of P35MK8 series, calcium hydroxide signals increased progressively and reached the highest values at 28 days [\(Fig. 4](#page-4-1)).

The effect of MK content on DTA signals can be seen in the specimens with 0.28 w/b at 28 days, which displays a reduction in DTA signals with the increasing amount of MK ([Fig. 5](#page-4-2)). The endothermic peaks near 170° C correspond to stratlingite (C_2ASH_8) which is the main product of the pozzolanic reaction of MK (Frias and Cabrera, [2001;](#page-7-19) Gleize et al., [2007](#page-7-17)). As is shown in [Fig. 5 n](#page-4-2)o C_2ASH_8 peaks are observed in the reference series. The amount of C_2ASH_8 in pastes with low w/b ratios is shown to be smaller than those with high w/b ratio, which may be attributed to the deficiency of water reducing ion mobility in a dense medium and hence resulting in a slower pozzolanic reaction.

The results from TG measurements were used to find out the calcium hydroxide contents of paste samples [\(Table 5\)](#page-5-0). In the reference pastes, there is a positive correlation between w/b ratio and the calcium hydroxide content. The use of MK reduced the calcium hydroxide content significantly, although this effect is less pronounced in specimens with greater w/b ratios particularly at early ages. Calcium hydroxide consumption ratios of each

Fig. 5. DTA Results of Samples with w/b Ratio of 0.28 and Different Amounts of MK: (a) Results for the Entire Temperature Range of 20 to 1000°C, (b) Focused on the First Part of the Diagram where the Samples with MK Display DTA Peaks Near 170°C (shown as a square)

		2 days	7 days			28 days	90 days	
P42	9.94		12.82		13.62		14.17	
P42MK8	9.34	(6.0)	12.23	(4.6)	11.02	(19.1)	10.36	(26.9)
P42MK16	9.15	(7.9)	9.18	(28.4)	8.88	(34.8)	8.66	(38.9)
P42MK24	7.90	(20.5)	8.54	(33.4)	7.06	(48.2)	6.01	(57.6)
P ₃₅	9.94		12.00		12.83		12.70	$\overline{}$
P35MK8	8.80	(9.4)	10.04	(14.6)	9.17	(26.8)	9.85	(20.8)
P35MK16	8.41	(11.0)	8.34	(27.0)	8.69	(28.9)	7.31	(39.1)
P35MK24	6.66	(26.5)	7.07	(35.8)	6.65	(43.1)	6.00	(52.8)
P ₂₈	9.35		10.29		9.86		10.78	
P28MK8	8.16	(10.3)	8.92	(11.1)	8.51	(11.4)	8.07	(23.0)
P28MK16	6.98	(20.5)	7.26	(25.1)	7.14	(23.0)	5.90	(41.1)
P28MK24	6.04	(28.1)	5.59	(39.1)	5.56	(36.8)	4.89	(48.4)

Table 5. The Variation of Calcium Hydroxide Content (g/100 g cement) obtained from TGA of Paste Specimens Containing Variable w/b Ratios with Time

Note: Values in parenthesis denote the calculated calcium hydroxide consumption ratios relative to the reference paste (in percent).

series with respect to their reference series are also presented in [Table 5.](#page-5-0) Almost in all series, calcium hydroxide consumption ratios by MK increased with both age and MK content. The results show that calcium hydroxide consumption with the pozzolanic reaction of MK was significant even at 2 days, but consumption ratios with respect to the reference paste slowed down at 7 to 28 days, which is probably due to the protective layers of reaction products formed around MK grains.

3.3 Mechanical Test Results

The compressive and flexural strength test results of mortar phases of cement pastes (mixture design was already given in [Table 3\)](#page-2-0) are summarized in [Table 6.](#page-5-1) The mechanical properties are shown to have been improved by using MK in all series. The mortars with MK, however, seem to have lower compressive strength than the reference samples at 2 days, and the difference between the two in compressive strengths becomes more noticeable with decreasing w/b ratio. Such low compressive strength is because of the fact that there is not enough amount of calcium hydroxide to react with metakaolin at these early ages. In the series M28MK24, for instance, a high amount of HRWRA

was used to obtain the fresh state consistency [\(Table 3](#page-2-0)), which increased the entrained air in the sample. It can be stated from [Table 6](#page-5-1) that at 24% of MK replacement there were slight decreases in both compressive and flexural strength. The most probable reason for this is that there was not enough calcium hydroxide to react with this amount of MK. Akcay et al. [\(2016](#page-6-4)) showed that the use of MK in more abundance resulted in smaller critical pore diameter, particularly at 28 days. The total porosity, which is taken as the volumetric ratio of intruded mercury to the total sample, on the other hand, increased with the MK content up to 24% of replacement ratio. The reason for this is that the amount of calcium hydroxide at early ages was not high enough for a reaction to take place with that amount of MK. In addition to the pozzolanic effect, the filler effect of MK was also operative in pastes with w/b ratio of 0.42, which led to a decrease in critical pore diameter significantly even in the first 2 days. In the pastes with w/b ratios of 0.28, in particular, the use of MK led to the larger pores to be eliminated almost completely. The higher total porosity of the pastes with the high amount of MK was found to be well correlated with their compressive strength results. For the mortars with 0.35 and 0.42 w/b ratios,

Table 6. Compressive Strength Test Results of Mortars (Each value is an average of 3 samples.)

			Compressive Strength (MPa)		Flexural Strength (MPa)				
	2 days	7 days	28 days	90 days	2 days	7 days	28 days	90 days	
M42	33.7	45.9	53.5	60.8	5.4	5.2	6.7	6.8	
M42MK8	33.9	49.1	59.9	65.7	7.1	6.7	7.4	7.5	
M42MK16	32.7	48.2	64.5	69.5	5.4	5.7	7.5	7.6	
M42MK24	28.5	48.7	59.4	65.1	6.6	6.7	6.8	6.9	
M35	48.8	56.6	70.6	73.6	5.9	6.6	7.0	7.2	
M35MK8	45.1	57.4	70.9	71.9	7.8	7.2	7.2	7.3	
M35MK16	42.2	63.0	73.6	77.2	6.7	7.3	8.9	8.4	
M35MK24	36.4	55.3	72.1	75.3	7.3	7.6	7.7	8.1	
M28	61.7	70.5	75.4	82.6	8.9	9.4	9.5	10.1	
M28MK8	59.2	71.4	80.6	81.5	8.9	9.5	9.8	10.2	
M28MK16	59.3	70.8	80.0	83.1	9.0	9.2	9.1	10.3	
M28MK24	49.8	68.2	80.7	81.3	7.3	7.6	7.5	8.6	
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there were decreases to some extent in flexural strength at 90 days, except for the M28MK24 samples, for which the flexural strength increased. This series of samples was already shown to have different temperature development in semi-adiabatic condition [\(Fig. 1\)](#page-3-0) because of the slower reactions at early ages, which also makes the strength development slower. If the amount of HRWRA was kept constant instead of choosing constant workability, the effect of MK content on mechanical strength might have been more obvious.

The main effecting factors of MK on strength are a pozzolanic reaction with calcium hydroxide, filler effect and accelerating cement hydration. It can be easily deducted from all experiments conducted that filler effect is moderate at first 24 hours while accelerating hydration effect is higher. The pozzolanic reaction is more effective after 2 days of age and there should be an optimum amount of MK considering the mechanical enhancement (Akcay et al., [2016;](#page-6-4) Akcay and Tasdemir, [2018](#page-6-14)).

4. Conclusions

The experimental test conducted led us to reach the following conclusions:

- 1. The use of MK has variable effects on volumetric autogenous deformation of pastes depending largely on w/b ratios. During the early ages, a significant decrease in autogenous shrinkage with increasing MK content was observed in specimens with high w/b ratios (0.42 and 0.35). For the samples with lower w/b ratios (0.28), however, autogenous shrinkage is governed primarily by the formation of calcium hydroxide and as a result of pozzolanic reactions with MK.
- 2. At later ages, the addition of MK was shown to have variable effects on linear autogenous shrinkage depending mainly on w/b ratio; it causes linear autogenous shrinkage to be reduced in 0.42 w/b ratios, while increased in lower w/b ratios.
- 3. Compressive strength test results showed that mortars with MK have lower strength than those with no MK at 2 days, and the difference between the two became more obvious with decreasing w/b ratio. For later ages, however, MK addition resulted in a significant increase in compressive strength for all the samples.
- 4. The most significant enhancement in flexural strength with the addition of MK was obtained in mortars with 0.42 w/b ratio. Thus, it can be said that especially in mixtures with lower cement content mechanical strength can be enhanced with MK addition.

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