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Effect of admixtures on the setting times of high-strength concrete

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Abstract

The effect of silica fume (SF), metakaolin (MK), fly ash (FA) and ground granulated blast-furnace slag (GGBS) on the setting times of high-strength concrete has been investigated using the penetration resistance method (ASTM C 403). In addition, the effect of a shrinkage-reducing admixture (SRA) on the setting times of normal and high-strength concrete was also studied. The setting times of the high-strength concrete were generally retarded when the mineral admixtures replaced part of the cement. While the SRA was found to have negligible effect on the setting times of normal strength concrete, it exhibited a rather significant retarding effect when used in combination with superplasticiser in high-strength concrete. The inclusion of GGBS at replacement levels of 40% and greater resulted in significant retardation in setting times. In general, as replacement levels of the mineral admixtures were increased, there was greater retardation in setting times. However, for the concrete containing MK, this was only observed up to a replacement level of 10%.

Keywords: Setting times; Penetration resistance; High-strength concrete; Mineral admixtures; Shrinkage-reducing admixture; Superplasticiser

1. Introduction

The utilization of high-strength and high-performance concrete has been increasing throughout the world. Due to the better engineering and performance properties, mineral admixtures such as silica fume (SF), fly ash (FA) and ground granulated blast-furnace slag (GGBS) are normally included in the production of high-strength and high-performance concrete [1]. Metakaolin (MK) is the most recent mineral admixture to be commercially introduced to the concrete construction industry. It has been claimed that concrete containing MK exhibits premium level engineering properties that are comparable to SF concrete [2]. Since the different mineral admixtures possess different chemical and mineralogical compositions as well as different particle characteristics, they could have different effects on the properties of concrete inclusive of the setting characteristics of concrete.

Knowledge of the setting characteristics of concrete is rather important in the field of concrete construction. These will help in scheduling the various stages involved in concrete construction operations such as transporting, placing, compacting and finishing of concrete. This information is a necessity when deciding whether or not to use a retarding admixture or accelerator. Recently, the Japan Concrete Institute (JCI) [3] proposed that the measurement of autogenous shrinkage, a particular feature of high-strength and high-performance concrete, should commence at the time of initial set. The setting of concrete can be defined as the onset of solidification in a fresh concrete mixture. Initial set approximates the time limit for handling the concrete, while final set indicates the onset of development of mechanical strength [4].

Previous research indicates that the setting times of concrete are influenced by water/binder ratio, initial and curing temperature, dosage, source and type of admixtures, as well as composition of the cement [5–19]. There is a general agreement that FA and GGBS retard the setting times of concrete with greater retardation at higher replacement levels [6–11]. However, at replacement levels of 70% and higher, the inclusion of FA was observed to cause rapid setting [7]. At temperatures greater than 20°C, the setting times of concrete containing GGBS were found to be less than those of ordinary Portland cement (OPC) concrete [6].

In the case of SF concrete, there seems to be some contradiction on its effect on setting times. Several investigators [12–14] reported that SF has a retarding effect on the setting times and the retardation increases

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with replacement level. On the contrary, the results of other researchers [15,16] indicated that it has a negligible effect. The retardation is sometimes associated with the effect of superplasticiser used in SF concrete [17]. The results of De Almeida and Goncalves [16] showed that for superplasticised concrete with a low water/binder ratio of 0.24, the effect of SF was to reduce the setting times. To the knowledge of the authors, there seems to be no information available on the effect of MK on the setting times of concrete. The objective of the present investigation was to determine the effect of mineral admixtures, namely, SF, MK, FA and GGBS, on the setting times of high-strength concrete. In addition, the influence of a shrinkage-reducing admixture (SRA) on the setting times of normal and high-strength concrete was also studied.

2. Materials and mix proportions

The cementitious materials used were OPC, SF, MK, FA and GGBS. The chemical compositions and the physical properties of the cementitious materials are given in Table 1. Details of the mix proportions for the concrete containing mineral admixtures are given in Table 2. Natural river sand and quartzite natural gravel with a nominal maximum size of 10 mm were used as the aggregates. The control mix was cast using OPC, while the other mixes were prepared by replacing part of the cement with the different mineral admixtures at three different replacement levels on mass-for-mass basis. An optimum water/cement ratio of 0.28 was obtained for the control high-strength concrete mix using the Cabrera Vibrating Slump test, a method developed by Cabrera and Lee [20]. The same water/binder ratio of 0.28 was used for the other concrete mixes with same amount of superplasticiser so that the workability changed due to the effect of the different mineral admixtures. The superplasticiser used is based on sulphonated vinyl copolymer.

The mix proportions of the concrete containing the SRA are given in Table 3. The control mixes, 325/1 (normal strength) and 450/1 (high-strength), were cast without using the SRA. Two percent of SRA by weight of cement was added to mixes 325/2 and 450/2. In the case of mixes 325/3 and 450/3, two percent of SRA was added while reducing the water at the same volume as the added SRA.

3. Test procedure

The setting times of the concrete mixtures were determined in accordance to ASTM C 403 [21]. The test was performed on mortar, which was obtained by sieving freshly mixed concrete through a 5-mm sieve, and measuring the force required for a needle to penetrate 25 mm into the mortar. To minimize the effect of ambient

Table 1
Chemical composition and physical properties of cementitious materials[a]

Item	Cementitious materials (%)				
	OPC	SF	MK	FA	GGBS
SiO_2	20.69	94.02	51.6	47.8	35.84
Al_2O_3	4.72	0.43	41.3	24.9	14.00
Fe_2O_3	3.06	1.65	4.64	8.7	0.53
CaO	63.76	0.13	0.09	1.8	39.72
MgO	2.08	0.53	0.16	1.2	8.57
Mn_2O_3	–	–	–	–	0.48
TiO_2	–	0.01	0.83	1.0	0.56
SO_3	2.92	–	–	0.9	0.15
K_2O	0.61	0.65	0.62	3.6	–
Na_2O	0.26	0.20	0.01	1.2	–
LOI	0.87	1.56	–	5.2	0.43
Compounds					
C_3S	53.9	–	–	–	–
C_2S	18.2	–	–	–	–
C_3A	7.2	–	–	–	–
C_4AF	9.2	–	–	–	–
Fineness					
SSA (m^2/kg)	380	20,000[b]	15,000	300–400[b]	426
>45 μm	–	–	–	8.9	–

[a] *Notations*: OPC: ordinary Portland cement; SF: silica fume; MK: metakaolin; FA: fly ash; GGBS: ground granulated blast-furnace slag.
[b] Typical values [17].

Table 2
Mix proportions for concrete containing mineral admixtures

Concrete mixes	Cement (kg/m³)	Admixture (kg/m³)	Sand (kg/m³)	Gravel (kg/m³)	Water (kg/m³)	SP (kg/m³)	Slump (mm)
OPC	450	–	675	1125	126	14	100
SF5	427.5	22.5	675	1125	126	14	200
SF10	405	45	675	1125	126	14	150
SF15	382.5	67.5	675	1125	126	14	90
MK5	427.5	22.5	675	1125	126	14	30
MK10	405	45	675	1125	126	14	20
MK15	382.5	67.5	675	1125	126	14	5
FA10	405	45	675	1125	126	14	180
FA20	360	90	675	1125	126	14	220
FA30	315	135	675	1125	126	14	250
GGBS20	360	90	675	1125	126	14	110
GGBS40	270	180	675	1125	126	14	170
GGBS60	180	270	675	1125	126	14	200

Table 3
Mix proportions for concrete containing the SRA

Concrete mix	OPC (kg/m³)	Sand (kg/m³)	Gravel (kg/m³)	Water (kg/m³)	SP (kg/m³)	SRA (kg/m³)	Slump (mm)
OPC325/1	325	757	1138	179	–	–	50
OPC325/2	325	757	1138	179	–	6.5	160[a]
OPC325/3	325	757	1138	172	–	6.5	60
OPC450/1	450	675	1125	126	14	–	70
OPC450/2	450	675	1125	126	14	9	90
OPC450/3	450	675	1125	116	14	9	80

[a] Shear slump.

temperature variation, the specimens were kept in a controlled environment of 20 ± 2°C and 65 ± 5% relative humidity throughout the test duration. Initial and final setting times are defined as the times at which the penetration resistance reaches values of 3.5 (500 psi) and 27.6 MPa (4000 psi), respectively.

4. Results and discussion

Figs. 1–3 show typical trends of penetration resistance of concrete as a function of time. The penetration resistance (P) of all the different concrete mixtures can be expressed as an exponential function of time (t) as follows:

$$P = ae^{bt}, \quad (1)$$

where a and b are coefficients.

In general, the correlation coefficients, R^2, for all the concrete mixes are greater than 0.9, and the acceptable accuracy of Eq. (1) supports the earlier findings of Polivka and Klein [22]. Subsequently, the setting times were estimated by Eq. (1) and checked by interpolation of experimental points, such as those shown in Figs. 1–3.

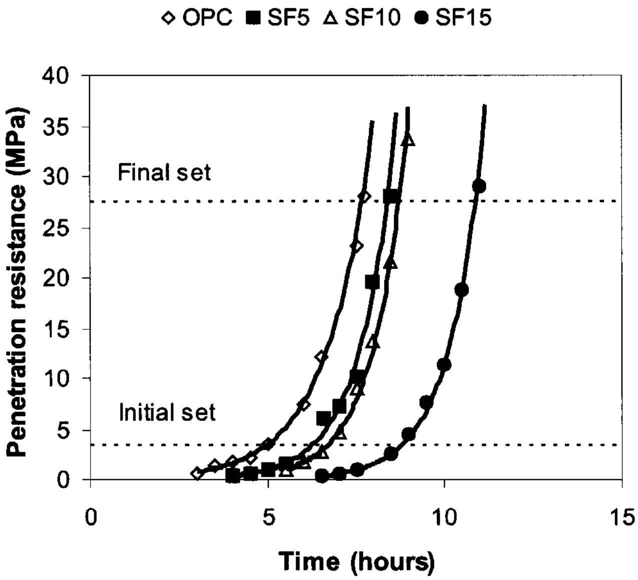

Fig. 1. Influence of silica fume on the penetration resistance of concrete.

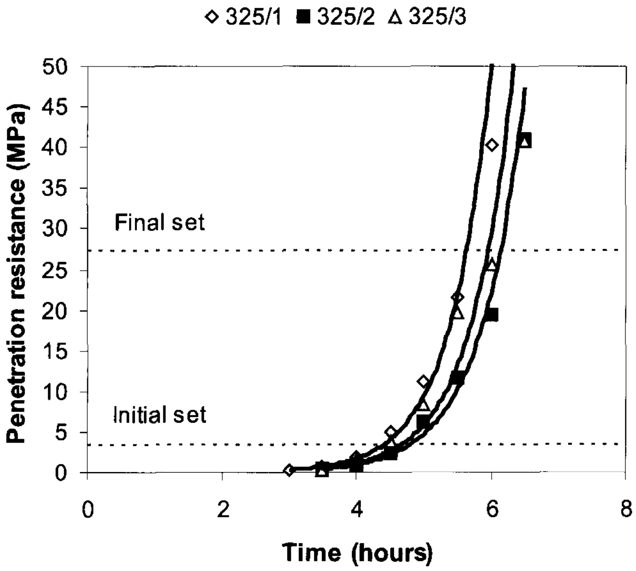

Fig. 2. Influence of the shrinkage-reducing admixture on the penetration resistance of normal strength concrete.

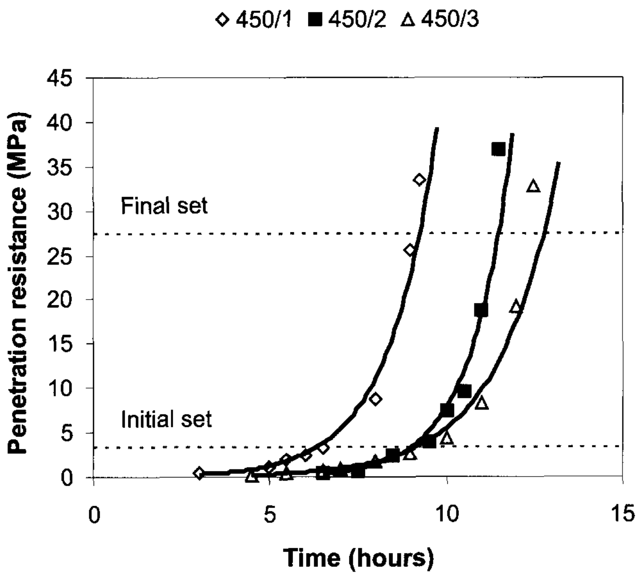

Fig. 3. Influence of the shrinkage-reducing admixture on the penetration resistance of high-strength concrete.

4.1. General effect of mineral admixtures

The setting times of the concrete mixes containing mineral admixtures are given in Table 4. From the table and Fig. 1, it is clear that the general effect of the different mineral admixtures is to increase the setting times of concrete. The observed retardation in setting times can be mainly attributed to the combined effect of a lower cement content and a higher "effective superplasticiser dosage" relative to the weight of cement for these concrete mixes, since part of the cement was replaced by the mineral admixtures. Previously [19] it has been reported that the general effect of superplasticisers is to retard the setting times of concrete and the extent of retardation depends upon the type and dosage of superplasticiser, type of cement and temperature. The hydration of cement and cement compounds, particularly tricalcium silicate (C_3S) which is responsible for the early strength of concrete, and tricalcium aluminate (C_3A) are normally retarded by a superplasticiser [19,23]. The retardation of hydration is due to adsorption of superplasticiser over the surface of cement particles [23]. Therefore, for the concrete mixes containing mineral admixtures, due to the lower amount of cement and higher effective superplasticiser dosage, a greater retarding effect could be expected. In addition, the dispersion effects provided by the superplasticiser and the mineral admixtures on the cement particles could have contributed to the retardation in the setting times. This is because the setting of cement paste has been postulated to result from two fundamental steps: coagulation establishing contacts between particles and the formation of hydrates in the contact zones making rigid the coagulation structure [24]. For the OPC concrete, which has higher cement content and lower effective superplasticiser dosage in comparison to the other mixes, the cement particles are expected to be more closely packed. This could result in greater interparticle contact, and thus could speed up setting. Furthermore, the mineral admixtures could also contribute to the change in setting times, depending on the onset and rate of the pozzolanic reaction.

4.2. Effect of the shrinkage-reducing admixture

The influence of the SRA on the setting times of normal and high-strength concrete is shown in Table 5 and Figs. 2 and 3. From these results, it can be seen that the effect of adding 2% SRA to mix OPC325/2, and adding 2% SRA to mix OPC325/3 while reducing equal volume of water, is to increase setting times by 9% and 6%, respectively. Mix OPC325/2 showed a slightly higher increase compared to mix OPC325/3. This can be attributed to the higher (water + SRA)/cement ratio. Kruml [5] showed that with the same cement content and aggregate grading, the setting times of concrete are governed by the water/cement ratio; the lower the water/cement ratio the shorter the setting times. In addition, the effect of the SRA in reducing the surface tension of water could reduce the interparticles force of attraction of the flocculated binder phase and retard the setting times. The latter reasoning could also be used to explain the smaller increase of 6% in setting times exhibited by mix OPC325/3.

For the high-strength concrete mixes, where the SRA was used in combination with superplasticiser, the

Table 4
Setting times of concrete containing mineral admixtures

Concrete mix	Initial set (IS) (h)	Relative IS	Final set (FS) (h)	Relative FS	FS – IS (h)
OPC	5	1	7.7	1	2.7
SF5	6.27	1.25	8.38	1.09	2.11
SF10	6.7	1.34	8.72	1.13	2.02
SF15	8.82	1.76	10.9	1.42	2.08
MK5	6.42	1.28	8.82	1.14	2.4
MK10	6.98	1.40	9.42	1.22	2.44
MK15	6.45	1.29	9.31	1.21	2.86
FA10	5.97	1.19	8.93	1.16	2.96
FA20	6.1	1.22	9.37	1.22	3.27
FA30	7.83	1.57	11.6	1.51	3.77
GGBS20	7.88	1.58	12.9	1.68	5.02
GGBS40	11.53	2.31	17.8	2.31	6.27
GGBS60	12.43	2.49	21.55	2.80	9.12

increase in setting time was more significant and consistent, even when the water was reduced. This could be due to the interaction between the SRA and superplasticiser. When a comparison is made between the setting times of mix OPC325/1 and mix OPC450/1, there seems to be a significant difference. Although the OPC325/1 mix has a much higher water/cement ratio than the OPC450/1, its setting times are much lower. Thus, the effect of superplasticiser could have offset the influence of water/cement ratio and retarded the setting times. In general the results obtained in this study on the effect of the SRA are in agreement with those reported by the manufacturer [25]. The use of SRA at a dosage of 1.5% and with water reduction of 8% as compared to the control was found to increase the initial and final sets by 16% and 5%, respectively. However, when 1.25% SRA was used with 0.25% superplasticiser and with higher water reduction of 14%, the initial and final sets were retarded by 25% and 24%, respectively [25].

4.3. *Effect of mineral admixtures replacement level*

In general, the effect of increasing the replacement level of mineral admixtures is to increase further the setting times of concrete. These will be in part due to the effect of a lower Portland cement content and the higher effective superplasticiser dosage suggested earlier. For SF concrete, when the replacement level was increased to 15% the initial and final set increased by factors of 1.8 and 1.4, respectively. This seems to be in agreement with previous findings of other investigators. Khedr and Abou-Zeid [12] reported that the use of SF as partial replacement of OPC increases the initial setting time of concrete prepared with the same water/binder ratio and containing the same dosage of superplasticiser; a greater retardation was noticed at higher replacement level. The results of investigations by Alshamsi et al. [13,14] also showed that the use of SF as partial OPC replacement extends the setting times of paste with greater retardation at a higher replacement level. They attributed this phenomenon to the decrease in the cement content, which is responsible for early stiffening of the paste. The results of an investigation by Bilodeau [18], as reported by Malhotra and Mehta [17], showed that the addition of 5% and 10% SF to non-superplasticised concrete resulted in marginal retardation in setting times. For concrete with a water/binder ratio of 0.4 containing 15% SF, a significant delay was observed. In this case, the retardation was attributed to the high dosage of superplasticiser used in the concrete. Pistilli et al. [15] and De Almeida and Goncalves [16] reported no significant difference in the setting times of concrete when SF was introduced to non-superplasticised concrete. On the other hand, for a lower water/binder ratio concrete containing a superplasticiser, the effect of SF was to reduce the setting times when compared to those of the control concrete containing the same amount of superplasticiser [16]. There could be some interaction between the SF and the superplasticiser used, thus reducing the setting times.

In the case of concrete containing up to 10% replacement of MK, there is a clear trend that both initial and final setting times are extended as the replacement level is increased. However, when the replacement level is further increased to 15%, there is a marginal reduction in setting times particularly the initial set when compared with those of the MK10 concrete. This could be due to the greater water demand at the higher MK content which could have produced a binder phase that is more dense and could speed up setting. In this case, the effect of higher water demand would have to offset the effect of lower cement content and higher effective superplasticiser dosage described earlier. However, a similar effect was not apparent with the corresponding replacement levels of cement by SF, which has a greater water demand due to its greater fineness. Therefore, the

Table 5
Setting times of concrete containing the SRA

Concrete mix	Initial set (IS) (h)	Relative IS	Final set (FS) (h)	Relative FS	FS – IS (h)
OPC325/1	4.40	1	5.64	1	1.24
OPC325/2	4.80	1.09	6.14	1.09	1.34
OPC325/3	4.68	1.06	5.97	1.06	1.29
OPC450/1	6.39	1	9.06	1	2.67
OPC450/2	9.01	1.41	11.24	1.24	2.23
OPC450/3	9.52	1.49	12.31	1.36	2.79

explanation of a denser binder phase with high replacement level is questionable.

For the FA concrete, greater retardation was observed at higher replacement levels. As replacement level was increased to 30%, the initial and final setting times increased by factors of 1.6 and 1.5, respectively. At the same replacement level, the results of Eren et al. [6] showed that the initial and final setting times increased by factors of 1.28 and 1.21, respectively. One of the reasons for this difference could be the use of a superplasticiser in the present investigation. Recent investigations by Naik and Singh [7] using FA from different sources showed that at 30% replacement level, the relative setting times tend to vary from 1.6 to 2.3. The effect of increasing the level of high lime FA was also found to increase the setting times of concrete [8].

In the same manner as the concrete containing SF and FA, the effect of increasing the replacement level of GGBS is to extend the setting times of concrete. Of all the 13 concrete mixes (Table 4), the concrete containing 60% GGBS exhibited the greatest retardation in both initial and final setting times. In fact at replacement levels of 40% and greater, the retarding effect of GGBS was rather excessive, the initial and final setting times being greater than 11 and 17 h, respectively. Therefore, for certain types of concrete work, such as slip forming and cold weather concrete the use of an accelerating admixture may be required. The test results of Eren et al. [6] indicated that the use of GGBS up to a 50% replacement level increased the setting time of concrete at normal temperature. Also, Hogan and Meusal [9] reported marginal increases in setting times when OPC was replaced with GGBS. However, in comparison, the results of the present investigation indicated greater setting times, which could be attributed to the use of a superplasticiser.

When the difference between the final and initial setting times of the concrete containing mineral admixtures is considered as shown in Table 4, it is clear that the concrete mixes containing SF and MK show smaller or comparable differences when compared to the OPC mix. On the other hand, the concrete containing FA and GGBS exhibit larger differences. This seems to suggest that SF and MK accelerate the hydration of cement after the time of initial set. The very high surface area of SF has been associated with its accelerating effect on cement hydration [26]. MK also possesses an accelerating effect on the hydration of cement [27,28].

The setting times of the concrete mixes containing mineral admixtures relative to those of the OPC concrete, as given in Table 4, are plotted as a function of replacement level of mineral admixtures as shown in Figs. 4–7. From these figures, the relative setting times of concrete containing mineral admixtures can be generally considered to be approximate linear functions of mineral admixtures replacement level as follows:

$$\mathrm{RST} = A + B(RL), \tag{2}$$

where RST is the relative setting time (RIST the relative initial setting time, RFST the relative final setting time), A the intercept to y-axis, ≈ 1, B the slope of the line with the relative setting time axis and RL is the replacement level of mineral admixtures in percent.

The values of A, B and the correlation coefficients, R^2 are given in Table 6. It is clear that except in the case of

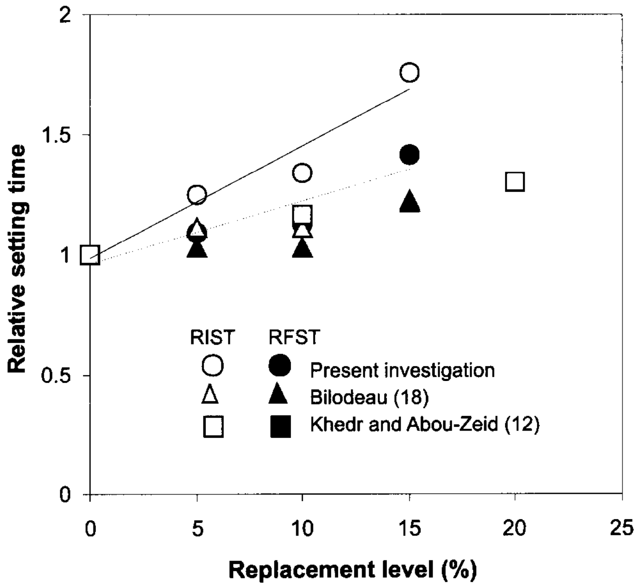

Fig. 4. Effect of silica fume on the setting times of concrete. Lines are regression fits for present investigation.

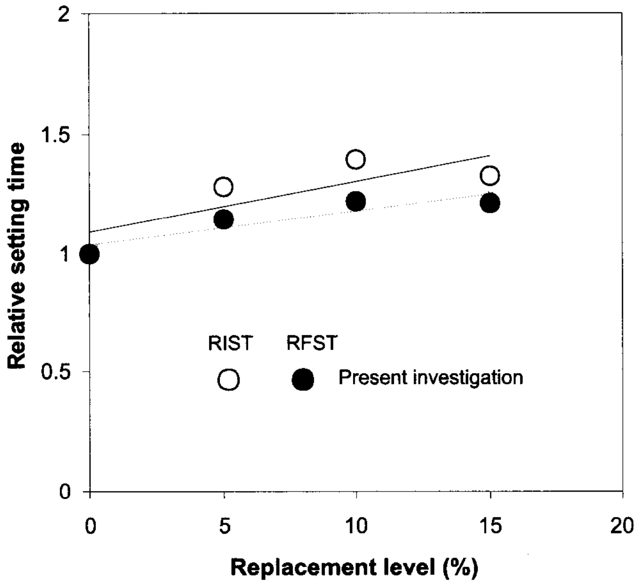

Fig. 5. Effect of metakaolin on the setting times of concrete. Lines are regression fits for present investigation.

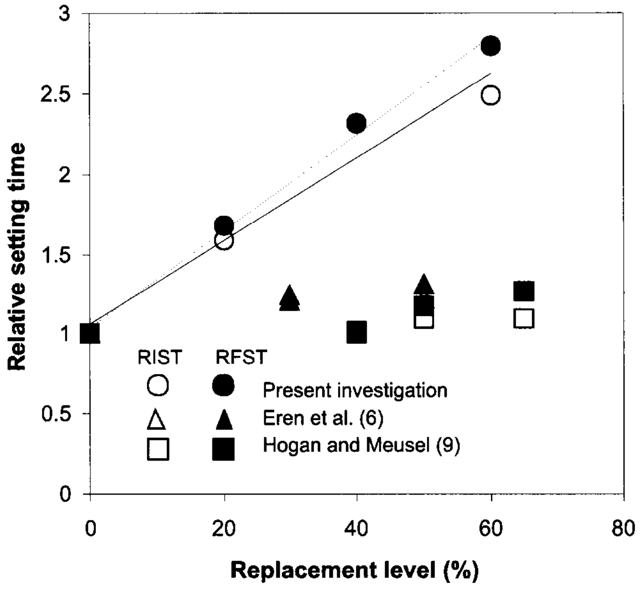

Fig. 7. Effect of ground granulated blast-furnace slag on the setting times of concrete. Lines are regression fits for present investigation.

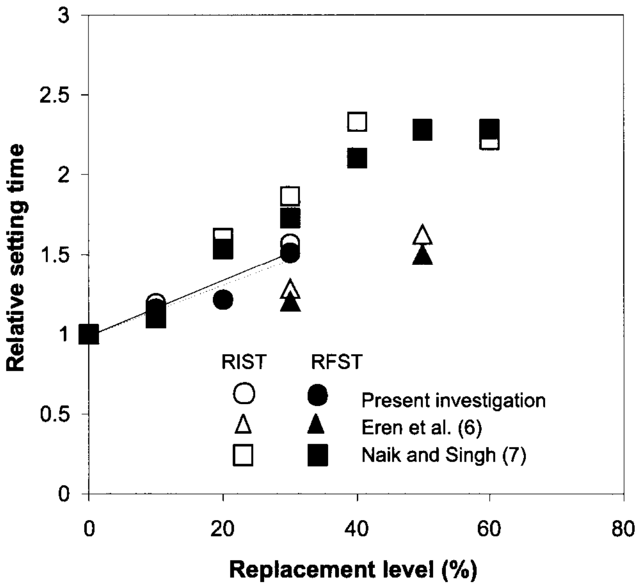

Fig. 6. Effect of Fly ash on the setting times of concrete. Lines are regression fits for present investigation.

concrete containing MK, there are reasonable correlation coefficients for the linear relationship between relative setting times and levels of mineral admixtures.

An attempt has been made to compare the results obtained in this study on concrete containing SF, FA and GGBS with those from previous investigators, as shown in Figs. 4, 6 and 7, respectively. It is emphasized that only the results, which have a minimum of two replacement levels apart from the control and within the range of normal replacement levels, were considered. For the same type of cement replacement material, different trends were obtained from the results of different researchers. The reason is that the setting characteristics of concrete are influenced by a number of other factors such as water/binder ratio, type and characteristics of cement, type and source of mineral admixtures, initial and curing temperature, soluble alkalies, chemical admixtures, etc. [5–19]. Consequently, the assumption of a linear relationship between relative setting times and replacement level is probably an oversimplification, and the other influencing factors will have to be quantified.

5. Conclusions

From the results presented in this paper, the conclusions are:

1. Penetration resistance of concrete with and without admixtures can be expressed as an exponential function of time.
2. The general effect of SF, MK, FA and GGBS is to retard the setting times of high-strength concrete.
3. The SRA has negligible effect on the setting times of normal strength concrete, but it has a significant retarding effect when used in combination with a superplasticiser in high-strength concrete.
4. The influence of increasing the levels of SF, FA and GGBS is to provide greater retardation in the setting times of high-strength concrete. The effect of GGBS at replacement levels of 40% and higher causes excessive retardation in setting times as the initial and final setting times were greater than 11 and 17 h,

Table 6
Coefficients of Eq. (2) for the results of the present investigation

Mineral admixtures	Initial setting time			Final setting time		
	A	*B*	R^2	*A*	*B*	R^2
SF	0.982	0.0474	0.936	0.965	0.026	0.854
MK	1.094	0.0198	0.562	1.039	0.0138	0.761
FA	0.984	0.0174	0.894	0.984	0.0159	0.929
GGBS	1.065	0.026	0.955	1.043	0.0302	0.995

respectively. For high-strength concrete containing MK, there was a progressive increase in the retarding effect up to 10% replacement levels. However, at a higher replacement level of 15%, the retarding effect appears to reduce.

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