

Influence of Superplasticizer Type on the Compressive Strength of RPM

By L. Coppola, R. Troli, A. Borsoi, P. Zaffaroni, M. Collepardi

Synopsis: Reactive powder mortar (*RPM*) mixtures cured at room temperature with different portland cement, silica fume and steel fibers were manufactured. The influence of the superplasticizer type on the *RPM* performance - in terms of *w/c* and compressive strength - was studied.

The acrylic polymer (*AP*) admixture performed better than the naphthalene (*SNF*) or melamine (*SMF*) based superplasticizers in regard to lower water-cement ratio and higher compressive strength at ages after 3 days.

The 1-day compressive strength of the *RPM* with the *AP* admixture was much lower than that of the corresponding mixtures with *SNF* or *SMF* when a C_3A -free portland cement with a low specific surface area was used. This was due to a strong retarding effect of the early hydration when this cement was used in combination with some silica fume types. With other portland cements, the retarding in the early hydration caused by *AP* did not occur and therefore the 1-day compressive strength was quite high.

The 28-day compressive strength of *RPM* specimens, cured at room temperature, were strongly dependent on the type of cement, silica fume and superplasticizer. However, the highest values obtained in this investigation (160-180 MPa) were lower than those reported by the inventors of *RPM* (170-230 MPa).

Keywords: Acrylic resins; compressive strength, melamine resins, naphthalene; plastics, polymers and resins, silica fume; superplasticizers

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INTRODUCTION

Reactive powder mortar (*RPM**) is a special high-strength, fiber-reinforced, superplasticized, silica-fume system with improved homogeneity because traditional aggregate are replaced by very fine sand with particle size in the range of 150-400 μm (1). Potential applications of *RPM* include prestressed structures without passive reinforcement (2, 3), pressure precast pipes (4), impermeable containers for hazardous fluids or nuclear wastes (5).

In previous investigations the influence of the cement type and silica fume brand on the water-cement ratio (w/c) and compressive strength of *RPM* was investigated (6, 7).

This paper reports the influence of the superplasticizer type (naphthalene, melamine and acrylic polymer) on the *RPM* performance in terms of water-cement ratio and compressive strength.

*In the original work (1), the reactive powder mortars were erroneously called "reactive powder concretes" (*RPC*). Hereafter, they will be referred to reactive powder mortars (*RPM*).

MATERIALS

Two ASTM Type V portland cements (*A* and *B*) were selected. Table 1 shows the composition and the properties of these cements. The main differences were the C_3A content and the Blaine fineness: the C_3A content was 0% and 4% whereas the specific surface area was 340 and 530 m^2/kg for cement *A* and *B*, respectively.

Many other portland cements were used to study the performance of *RPM*. However, for the sake of brevity, only the results with cement *A* and *B* will be examined in the present paper: Cement *A* (a C_3A -free portland cement with a low specific surface area) was selected for its peculiar behavior relative to all the other cements; Cement *B* is representative of typical portland cements.

Three silica fume brands (*W*, *G*, *D*) were used which were different with respect to colour (white, grey and dark respectively), particle size, minor components and unburnt content (Table 2). The most important characteristics seems to be the bulk gravity which is much lower for the *G* silica fume than for the others. This indicates that the *W* and *D* silica fume brands were available in a densified form, whereas the *G* brand was a loose un-compacted silica fume.

Fine natural sand (150-400 μm) and pure precipitated silica ($SiO_2 > 99.8\%$) were also used in agreement with the composition proposed by Richard and Cheyrezy for *RPM* with 200 MPa compressive strength (1).

Two steel fibers were used with an aspect ratio of 31 (length: 11 mm, diameter: 0.35 mm) and 72 (length: 13 mm, diameter: 0.18 mm).

In general, the fibers with an aspect ratio of 31 were used; however for some special mixtures fibers with the aspect ratio of 72 were used.

Three different types of superplasticizers were used:

- a) 30% aqueous solution of acrylic polymer (*AP*);
- b) 40% aqueous solution of sulfonated melamine-formaldehyde condensate (*SMF*);
- c) 40% aqueous solution of sulfonated naphthalene-formaldehyde condensate (*SNF*).

Details on the above mentioned superplasticizers can be found elsewhere (8, 9).

MIXTURES

Table 3 shows the mixture proportion of the powder ingredients including the solid portion of the superplasticizer aqueous solutions. The amount of superplasticizer in manufacturing *RPM* was in general 4 to 6 times higher than that used for usual concrete mixtures.

The amount of the dry *AP* superplasticizer was 1.36% by mass of cement as

in the original mixture proposed by Richard and Cheyrezy (1). The amount of the dry *NSF* or *MSF* superplasticizer (1.78%) was selected after preliminary tests in order to find the best performance in terms of water reduction at a given workability level. In other words, no significant further reduction in the mixing water was recorded when amounts of *SNF* or *SMF* higher than 1.78% were used.

After mixing all the ingredients including water in a laboratory pan mixer for 5 min, it was observed that the fresh mixtures were very sticky. Therefore the original flow table test (with 10 drops) did not appear to be adequate to assess the workability behaviour under the vibration required to compact the sticky mix into the molds. Then a modified flow table test was used by substituting a Vebè vibrating table (10 seconds of vibration) for the original flow table. Eighteen different mixtures - all at same workability level: 150-160 mm for the modified flow table test - were manufactured by changing the portland cements (*A* and *B*), the silica fume brands (*W*, *E* and *D*), and the superplasticizer types (*AP*, *SNF* and *SMF*). Tables 4 and 5 show the mixture composition, the water to cement ratio (*w/c*) as well as the water to binder ratio (including silica fume) of the 18 above mixtures manufactured with cement *A* and *B*, respectively. The amount of water coming from the aqueous solution of the superplasticizers was included in the *w/c* as well as in the water to binder ratio.

CASTING AND CURING

Cube specimens (40 mm) were consolidated by vibration and then cured at room temperature (20°C).

Compressive strength were measured at 1, 2, 3, 14 and 28 days. In some cases XRD analysis was carried out to study the effect of the admixture on cement hydration.

RESULTS

The results will be examined regard to water to cement ratio and compressive strength.

Water to Cement Ratio

The *w/c* of the mixtures (Table 4 and 5) was affected by the type of cement, silica fume and superplasticizer:

- Independently of the superplasticizer type, mixtures with Cement *A* (a C_3A -free portland cement with low specific surface area) always needed lower

w/c as compared to the mixtures with Cement *B*; similar results were obtained with other portland cements in the presence of the *AP*-superplasticizer (6, 7);

- Regardless of the superplasticizer type, the w/c in mixtures with white silica fume was significantly lower than in those with dark silica fume, whereas the grey silica fume performed a little better than the dark one.
- Independently of the cement and silica fume types, the w/c in the presence of the acrylic polymer was much lower than that with the *NSF*- or *MSF*-based superplasticizer, although the amount of *AP* was lower than that of the other admixtures (1.36% versus 1.78% by cement mass).

Therefore the best performance in terms of the lowest w/c occurred with the following specific combination: C_3A -free portland cement, white silica fume and acrylic polymer (Table 4, Mixture No. 1 : $w/c = 0.18$). On the other hand, the highest w/c was obtained with cement *B*, dark silica fume and melamine-based superplasticizer (Table 5, Mixture No. 18 : $w/c = 0.42$). However, not necessarily the highest compressive strength corresponded to the lowest w/c , and this is shown below.

COMPRESSIVE STRENGTH

Tables 6 and 7 show the compressive strength results of Mixtures 1 to 9 and 10 to 18, respectively, at 1 to 28 days.

Figures 1 to 3 show the compressive strength as a function of the hydration time (1-28 days) in mixtures with cement *A* and steel fibers with the aspect ratio of 31. In the presence of white silica fume (Fig. 1), the strength at early ages with the *AP*-based superplasticizer was much lower than that with the *SNF*- or *SMF*-based admixtures. However, at later ages the mixture incorporating *AP* attained higher strength with respect to the corresponding cement mixtures with the *SNF* or *SMF* superplasticizer: the 28-day compressive strength was about 160 MPa with *AP* versus about 100 MPa with *SNF* or *SMF*. This behavior can be explained in terms of a strong retardation of the cement hydration caused by the *AP* superplasticizer at early ages, whereas at later ages the lower w/c of this mixture (0.18) is responsible for the higher strength level. The early strong retardation caused by the *AP* admixture can be ascribed to the high dosage of this superplasticizer (4-5 times higher than the usual dosage, i.e. 0.3% of dry polymer by cement mass) and the specific combination of the white silica fume with a C_3A -free portland cement having a low specific surface area.

The substitution of the grey silica fume for the white one reduced the retardation of the cement hydration at 1-day caused by the *AP* admixture but also reduced the 28-day compressive strength (Fig. 2) because of the higher w/c (0.22 vs. 0.18, Table 4). The acrylic polymer performed better than *SNF*- and *SMF*-based superplasticizers in terms of both lower w/c (Table 4) and higher strength after 2 days.

In the presence of the dark silica fume (Fig. 3), again the hydration of the C₃A-free portland cement was strongly retarded by the *AP* superplasticizer. However, at later ages (after 3 days) there is no significant difference in the compressive strength of superplasticized mixtures regardless of the specific admixture.

The only property of silica fumes which could explain their different effect on the 1-day strength of *RPM* with a C₃A-free portland cement seems to be the densification characteristics. The *G* silica fume, with a bulk gravity of 0.12 g/cm³ (Table 1), was a loose and un-densified powder, whereas *W* and *D* silica fumes (with a bulk gravity of 0.45-0.47 g/cm³) were available in form of compacted and densified powders. Therefore, since the individual grains of *W* and *D* silica fumes were less dispersed at the end of the mixing time (5 min), they adsorbed a lower amount of the *AP*-superplasticizer and this caused a strong retardation in the cement hydration with respect to the loose and un-densified *G* silica fume. This effect - as it will be examined later - is aggravated by the presence of a low-fineness C₃A-free portland cement which adsorbs a lower amount of superplasticizer with respect to other portland cements with higher fineness and richer C₃A content (10).

Figures 4 to 6 show the compressive strength as a function of the hydration time (1-28 days) in mixtures with cement *B* and steel fibers with the aspect ratio of 31. These results are representative of the performance of *RPM* with other portland cements as obtained in the laboratories of the authors of the present paper.

In general, the performance in terms of *w/c* was much better with Cement *A* (Table 4) than with Cement *B* (Table 5). However, the compressive strength development was not always in agreement with the *w/c* data. For instance, the mixture No. 10 with cement *B*, white silica fume and *AP* superplasticizer (*w/c* = 0.25) performed much better than the corresponding mixture No. 1 with cement *A* (*w/c* = 0.18) in terms of 1-day compressive strength: 45 MPa (Fig. 4) versus 1 Mpa (Fig. 1). This difference can be ascribed to the specific combination of the *AP* admixture (at very high dosage of 1.37%) with a low specific surface area C₃A-free portland cement which was responsible for the retardation of the early hydration of cement *A*.

The XRD data of the anhydrous cements, as well as of the cements hydrated at 1 and 28 days, confirmed this hypothesis: Fig. 7 shows the C₃S and C₂S XRD peaks (2θ at 32.2 and 32.6°) of Cement *A* and *B* in the presence of white silica fume and *AP* superplasticizer. The hydration degree of Cement *A* at 1 day was negligible - in terms of the 32.6° peak reduction - and much lower - in terms of the 32.2° peak decrease - than that recorded with Cement *B*, and this explains the higher strength of Cement *B* with respect to Cement *A*. At longer ages, the difference in the hydration degree between Cement *A* and *B* decreased and therefore the 28-day compressive strength of the mixture with Cement *A* was higher because of the lower *w/c* than that of the mixture with Cement *B*.

These results are, in general, in agreement with other published literature on

the relationship between the cement type and superplasticizer effect (10): cements with reduced amount of C_3A and low specific surface area - such as Cement *A* in the present study - adsorb a lower amount of superplasticizer which, therefore, is available in the aqueous phase in larger amounts and is responsible for a greater dispersion effect of cement particles as well as for a stronger retardation of C_3S and C_2S . This model can explain why with all admixtures of this work - regardless of their nature - the superplasticizing effect, in terms of reduced w/c at equal workability, was higher (Table 4 and 5) and the 1-day compressive strength was lower (Fig. 1-6) when Cement *A* was used instead of Cement *B*. What is peculiar in the present study is that this effect, particularly with a C_3A -free portland cement, is much more significant with the *AP* admixture than with the *SNF*- or *SMF*-based superplasticizer. This seems to be related with the stronger retardation in the hydration caused by the *AP*-superplasticizer at the dosage as high as that used in this study.

The 28-day compressive strength of *RPM* in the present work was in the range of 70-160 MPa with the best performance when cement *A* was used in combination with white silica fume and the *AP* superplasticizer (Fig. 1) and the worst one when cement *B* was used with dark silica fume and the *SMF*-based superplasticizer (Fig. 6).

All these data are not in agreement with the compressive strength results (170-230 MPa) reported by Richard and Cheyrezy (1). The main difference between the materials used for the present study and those described by Richard and Cheyrezy seems to be the aspect ratio of the steel fibers which was higher (72 versus 31) in the Richard and Cheyrezy tests than in the present investigation (Fig. 1-6). Supplementary tests with steel fibers of the same aspect ratio as that of fibers used by Richard and Cheyrezy were carried out. Figure 8, for instance, shows the effect of the fiber aspect ratio on the compressive strength of the cement mixture No. 4 (Cement *A*, grey silica fume, *AP* superplasticizer). Similar results were obtained with other combinations of cement, silica fume and superplasticizer: the compressive strength increased by 20-30 MPa by increasing the fiber aspect ratio from 31 to 72. In general, change in the aspect ratio of fibers does not affect significantly the compressive strength of cement mixtures. The relatively high amount of steel fibers used in the present study (20% by mass of cement) could justify this unexpected result. However, even with the most favorable aspect ratio of fibers, the compressive strength of the *RPM* mixtures studied in the present work and *cured at room temperature* (Fig. 8) was lower than the 200 MPa value reported by Richard and Cheyrezy for apparently the same cement mixture. Only with a special heat treatment at a temperature as high as 160°C the authors were able to attain to compressive strength levels slightly over 200 MPa (7).

CONCLUSIONS

The acrylic polymer admixture performed better than *SNF*- or *MSF*-based superplasticizers in terms of lower *w/c*, regardless of the cement and silica fume type used in manufacturing *RPM* mixtures.

However, when a C_3A -free portland cement with low specific surface area ($340 \text{ m}^2/\text{kg}$) was used, the 1-day compressive strength was much lower with the *AP* admixture than with the *SNF*- and *SMF*-based superplasticizers particularly in the presence of white or dark silica fumes. This behavior should be related with a specific retarding effect on the early hydration of this cement caused by *AP* in the presence of densified silica fumes (white and dark-colored). With other portland cements, and in the presence of a loose un-densified silica fume (grey-colored), the *AP* superplasticizers did not cause any early retardation.

At later ages (after 3 days) the compressive strength of *RPM* mixtures with the *AP* admixture were always higher than with *SNF*- or *SMF*-based superplasticizers independently of the cement and silica fume type.

By increasing the aspect ratio of the steel fibers from 31 to 72, the compressive strength increased by 20-30 MPa. However, even with the most favorable fiber aspect ratio, the compressive strength of the specimens cured *at room temperature* was at most 160-180 MPa and therefore lower than the 200 MPa strength level as claimed by Richard and Cheyrezy (1).

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Table 1 - Composition and properties of portland cements.

ASTM type	V	V
ENV 197/1 type	CE I 42.5R	CE I 52.5R
Cement Brand	A	B
SiO ₂ (%)	20.59	20.79
Al ₂ O ₃ (%)	3.66	4.32
Fe ₂ O ₃ (%)	6.10	4.40
CaO (%)	63.78	64.58
MgO (%)	0.95	1.03
K ₂ O (%)	0.48	0.52
Na ₂ O (%)	0.26	0.14
SO ₃ (%)	2.50	2.68
C ₃ A (%)	0	4.00
Blaine (m ² /kg)	340	530

Table 2 - Composition and properties of silica fume.

Silica fume brand	W	G	D
Colour	White	Grey	Dark
SiO ₂ (%)	94.43	98.87	95.12
Al ₂ O ₃ (%)	2.48	0.01	0.02
SO ₃ (%)	0.01	0.23	0.54
K ₂ O (%)	0.04	0.08	0.91
Na ₂ O (%)	0.01	0.00	0.01
CaO (%)	0.04	0.23	0.49
MgO (%)	0.01	0.01	0.02
TiO ₂ (%)	0.21	0.08	0.23
MnO ₂ (%)	0.20	0.01	0.21
Fe ₂ O ₃ (%)	0.46	0.30	0.40
mean particle size (µm) from laser granulometry:			
- without superplasticizer	0.69	13.87	63.59
- with superplasticizer	0.62	0.76	0.73
Unburnt (%)	0.0	0.0	1.6
Bulk gravity (g/cm ³)	0.45	0.12	0.47

Table 3 - Composition of powder mixtures by mass (without the mixing water).

Mixture with	AP	SNF	SMF
Portland cement	100	100	100
Silica fume	24	24	24
Precipitated silica	1	1	1
Fine sand	110	110	110
Steel fibers	20	20	20
Superplasticizer	1.36	1.78	1.78

Table 4 - Composition of superplasticized mixtures with the C₃A-free portland cement (A) and steel fibers with aspect ratio 31.

Mixture No.	1	2	3	4	5	6	7	8	9
Silica fume brand	White			Grey			Dark		
Superplasticizer	AP	SNF	SMF	AP	SNF	SMF	AP	SNF	SMF
Composition (kg/m ³):									
- Portland cement	937	900	876	903	876	847	872	854	847
- Silica fume	225	216	210	217	210	203	209	205	203
- Precipitated silica	9.4	9.0	8.8	9.0	8.8	8.5	8.7	8.5	8.5
- Fine sand	1031	990	963	993	964	932	959	939	932
- Steel fibers	187	180	175	181	175	169	174	171	169
- Superplasticizer*	12.7	16.0	15.6	11.3	15.6	15.1	11.9	15.2	15.1
- Water	169	198	219	199	219	246	227	239	246
W/C	0.18	0.22	0.25	0.22	0.25	0.29	0.26	0.28	0.29
W/(C + SF)	0.14	0.18	0.20	0.18	0.20	0.23	0.21	0.22	0.23
Flow table (mm)	160	150	150	160**	150	150	160	160	160

* Dry polymer

** 150 mm when steel fibers with aspect ratio 72 were used

Table 5 - Composition of superplasticized mixtures with the low C₃A content (4%) portland cement (B) and steel fibers with aspect ratio 31.

Mixture No.	10	11	12	13	14	15	16	17	18
Silica fume brand	White			Grey			Dark		
Superplasticizer	AP	SNF	SMF	AP	SNF	SMF	AP	SNF	SMF
Composition (kg/m ³):									
- Portland cement	879	847	833	850	793	768	843	787	763
- Silica fume	211	203	200	204	190	184	202	189	183
- Precipitated silica	8.8	8.5	8.3	8.5	7.9	7.7	8.4	7.9	7.6
- Fine sand	967	932	916	935	872	845	927	866	953
- Steel fibers	176	169	167	170	159	154	169	157	153
- Superplasticizer*	12.0	15.1	14.8	11.6	14.1	13.7	11.5	10.7	13.6
- Water	220	246	258	247	293	315	254	299	321
W/C	0.25	0.29	0.31	0.29	0.37	0.41	0.30	0.38	0.42
W/(C + SF)	0.20	0.23	0.25	0.23	0.30	0.33	0.24	0.30	0.34
Flow table (mm)	155	150	150	150	155	150	155	160	155

* Dry polymer

Table 6 - Compressive strength as a function of hydration time for Mixtures No. 1-9 with Cement A and steel fibers with aspect ratio 31.

TIME (days)	COMPRESSIVE STRENGTH (MPa) of Mixture No:								
	1	2	3	4	5	6	7	8	9
1	0.0	56.0	62.4	28.8 (54.4)*	60.8	41.6	0.0	44.8	35.2
2	67.2	76.8	83.2	72.0 (96.0)*	89.6	62.4	44.8	67.2	56.0
3	96.0	80.0	86.4	100.8 (115.2)*	88.1	78.4	76.8	86.4	68.8
14	132.8	108.8	116.8	139.2 (166.4)*	124.8	108.8	108.8	102.4	92.8
28	150.4	110.0	118.1	150.4 (180.4)*	136.0	118.4	105.6	100.8	93.0

* Fibers with aspect ratio 72

Table 7 - Compressive strength as a function of hydration time for Mixtures No. 10-18 with Cement B and fibers with aspect ratio 31.

TIME (days)	COMPRESSIVE STRENGTH (MPa) of Mixture No:								
	10	11	12	13	14	15	16	17	18
1	41.6	54.4	64.0	75.2	59.2	44.8	57.6	46.4	38.0
2	70.4	83.2	92.8	86.4	72.0	59.2	72.0	60.8	44.8
3	86.4	96.0	102.4	99.2	83.4	70.4	75.2	67.2	57.6
14	142.4	124.8	128.0	129.6	112.0	100.0	102.4	86.4	72.0
28	155.2	134.4	140.8	144.0	131.2	116.8	100.2	92.8	80.0

COMPRESSIVE STRENGTH (MPa)

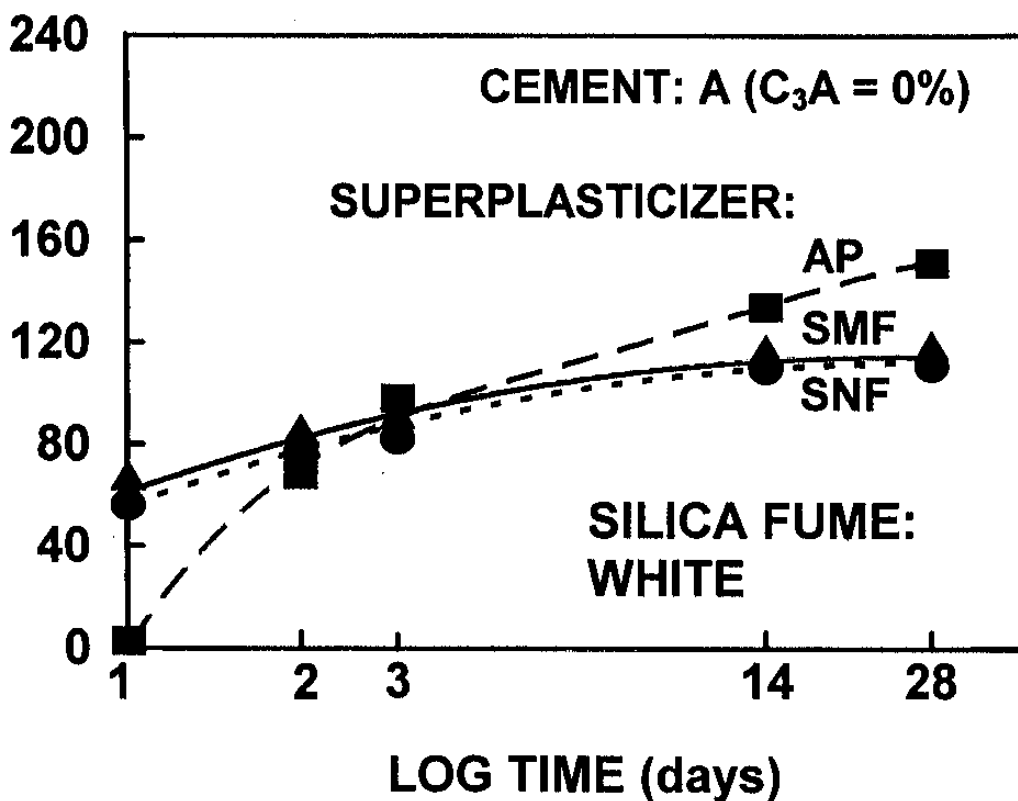


Fig. 1—Compressive strength versus time for cement A, white silica fume, and steel fibers with as aspect ratio of 31

COMPRESSIVE STRENGTH (MPa)

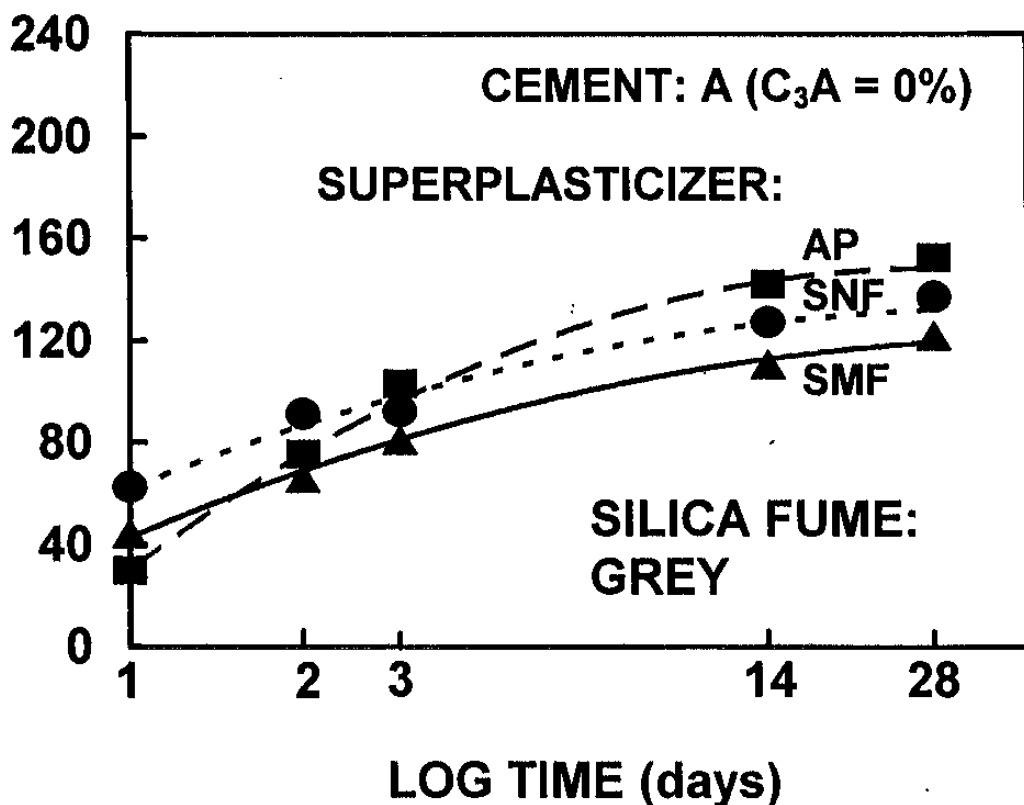


Fig. 2—Compressive strength versus time for cement A, gray silica fume, and steel fibers with as aspect ratio of 31

COMPRESSIVE STRENGTH (MPa)

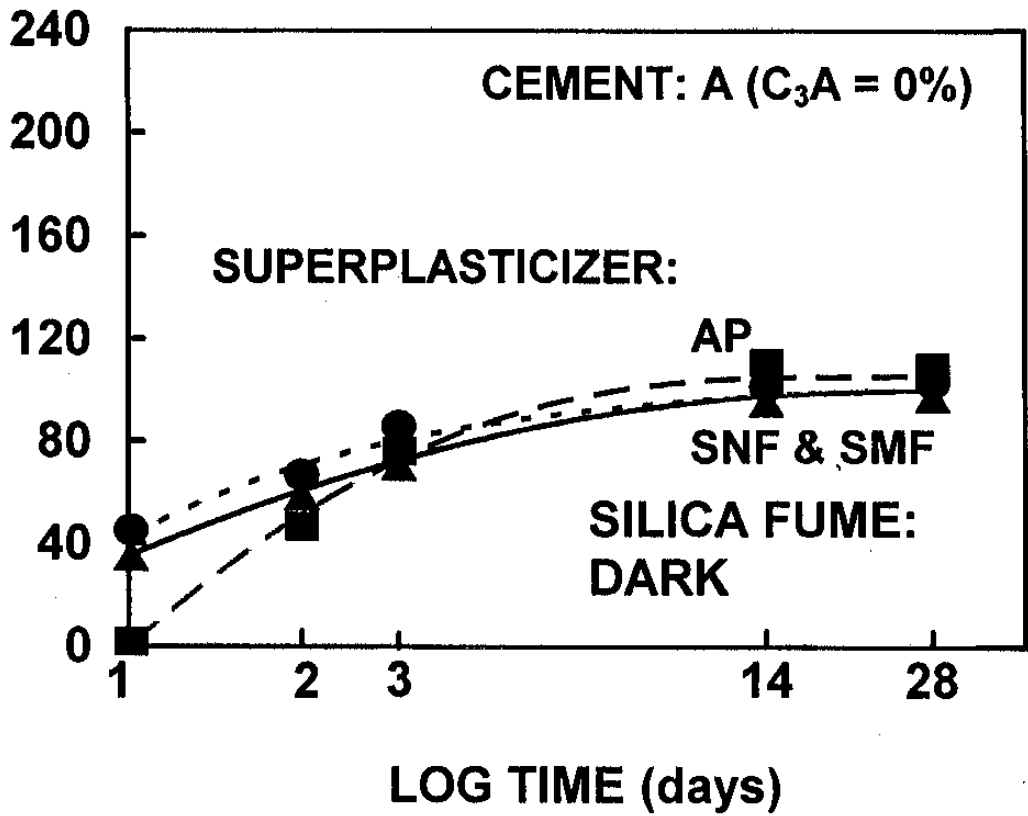


Fig. 3—Compressive strength versus time for cement A, dark silica fume, and steel fibers with as aspect ratio of 31

COMPRESSIVE STRENGTH (MPa)

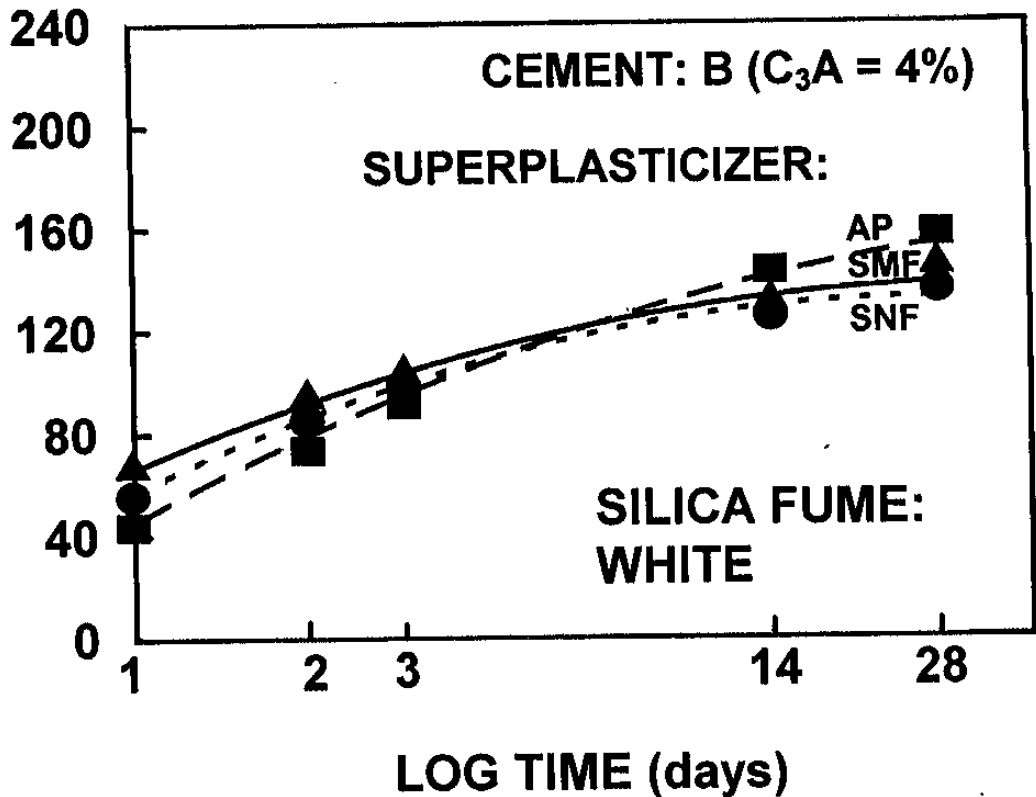


Fig. 4—Compressive strength versus time for cement B, white silica fume, and steel fibers with as aspect ratio of 31

COMPRESSIVE STRENGTH (MPa)

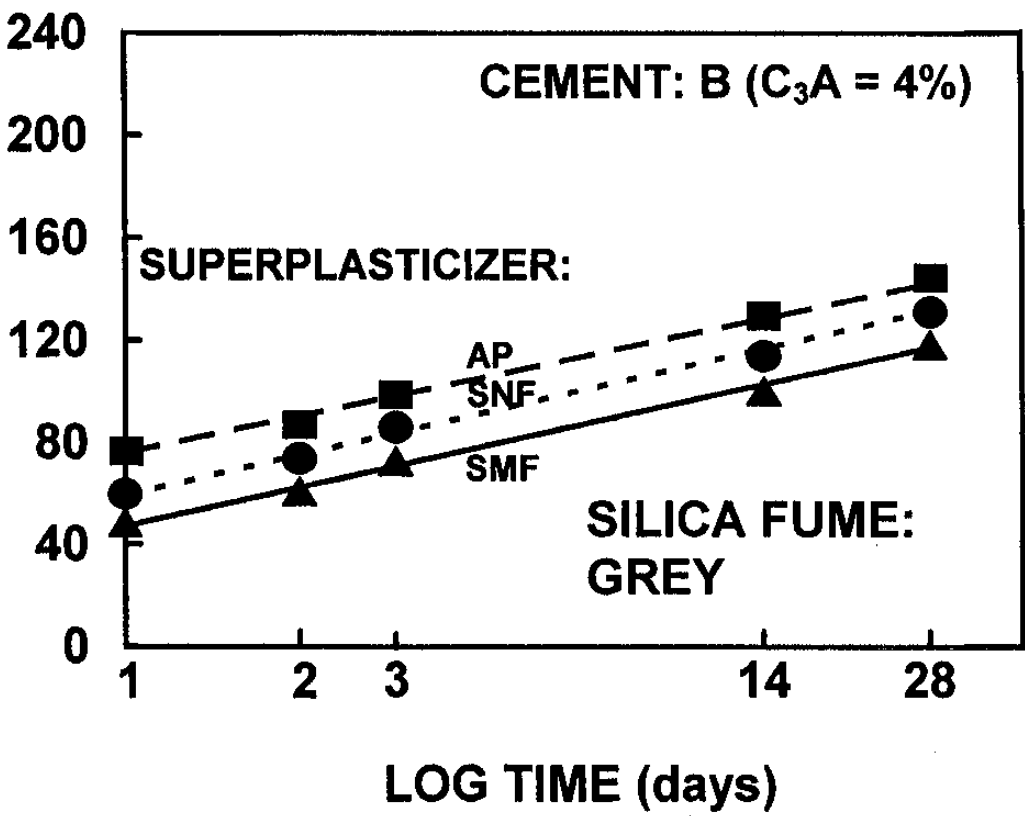


Fig. 5—Compressive strength versus time for cement B, gray silica fume, and steel fibers with as aspect ratio of 31

COMPRESSIVE STRENGTH (MPa)

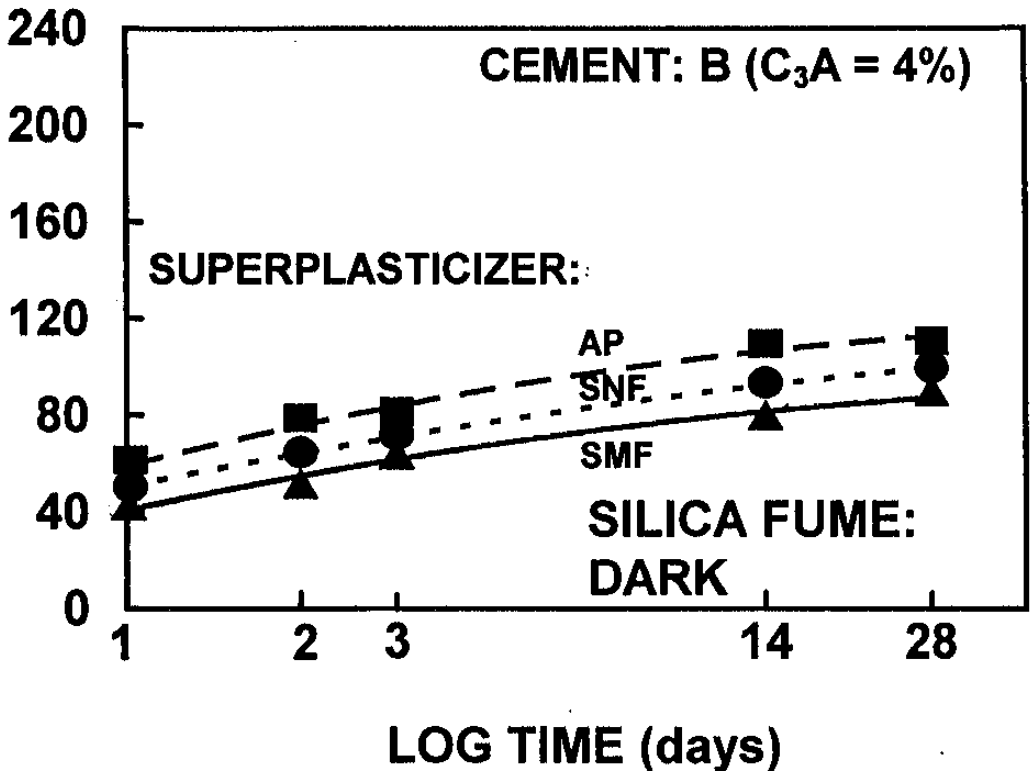


Fig. 6—Compressive strength versus time for cement B, dark silica fume, and steel fibers with as aspect ratio of 31

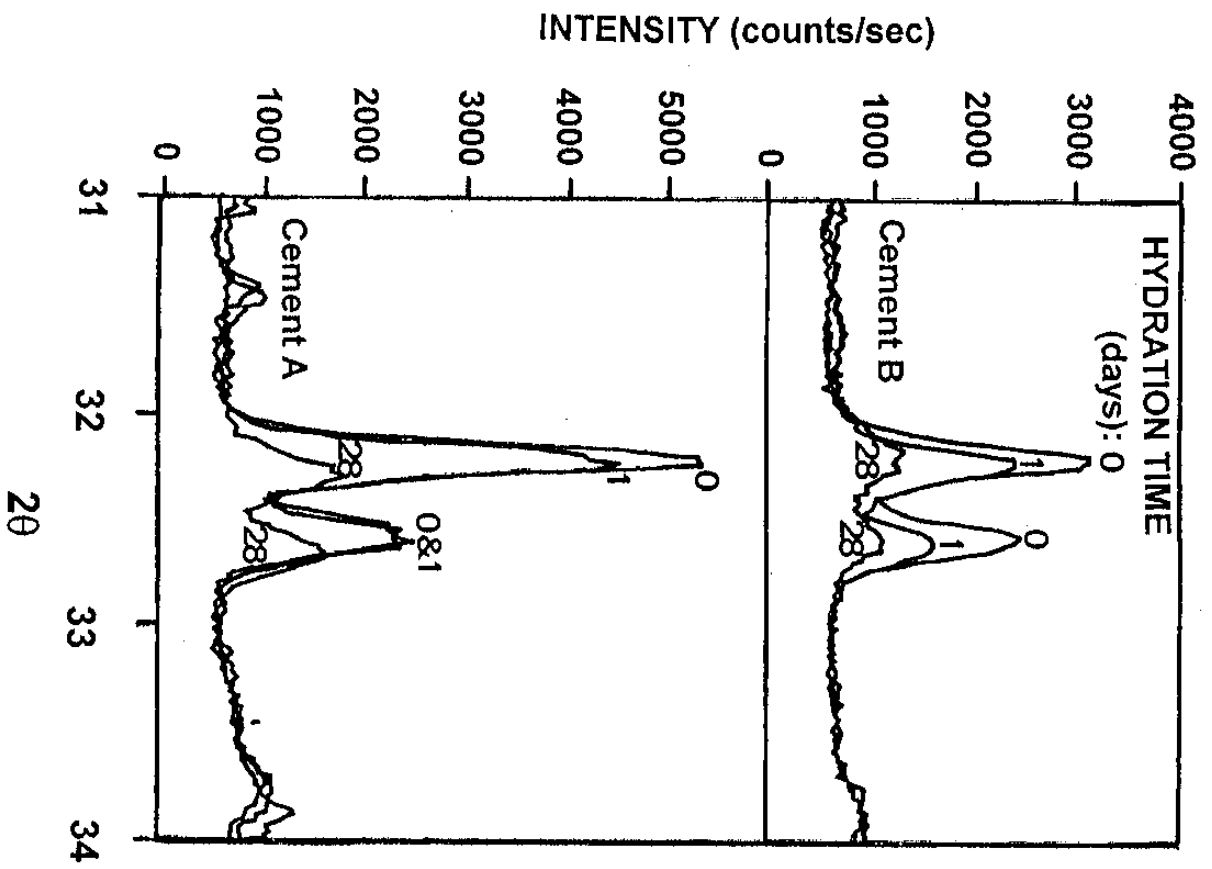


Fig. 7—The influence of cement (A or B) on the XRD of RPC with white silica fume and AP-superplasticizer (mixtures no. 1 and 10)

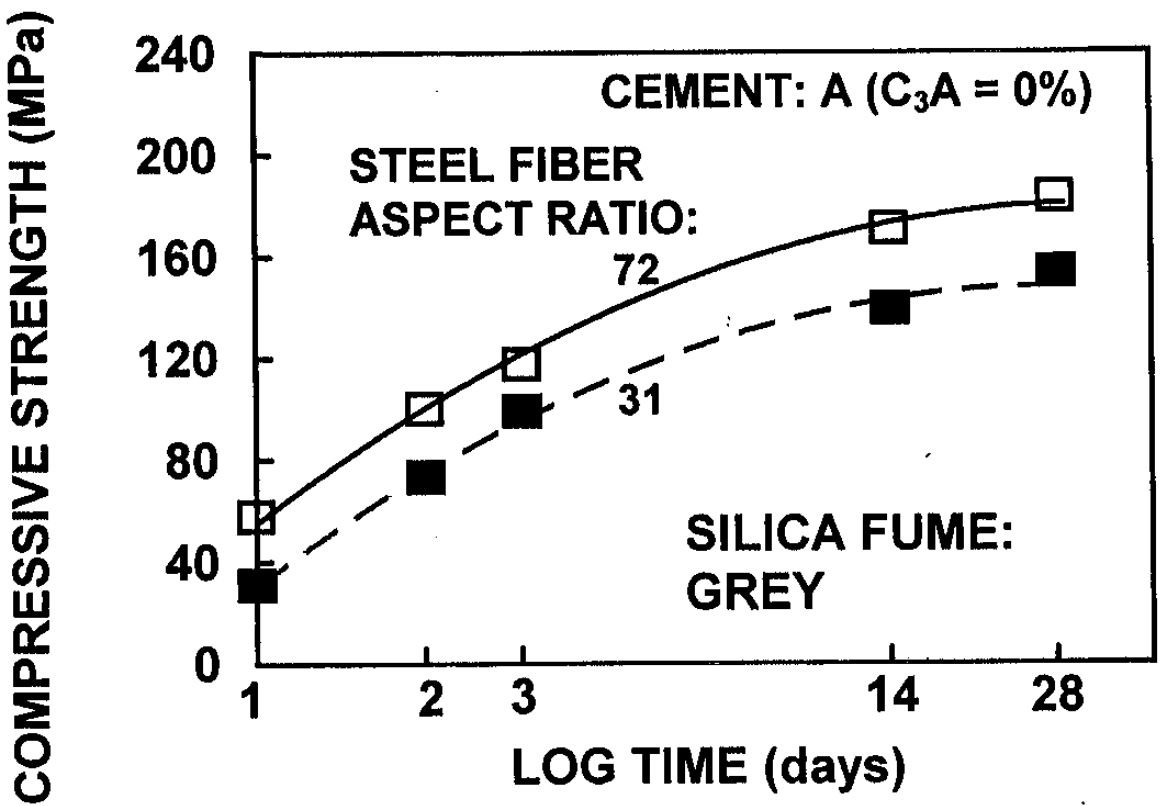


Fig. 8—The influence of steel fiber aspect ratio on the compressive strength versus time for cement A, gray silica fume and AP-superplasticizer (mixture no. 4 of table 4)