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The effect of fly ash and limestone fillers on the viscosity and compressive strength of self-compacting repair mortars

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Abstract

Today, self-compacting mortars are preferred for repair purposes due to the application easiness and mechanical advantages. However, for self-compactability, the paste phase must meet some certain criteria at fresh state. The cement as well as the ingredients of the paste, powders with cementitious, pozzolanic or inert nature and plasticizing chemical admixtures should be carefully chosen in order to obtain a suitable paste composition to enrich the granular skeleton of the mix. The physical properties of powders (shape, surface morphology, fineness, particle size distribution, particle packing) and physico-chemical (time-dependent hydration reactions, zeta potentials) interactions between cement powder and plasticizer should be taken into consideration. All these parameters affect the performance of fresh paste in different manners. There is no universally accepted agreement on the effect of these factors due to the complexity of combined action; thus, it is hard to make a generalization.

This study deals with the selection of amount and type of powders from the viewpoint of fresh state rheology and mechanical performance. The influence of powder materials on self-compactability, viscosity and strength were compared with a properly designed set of test methods (the mini-slump, V-funnel tests, viscosity measurements and compressive strength tests). It may be advised that, for each cement–powder–plasticizer mixture, a series of test methods can be used to determine the optimum content and type of materials for a specified workability.

Keywords: Admixture; Cement paste; Rheology; Particle size distribution; Compressive strength

1. Introduction

Self-compacting repair mortars, as new technology products, are especially preferred for the rehabilitation and repair of reinforced concrete structures [1]. The water/powder (cement, fly ash, limestone filler, silica fume, etc.) ratio of mortar and the type of chemical admixtures should be determined, in order to place the fresh mortar without any external compaction and at the same time without causing any segregation. In other words, the paste phase rheology of repair mortar should possess suitable properties from the viewpoint of flowability and segregation [2–5]. The self-compactability of repair mortars may bring considerable advantages at narrow mould systems such as coating [6]. With the development of new-generation plasticizers, to obtain high filling rates is possible even for complex molding systems.

The fresh rheological characteristics, strength and durability of repair mortars can be enhanced by the addition of powders which can be collected in two groups as inert or pozzolanic [7]. The selection of amount and type of cementitious or inert powders depends on the physical and physico-chemical properties of these powders which are affecting the performance of fresh paste such as particle shape, surface texture, surface porosity and rate of superplasticizer adsorption, surface energy (zeta potential), finest fraction content, Blaine fineness and particle size distribution. There is no universally accepted agreement on the effect of these factors due to the complex influence of the combination of these factors [8].

In general, the increase in fine-grounded materials content in cements brings about the modification of rheological properties of pastes and consequently influences the workability of mortars and concrete mixtures. The observed changes can be advantageous or not. This is because of many factors influencing the rheology of cement pastes [9]. It is usually expected that, if the volume concentration of a solid is held constant, for a specific

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Table 1
The physical and chemical properties of cement and powders

Chemical analysis					Physical properties			
Basic oxide	OPC (%)	FA (%)	EM (%)	OK (%)	Powder type	Specific gravity	Blaine (m^2/kg)	Average zeta potential in de-ionized water (mV)
CaO	63.7	26.96	52.98	55.63	OPC	3.14	340	13.75
SiO_2	19.68	42.14	1.84	0.13	FA	2.20	290	−38.70
Al_2O_3	5.75	19.38	1.37	0.09	EM	2.58	443	5.22
Fe_2O_3	3.00	4.64	0.47	0.24	OK	2.65	538	7.12
MgO	0.90	1.78	0.42	0.44				
Na_2O	0.20	–	–	–				
K_2O	0.83	1.13	0.18	0.11				
SO_3	2.78	2.43	0.08	0.05				
Cl	0.001	0.001	–	–				
LOI	2.84	–	40.84	42.33				
Free CaO	1.55	4.34	–	–				
Insoluble residue	0.70	1.21	1.15	0.54				

workability, the replacement of cement with a fine powder will increase the water demand due to the increase in surface area. This is valid for silica fume [10]. However, in some cases, the above-mentioned conclusion is not appropriate. Lange et al. [11] concluded that for a specific workability, the inclusion of specified amount of fly ash reduced the water content and improved the workability. The workability enhancement is explained by the spherical shape of fly ash which causes the particle to easily roll over one another, reducing the interparticle friction [12]. The spherical shape also minimizes the particle's surface-to-volume ratio, resulting in low fluid demands.

Another factor influencing the rheology is the fineness of the powder used. Collins and Sanjayan [10] reported that, in concrete containing alkali-activated ground granulated slag as the binder, the workability was improved by replacing part of the binder with ultrafine materials. Yijin et al. [13] found that the addition of ultrafine fly ash (UFA) to cement paste, mortar and concrete can improve their fluidity, but some coarse fly ash cannot reduce water. Baoju et al. [14] reported similar findings. In their study, the addition of ultrafine fly ash with a Blaine specific surface area of 740 m^2/kg improved the fluidity and reduced the water demand for normal consistency. It should be noted that the method of detection of fineness is also important to characterize the effect of powders. According to Grzeszczyk and Lipowski [9], the grinding of the high-calcium fly ash brings about the rheological properties improvement (increase of fluidity) as compared with the paste containing coarse fly ash. However, the cement pastes containing fly ashes of similar grain size distribution in the range below 24 μm reveal similar rheological properties, although they show different specific surface area values. Grzeszczyk and Lipowski [9] have concluded that the finest fractions content is a better parameter to characterize the rheological properties of cement pastes than the fly ash Blaine specific surface.

Ferraris et al. [8] studied on the effect of addition of fine grounded materials with a comprehensive literature survey. They have concluded that the selection of a fine mineral admixture for improved concrete workability is not a trivial problem. At present, this selection cannot be predicted from the physical or chemical characteristics of the powders and can only be determined using the properly designed tests.

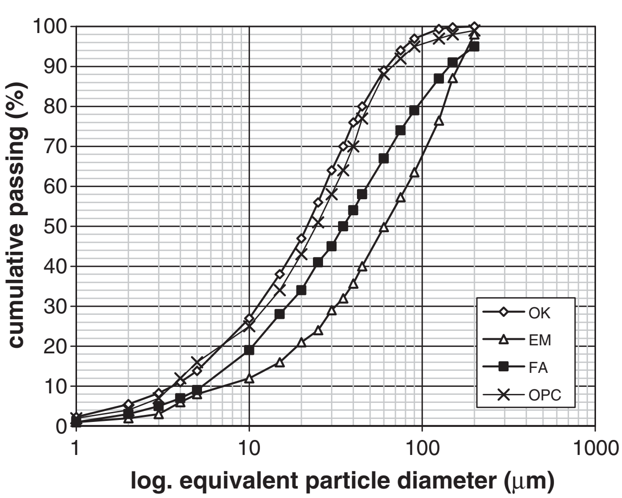

Fig. 1. Particle size distributions of cement and other powders in semi-logarithmic scale.

Table 2
Paste designs and self-compactability properties

Design label	Paste composition % (by mass)			Design parameters and self-compactability test results			
	Cement	Limestone filler	Fly ash	Admixture dosages (by weight % of PM)	W/PM[a]	Mini-slump diameter (mm)	V-funnel flow time (s)
K	100	0	0	1.2	0.25	410	8
EM20	80	20	0	1.2	0.25	298	13
EM40	60	40	0	1.2	0.25	258	12
EM60	40	60	0	1.2	0.25	335	11
OK20	80	20	0	1.2	0.25	439	13
OK40	60	40	0	1.2	0.25	449	10
OK60	40	60	0	1.2	0.25	481	8
FA20	80	0	20	1.2	0.25	305	–
FA40	60	0	40	1.2	0.25	275	–
FA60	40	0	60	1.2	0.25	293	–

[a] PM: powder materials (cement, fly ash, limestone powders).

For improving strength and durability properties; limestone fillers produce a more compact structure by pore-filling effect. In the case of fly ash, it also reacts with cement by binding Ca $(OH)_2$ with free silica by a pozzolanic reaction forming a non-soluble CSH structure [15,16].

In this study, the effect of fly ash and two types of limestone fillers on the fresh properties of paste phase of repair mortars was studied. The replacement ratios were by masses of 20%, 40% and 60% of cement, respectively. Additionally, compressive strength developments of mixes were determined.

2. Experimental studies

The experimental studies consist in two stages. In the first stage, the fresh paste flow diameter, V-cone flow time and viscosity measurements were conducted; in the second stage, compressive strengths of the specimens prepared from the paste mixtures were determined after 1, 7 and 28 days of standard curing.

2.1. Materials

An ordinary Portland cement (CEM I 42.5) conforming to the ASTM C150 standard, a C type fly ash from the Soma-B Thermal Power Plant and two types of limestone powders derived from different sources and production techniques were used. The physical and chemical properties of powders are given in Table 1. The particle size distributions of cement (OPC), fly ash (FA) and limestone fillers (EM-OK) were also compared in Fig. 1. They were determined by the help of a laser granulometer (Malvern Mastersizer). The 28-day pozzolanic activity index of fly ash was 93.8%. EM was a light pink and gray filler which was a filtration system by-product of crushed stone production. OK was a specially grounded and whitened limestone filler.

In all mixtures, a polycarboxylate-type new-generation High-Range Water Reducing Admixture (HRWRA) conforming to the ASTM C 494 [17] standard (Type F) was used. The solid material content, pH and specific gravity are 35.7%, 6.5 and 1.11, respectively.

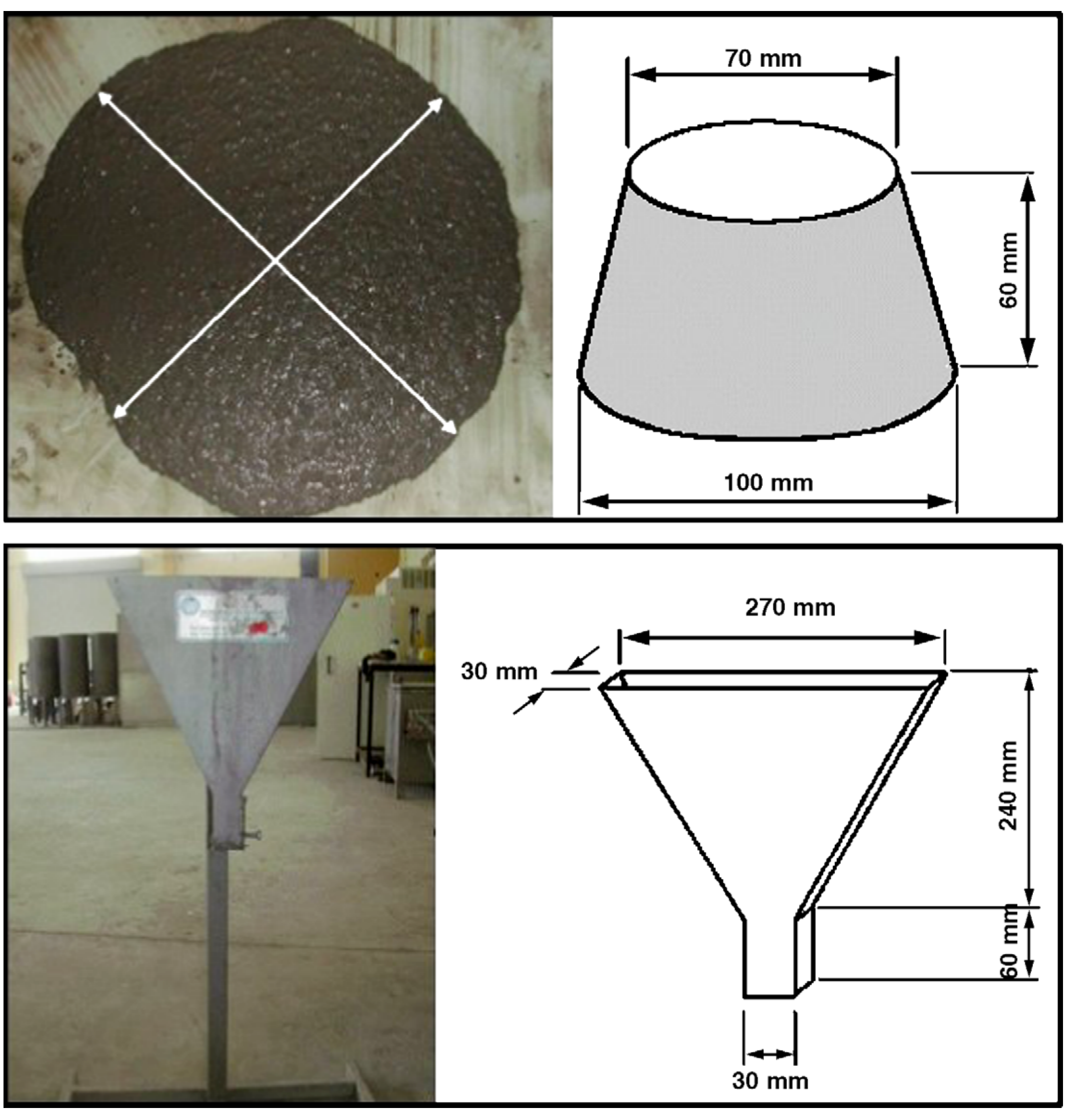

Fig. 2. Mini-slump and V-funnel (1 l capacity) tests for fresh paste.

2.2. Mixture proportions, preparation of cement pastes and fresh state tests

Cement paste mixture proportions are given in Table 2. Tests were performed at a constant temperature 21 °C and at a constant water-to-cement+powder ratio of 0.25. The cement paste was prepared by using the following procedure. First, powders were dry mixed for 1 min, then HRWRA with water was added and the mixing was continued for 3 min more. The compatibility of powders with cement and HRWRA was investigated by using the mini-slump and V-funnel tests (1 l capacity) in conformity with EFNARC [18] standards (Fig. 2). The averages of three test results are given in Table 2. The highest coefficients of variations were 15% and 7% for the mini-slump and V-funnel test results, respectively. Assuming that the cement paste rheology defined by Bingham Model, the mini-slump flow diameter values can be related with yield stress and V-funnel flow time can be related with plastic viscosity [19]. However, in recent work [8], the coefficients of correlations were usually weak and sometimes no correlation was observed, and it was concluded that more sophisticated tests need to be further developed.

Viscosity measurements were conducted with a Brookfield DV-E model viscosimeter (Fig. 3). It is a rotational viscosimeter with a smooth-walled concentric cylinder. At low stress values nearly yield stress, wall slip occurs resulting in inaccurately low yield stress measurements [20]. Slip was most pronounced at low strain rates and led to unusual low viscosity readings. As the deformation rate increased, the influence of slip decreased.

Fig. 3. Brookfield DV-E type viscosimeter.

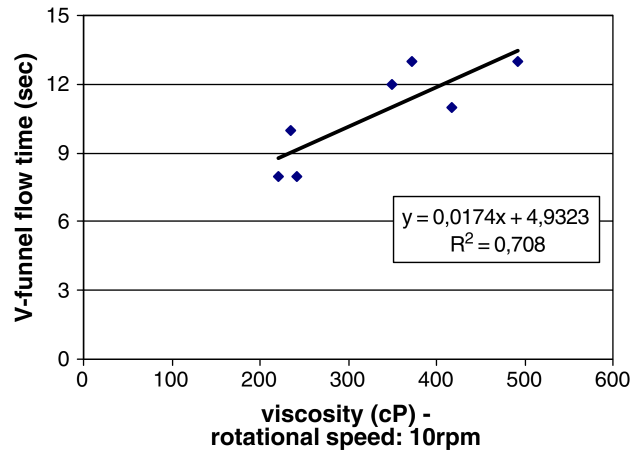

Fig. 4. Relationship between V-funnel flow time and viscosity values measured at rotational speed of 10 rpm.

For this reason, the viscosity measurements were conducted at different rotational speeds and time-dependent viscosity measurements were conducted. The flowable fresh paste was prepared and poured into the pot of the viscosimeter. Pre-mixing was performed by increasing the rotational speed from 0 to 60 rpm in 120 s. As soon as the highest rotational speed was reached, the viscosimeter stopped. After this initial preparation, a full cycle of increasing rotational speed by 14 steps from 0.3 to 20 rpm and back to rest with another 14 steps was performed. The average of viscosity values determined at upwards and downwards of each rotational speed steps were recorded. This procedure was repeated at 0, 20 and 40 min at different rotational speeds.

3. Results and discussion

Mixtures incorporating EM, OK type limestone powders showed higher V-funnel flow times when compared with control paste (Table 2). In other words, both of these two powders reduced the flowability when replacing with cement. However, the increase in replacement ratio reduced this effect; moreover, at 60% replacement of OK type limestone powder, the same flow time was measured with the control mix. The V-funnel flow times of mixtures with fly ash could not be determined due to the high cohesive nature of mixtures.

The relationships between the V-funnel flow time and viscosity measurements at different rotational speeds for limestone-incorporated mixtures are investigated and the best correlated results were derived from 10 rpm which was presented in Fig. 4. As a less sophisticated test, V-funnel flow time measurements correlate in certain cases with the viscosity. However, the coefficient of correlation of the relations seem not very strong and may only reflect the general tendency. In V-funnel test, the flow time of a constant volume of paste flowing from a definite opening is measured. In viscosimeter measurements, the resistances of fresh paste against the viscosimeter mill rotating at a set of constant rotational speeds are measured. The above-mentioned two methods may be alternatively used in optimization of paste viscosity for comparison purpose if the viscosity

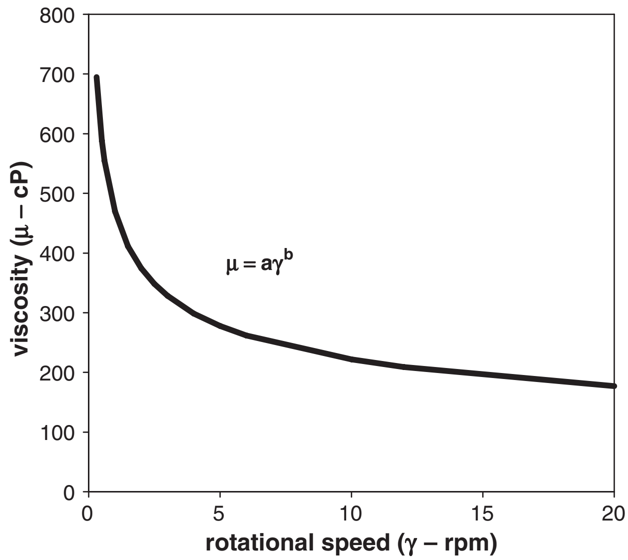

Fig. 5. The fitted equation curve of time-dependent viscosity change of control mixes at different rotational speeds.

is in the range of viscosimeter employed. However, at low viscosity changes, the V-funnel test can also be insufficient to measure the magnitude of change.

Time-dependent viscosity changes of mixes were measured at the beginning, 20 and 40 min after mixing. It has been realized that the behavior of all mixes at all measurement times have a similar tendency. The equation of $\mu=a\gamma^b$ was well fitted with the measurements, where μ is the viscosity in centipoise, and γ is the rotational speed in revolutions per minute. The constants a and b were calculated by the help of the best fit equations. An example of the curve obtained is illustrated in Fig. 5. The coefficient of correlation of equation curves and the values of constants are presented in Table 3.

Time-dependent viscosity changes of all mixtures at different rotational speeds are given in semi-log-scale graphs in Fig. 6. The behavior of all paste mixtures containing or not containing any powders can be defined as pseudoplastic. In other words, the viscous behavior is evident at low deformation rates, while at high deformation rates, flowable behavior is dominant. While mixing, the shear thinning effect at high rotation speeds breaks down the formation of high viscous behavior of mix at rest. At the stage of preparation, if paste is mixed with an efficient blender with a high shear rate, a more fluid mixture may be obtained; thus, the self-compactibilitiy and filling ability of mixture may also be improved. For this reason, to reach a more flowable consistency, high rotational speeds may be necessary. In practice, pumping may be an alternative way of increasing the shear rate applied on paste mixtures at the stage placement. High rotational speeds may also reduce the effect of wall slip problem, which tends to deviate the measured viscosity from its actual value.

If the change of viscosity with time is investigated, it can be observed that in all mixtures, the viscosity reducing effect of increase in rotational speeds weakens with time. The rate of decrease depends on the type and amount of mineral or inert admixture employed.

As can be seen from Fig. 6, if cement is replaced by EM type limestone filler, at all replacement ratios, the viscosity is increased when compared with control mix. It can be said that the energy needed to reach a flowable consistency should be higher for EM type rather than OK type limestone filler-incorporated mortars. The highest initial viscosity values were derived from 40% replacement ratios. However, all replacements showed a similar behavior independent of the powder type when measurements were conducted at the 40th minute.

When cement was replaced by OK type limestone filler at 20% ratio, the viscosity was increased when compared with the control mix (Fig. 6). However, by increasing the replacement ratio, viscosity was reduced again and at 60% replacement ratio the viscosity values were under the control mix values.

The replacement of cement with fly ash increased the viscosity when compared with control mix (Fig. 6). When the replacement ratio of fly ash is increased, even at high rotational speeds, no reduction in viscosity is observed; in other words, the increase in rotational speed could not reduce the viscosity of the paste. Tang et al. [21] mentioned about the effect of fly ash on viscosity increase and proposed that fly ash can increase the energy demand in order to reach sufficient workability.

In order to understand the possible mechanism of different mineral and inert admixtures on time-dependent viscosity, it is also important to characterize the micro-shape, surface texture, angularity and particle size distribution of powders. For this

Table 3
The equation constants and regression coefficients of the best fit curves

Equation constants $\mu=a\ \gamma^b$		K	EM20	EM40	EM60	OK20	OK40	OK60	UK20	UK40	UK60
a	t=0 min	934.1	2703.7	3346	2551.6	1476.6	842.4	469.3	1848.4	2112.1	1627.5
b		−0.59	−0.74	−0.98	−0.79	−0.60	−0.55	−0.33	−0.85	−0.76	−0.66
R^2		0.93	0.98	0.99	0.99	0.99	0.96	0.93	0.96	0.87	0.84
a	t=20 min	708.2	1236.7	1220.7	656.9	470.9	288.0	180.9	558.8	1618.8	783.8
b		−0.45	−0.70	−0.64	−0.48	−0.54	−0.30	−0.16	−0.41	−0.68	−0.15
R^2		0.90	0.99	0.96	0.88	0.89	0.76	0.66	0.83	0.90	0.71
a	t=40 min	632.8	669.9	594.2	527.9	383.2	181.0	115.7	370.5	683.1	552.0
b		−0.49	−0.49	−0.54	−0.48	−0.55	−0.22	−0.04	−0.34	−0.32	−0.08
R^2		0.85	0.97	0.91	0.89	0.92	0.71	0.08	0.81	0.97	0.42

μ: viscosity (cP); γ: rotational speed (rpm); a, b: equation constants.

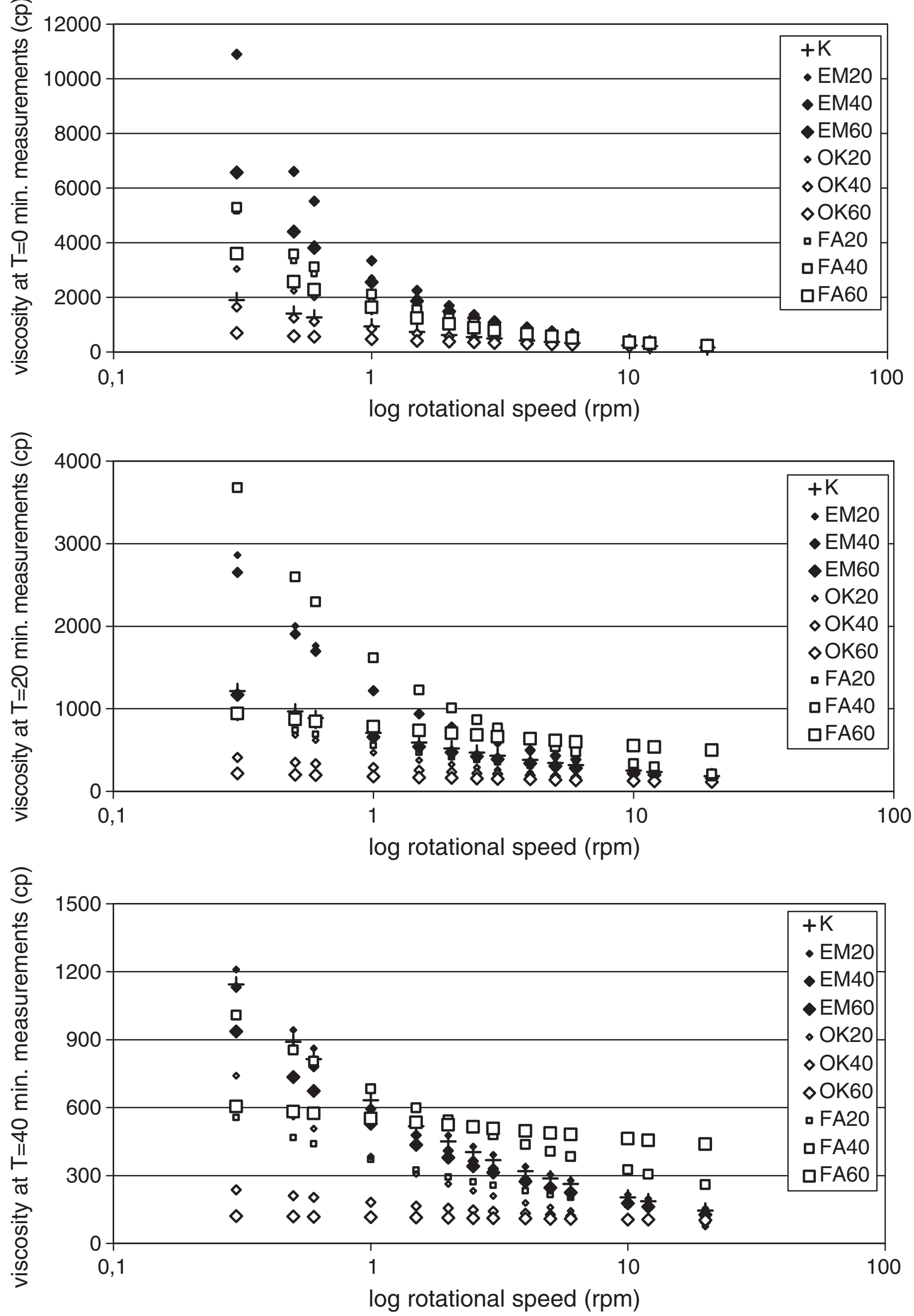

Fig. 6. The time-dependent viscosity change of plain cement paste and EM, OK filler and fly ash (FA) incorporated mixes at different rotational speeds.

purpose, SEM images of powders were investigated. As can be expected, micro-shapes of fly ash particles were mostly spherical or semi-spherical and angularity was rarely detected. The EM limestone fillers have a more angular and coarse structure when compared with OK limestone fillers. According to particle size distribution curves, equivalent particle diameters of fly ash particles were mostly in the order of 10–100 μm (Fig. 1). The equivalent diameters of EM limestone fillers were mostly in the order of 20–120 μm. The finer limestone filler was OK and the particle diameter mostly lies between 7–50 μm. It can be said that fly ash is coarser than OK limestone filler and cement (Fig. 1).

However, contrary to SEM investigations and particle size analysis, the Blaine surface area of fly ash (290 m^2/kg) was lower than the EM (443 m^2/kg) limestone filler. If one thinks that the fly ash is coarse, he might expect a lower water demand or lower admixture requirement (compared with limestone fillers) to obtain a defined viscosity due to this pseudo-coarse nature. However, test results indicate that at constant water content and HRWRA dosage, fly ash-incorporated mixes gave higher viscosity values. This behavior was more evident at later ages of viscosity measurements. Higher viscosity values of fly ash-incorporated mixes may be attributed to the water and/or HRWRA adsorption capacity of fly ash particles and change in

Table 4
The compressive strengths of mixtures at different ages

Mix code	Compressive strength (MPa)			
	1 day	7 days	28 days	90 days
K	25.2	71.5	95.9	100.5
EM20	23.3	69.5	89.3	93.4
EM40	16.5	59.3	71.5	77.4
OK20	27.5	77.5	92.5	99.5
OK40	20.3	72.5	79.3	84
FA20	11.2	70.5	93.2	107.3
FA40	4.6	56.3	75.8	90.4

zeta potential in the presence of HRWRA. Conversely, the finer limestone filler (OK) reduced or did not significantly change the viscosity when compared with fly ash. It should also be noted that coarser limestone filler (EM) was less effective in reducing the viscosity. The difference between the performances of limestone fillers may be explained by the production methods of these fillers. EM filler is a filtration system by-product of crushed stone production. However, the OK filler is a special production of whitened limestone filler having lower adsorptivity. Increasing the surface area may enhance the adsorption of HRWRA and, thus, the dispersing ability of system.

Another factor affecting the fluidity of mixes is the zeta potentials of powders. Cement and limestone fillers have positive zeta potentials while fly ash has negative zeta potentials. When the cement was replaced with fly ash, the particles with high negative zeta potential increased. It should be noted that the absolute value of zeta potential of fly ash particles (−38.7 mV) were higher than other powders (5.22 and 7.12 mV). In the presence of HRWRA, the enhancement in the rheological characteristics may be attributed to the difference in zeta potential values between fly ash and limestone powders.

From the above-mentioned studies, it can be concluded that particle size distribution, micro-shape, surface structure and zeta potential changes are better parameters for rheological characterization of cement–powder–superplasticizer dispersion systems rather than Blaine fineness.

The compressive strengths of pastes at 20% and 40% replacement of all powders at different ages are presented in Table 4. When the 28-day compressive strengths were compared, the nearest values to the 100% cement containing control paste (95.9 MPa) was derived from 20% fly ash replacement (93.2 MPa). Beyond 28 days, if compressive strengths were compared, 20% fly ash-replaced mixes gave higher values than the control mixes due to the pozzolanic effect. At EM and fly ash-replaced mixes, a reduction in 1, 7 and 28 days compressive strengths were observed as the powder replacement increases. However, at OK replaced mixes at 20% replacement ratio, the compressive strength values were increased at 1 and 28 days. The increase in compressive strength can be explained with pore-filling effect of fine-grounded limestone powder and also provided suitable nucleus for hydration and by this way catalyzing the hydration [22]. Additionally, limestone fillers reacted with C_3A phase of cement and supplied the formation of monocarboaluminate that partially takes part of ettringite; thus, increase at early strength values can be provided [23]. However, it seems that no additional hydration reactions take place to enhance the long-term strength of pastes incorporating limestone fillers. At 1-day strengths, the mixes containing limestone fillers showed higher compressive strength values when compared with fly ash. This situation was caused by the slow pozzolanic reaction between the cement and fly ash that was well suited with the literature [24]. It should also be noted that the use of polycarboxylate-based HRWRAs at high dosages may also lengthen the setting times and negatively affect the early compressive strength of mixes incorporating fly ash [25].

4. Conclusions

Based on the test results presented in this paper, the following conclusions can be drawn:

1. The behavior of all paste mixtures with or without any mineral admixtures or inert fillers can be classified as pseudoplastic. In other words, the viscous behavior is evident for low rotational speeds of mixing, while at higher speeds, flowable behavior becomes dominant. While mixing, the shear thinning effect at high rotation speeds breaks down the formation of the high viscous behavior of the mix at rest. At the stage of preparation, in case of using an efficient blender with a high rotational speed capacity, a more fluid mixture may be obtained. Thus, self-compactability and filling ability of the mixture can be improved. For this reason, to achieve a more flowable consistency, high rotational speeds may be necessary. In practice, pumping may be an alternative method of increasing the shear rate applied on paste mixtures at the stage placement.
2. Except the finest limestone filler (OK), all powders increased the initial viscosity when compared with plain cement paste. However, the rate of increase depended on the replacement ratio of each mineral or inert admixture. The amount of change depends on the particle shape, surface texture, size distribution and zeta potentials of powders used.
3. It may be suggested that for each cement–mineral admixture or filler–plasticizer mixture, a series of test methods (the mini-slump, V-funnel tests and/or viscosity measurements and compressive strength tests) should be employed to determine the optimum content and type of materials for a specified workability.
4. The results derived from compressive strength tests showed that both limestone fillers were more effective than fly ash in terms of early strength gain. However, beyond 28 days, mixes incorporating fly ash gave higher strength values than the control mixtures due to the pozzolanic effect of fly ash.

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