

## ACRYLIC BASED SUPERPLASTICIZER

BY

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### ABSTRACT

A research program, including physico-chemical and technological laboratory tests as well as field tests, has been carried out to study a new type of concrete superplasticizer based on the carboxylic acrylic ester (CAE) copolymer in comparison with sulfonated melamine or naphthalene formaldehyde condensate type (SMF or SNF).

The CAE copolymer can be used to manufacture zero slump-loss superplasticized concrete for a transportation time of at least 2 hours even at lower dosage than usually utilized for MSF or NSF based admixtures.

The compressive strength of CAE treated concrete is higher than that of the corresponding mixtures with MSF or NSF polymers at early ages and to a greater extent at later ages, independently of the curing temperature.

The mechanism of the CAE copolymer has been studied to explain the more effective superplasticizing action and the lower slump-loss effect by examining the adsorption zeta potential and X-ray diffraction analysis results.

**Keywords:** Acrylic superplasticizer, melamine superplasticizer, naphthalene superplasticizer, slump-loss, compressive strength.

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## INTRODUCTION

All the properties of hardened concrete, including strength and durability can be improved by reducing the water/cement ( $w/c$ ). However, at a given cement content ( $c$ ), the reduction in mixing water ( $w$ ) decreases the fresh mix workability so that concrete placing becomes very difficult at the work site. Therefore, special chemical admixtures (superplasticizers) are used to place low  $w/c$  concretes without sacrificing the workability of fresh mixes, particularly those devoted to high quality concrete structures.

The main components of the superplasticizers are substantially based on sulfonated melamine formaldehyde (SMF) condensate type or sulfonated naphthalene formaldehyde (SNF) condensate type in form of a 40% aqueous solution [1]. Both of these admixtures, at a dosage of about 1% by weight of cement, are able to transform a no-slump concrete into a self-levelling mixture with a slump increase of about 200 mm. However, when concrete mixture should be transported for a long time, particularly in hot weather, the initial slump level is lost to a significant extent.

Since slump-loss is un-avoidable because of the intrinsic requirement for cement mixes which should set and harden in a relatively short time, a right and proper compromise would be a zero slump-loss concrete mixture for 1-2 hours.

Fujiu et al. [2], Fukuda et al. [3], Yamakawa et al. [4] and Mitsui et al. [5] have studied alternative chemical composition to limit the slump-loss problem. Many of these superplasticizers are mainly based either on bicomponent admixtures containing the SNF polymer and a reactive polymer (in form of an insoluble precursor) as a slump-loss reducer [3, 4, 5], or a monocomponent polymer which is not soluble in mixing water but, under the alkaline environment formed in the aqueous phase in contact with cement, is slowly transformed into an aqueous soluble product having superplasticizing effect [2].

The purpose of the present work was to develop and examine a new water soluble monocomponent superplasticizer, based on carboxylated acrylic ester (CAE) copolymer, which is able to give a superplasticized concrete mixture with low  $w/c$  without significant slump-loss for 1-2 hours of the fresh mixture and strength reduction at early as well as at longer ages of the hardened concrete.

## MATERIALS

The cement pastes, the mortars and the concrete mixtures were made using the following materials.

### 1. Cement

Normal portland cement was used. The physical properties and chemical analysis of the portland cement are given in Table 1.

## 2. Aggregates

Natural sand (F.M. = 2.7) and gravel with nominal maximum size of 19.0 mm or 25 mm were used for making concretes.

## 3. Superplasticizers

A sulphonated naphthalene or melamine formaldehyde condensate, SNF or SMF (40% of polymer in aqueous solution) and a superplasticizer based on the carboxylic acrylic ester, CAE, (30% of polymer in aqueous solution) were used.

### MIXTURE PROPORTIONS

The composition of the concrete mixtures used to study the effect of the way of addition of superplasticizers on the concrete workability are given in Table 2. Table 3 shows the composition of concrete mixtures for laboratory and field test on slump-loss and compressive strength.

### EXPERIMENTAL

In the present paper the experimental results concerning the three following section will be shown:

- (i) physico-chemical tests on the mechanism of the new superplasticizer;
- (ii) laboratory tests on concrete technology;
- (iii) field tests on concrete transportation.

For each of the above three sections the CAE copolymer (in form of a 30% aqueous solution) will be examined in comparison with the other two main polymers (SMF and SNF in form of a 40% aqueous solution) usually utilized all over the world to produce superplasticized concretes. Figure 1 shows the chemical composition of the CAE copolymer beside the other two well known chemical polymers (SMF and SNF). The main difference between the new superplasticizing copolymer and the others is that the CAE copolymer contains carboxylic ( $\text{COO}^-$ ) instead of sulfonic ( $\text{SO}_3^-$ ) anionic groups as those present in the SMF or SNF polymers. An other important difference is the molar ratio of negative anionic groups ( $\text{COO}^-$  or  $\text{SO}_3^-$ ) per organic monomer unit (melamine, or naphthalene or acrylic group) which is 1 for SMF or SNF polymers and much lower than 1 in the CAE copolymer ( $m/(m + n) < 1$ ).

### PHYSICO-CHEMICAL TESTS ON THE MECHANISM OF SUPERPLASTICIZERS

The fluidizing effect of superplasticizers has been basically related [1] to the three following physico-chemical phenomena: dispersion, adsorption and electrical repulsion (zeta potential).

Superplasticizers cause dispersion into smaller agglomerates of cement particles which predominate in the cement paste of the concrete mixture. The dispersion effect is generally ascribed to the polymer adsorption on the cement surface and consequently to the development of

the same electrostatic (negative) charge on the cement particles. Therefore, the attractive forces existing among cement particles and causing agglomeration would be neutralized by adsorption of anionic polymers negatively charged, such as SNF or SMF for the  $\text{SO}_3^-$  group, on the surface of cement particles.

The experimental results of the present work do not confirm this mechanism for the superplasticizing action of the CAE, as it will be demonstrating later.

The fluidity of portland cement mortars with w/c of 0.40 and sand/cement of 3 has been measured (at 5 min) in the absence or in the presence of CAE, SMF or SNF polymers with different dosages of admixtures (0.2-0.6% of dry polymer by weight of cement). Figure 2 shows the flow table measurements of these mortars and indicates that the CAE copolymer performs much more effectively than the SMF or SNF polymers.

The aqueous phase of portland cement pastes (w/c = 2) has been analyzed by the total organic carbon (TOC) technique [6] to determine the residual concentration of polymer after 5 min of agitation, so that the content of admixture adsorbed on cement has been calculated. Figure 3 shows the percentage of polymer adsorbed as a function of the admixture dosage expressed as percentage of dry polymer by weight of cement. The adsorption of CAE copolymer (about 85%) appears to be a little higher than that recorded for the SMF (about 80%) or SNF (75%) polymers.

Figure 4 shows the results of zeta potential measurements on the same portland cement pastes determined by the multiangle electrophoretic light scattering (ELS) technique [7]. The zeta potential of cement particles treated by CAE appears to be much lower than those recorded in the presence of SMF or SNF. In particular, when 0.3% of CAE by weight of cement is used, the cement particles appear to be electrically neutral even though the corresponding mortar mixture, with the same percentage of admixture (0.3%), is much more fluid than the plain mixture and those with SMF or SNF polymers (Fig. 2).

All these results would indicate that the dispersion of cement particles, responsible for the fluidity increase caused by superplasticizer (Fig. 2), is not necessarily related to the electrostatic repulsion associated with zeta potential measurements (Fig. 4). It would seem that, at least for the CAE admixture, the polymer adsorption (Fig. 3) rather than the electrostatic repulsion is responsible for the dispersion of large agglomerates of cement particles into smaller ones and then for the remarkable increase in the fluidity of cement mixes.

The different mechanism of superplasticizing action performed by the CAE polymer could be related more to a steric hindrance effect rather than to the presence of negatively charged anionic groups ( $\text{COO}^-$ ). In other words, the polymer molecules of CAE by themselves on the surface of cement would hinder from flocculating into large and irregular agglomerates of cement particles. This mechanism would be in agreement with the relatively small number of negative anionic groups ( $\text{COO}^-$ ) in the CAE copolymer in comparison with those present as  $\text{SO}_3^-$  in the SMF and SNF polymers (Fig. 1).

## LABORATORY TESTS ON CONCRETE TECHNOLOGY

Concrete mixtures have been produced in the absence and in the presence of CAE, SMF or SNF polymers as superplasticizing admixtures. In the present section only laboratory tests concerning portland cement mixtures will be examined.

Preliminary tests have been carried out to study the effect of the way of addition of superplasticizers on the slump of concrete mixture. Table 4 summarizes some typical results concerning concrete mixtures, with portland cement content of about  $410 \text{ Kg/m}^3$ ,  $w/c$  of about 0.40, natural aggregates, coarse maximum size of 19 mm, percentage of sand in the aggregate of 40%. The superplasticizing effect of SMF or SNF is strongly depending on the way of addition, and a delayed addition of superplasticizer (after 1 min of mixing) appears to be much more effective than an immediate addition (superplasticizer with mixing water).

On the other hand, the effect of CAE polymer does not substantially depend on the way of addition and it performs as well as the SMF or SNF polymers in the delayed addition with a dosage which is much lower (0.30% versus 0.50 or 0.48% respectively).

It is worthwhile noting that the immediate addition is the most practical, safe and easy way to dose the superplasticizer at the batching plant, since dosing superplasticizer at the work site - corresponding to a delayed addition - consumes too much time and does not allow an accurate control of the final slump and admixture dosage.

All these results together mean that the CAE copolymer is potentially more effective than the SMF or SNF polymers from both a scientific and a practical point of view.

The influence of the polymer type on the slump-loss of superplasticized concrete mixture is shown in Fig. 5. Again, the CAE copolymer appears to be more effective than the SMF or SNF polymers (with delayed addition) in reducing the slump-loss of concrete mixes (portland cement content of about  $350 \text{ Kg/m}^3$ , natural aggregates, maximum coarse aggregate of 25 mm, percentage of sand in the aggregate of 50%). Although the different slump-loss would indicate that the cement hydration would be retarded by the CAE copolymer more strongly than by SMF or SNF, the compressive strength of the CAE treated concrete appears to be slightly higher than that of SMF or SNF concrete mixtures at early ages and even more at later ages (Fig. 6).

Similar results have been obtained for concrete mixtures produced and cured in cold weather (Fig. 7 and 8) as well as for concretes subject to steam curing (Fig. 9).

The X-ray diffraction (XRD) analysis (Fig. 10) of portland cement mixtures ( $w/c = 0.40$ ) indicates that ettringite ( $\text{C}_3\text{A} \cdot 3\text{CaSO}_4 \cdot 32\text{H}_2\text{O}$ ) whose production is responsible for an appropriate regulation of the setting time in portland cement mixtures, is much more promptly formed during the first hours in the presence of CAE copolymer, and could explain why the slump-loss is reduced in the concrete mixture containing this admixture (Fig. 5 and 7). On the other hand, the slower formation of  $\text{Ca}(\text{OH})_2$  at early ages could mean a slightly lower  $\text{C}_3\text{S}$  hydration:

however this small retarding effect does not affect the concrete compressive strength neither at later nor at earlier ages independently of the curing temperature (Fig. 6, 8 and 9).

Other laboratory tests on the concrete technology including shrinkage, creep and durability [8] - not shown in the present paper for brevity reasons - indicate that all the performances of hardened concrete with CAE copolymer and SMF or SNF polymers are substantially the same, provided that the concrete mixture compositions (and in particular  $w/c$  and aggregate/cement) are the same. This is in agreement with the scanning electron microscopy (SEM) analysis which demonstrated that the morphology of hydrated cement particles is not changed when SMF or SNF superplasticizers are replaced by the CAE polymer in a given cement system [9].

### FIELD TESTS ON CONCRETE TRANSPORTATION

Concrete batches ( $5 \text{ m}^3$ ) with the same mixture proportions as those adopted for the laboratory tests, with CAE copolymer (0.30%), SNF or SMF polymers (0.40%), have been manufactured in a batching plant of ready mixture concrete company and then transported by truck-mixer for at least 3.5 hours at different temperatures.

In the present paper only the field test results of slump-loss (Fig. 11) and compressive strength (Fig. 12) at a temperature of  $21^\circ\text{C}$  will be discussed in order to compare them with the results of the corresponding mixtures (Fig. 5 and 6) examined in laboratory at approximately the same temperature ( $20^\circ\text{C}$ ). In order to make this comparison less favourable for the CAE copolymer, a delayed addition of superplasticizers has been adopted even for the field tests, since this way of addition favours the SNF or SMF performances rather than those of CAE copolymer (Table 4).

The slump-loss behaviour of a concrete mixture on a small scale (about  $0.03 \text{ m}^3$ ), as that adopted for the laboratory tests (Fig. 5), is generally much more severe than that recorded for the same mixture proportion on a large scale when concrete is transported by a truck-mixer. This assumption is confirmed by the comparison of the laboratory results (Fig. 5) and field tests (Fig. 11). Moreover, the slump-loss curves of Fig. 11 indicate that by using the CAE superplasticizer (0.30%) a zero slump-loss concrete mixture can be produced for at least 2 hours of transportation, whereas in the presence of SNF or SMF (0.40%) the slump-loss is more than 100 mm after 1 hour and about 200 mm after 2 hours. Different dosages of CAE superplasticizer - in the range of 0.2 to 0.4% - should be adjusted to obtain similar slump-loss performances at different temperatures - in the range of 5 to  $40^\circ\text{C}$  - and with cements of different reactivity.

Figure 12 shows that compressive strengths of concrete with CAE appear to be higher than those of SNF or SMF superplasticized concrete, because of the lower  $w/c$  (0.43 versus 0.47) at equal initial slump (230 mm). The difference in the compressive strength is smaller at early ages probably because the early  $\text{C}_3\text{S}$  hydration is slightly retarded more by the CAE copolymer than by the SNF or SMF polymers (Fig. 10).

Compressive strength values of the ready-mixed concrete specimens (Fig. 12) appear to be lower than those of the corresponding mixes manufactured in laboratory particularly at later



ages (Fig. 6). Although a reasonable explanation of this discrepancy has not yet been found, it is confirmed that the CAE copolymer performs better than the SNF or SMF polymers as far as the concrete strength is concerned even with a lower dosage (0.30% versus 0.40) and initial equal slump.

## CONCLUSIONS

A new superplasticizer based on the CAE copolymer has been developed and studied in comparison with other polymers (SMF or SNF) usually utilized to manufacture superplasticizers.

The CAE copolymer performs better than the SMF or SNF polymers even with a lower dosage (0.30% versus 0.40% as dry product by weight of cement) because it reduces significantly the slump-loss, which can be completely avoided during the transportation from the batching plant to the work site for at least 2 hours, provided that the proper dosage is adjusted (0.2 - 0.4%) as a function of the cement reactivity and environment temperature.

This is very important from a practical point of view because the practice of redosing the concrete with water above and beyond that required in the mixture design can be avoided. Therefore, the properties of the hardened concrete which depend on the  $w/c$  (strength, durability, abrasion resistance, etc.) are not lowered as those of retempered concretes.

The superplasticizing action of the CAE polymer does not appear to be dependent on the electrostatic repulsion of cement particles as it occurs for the SMF and SNF polymer. The adsorption of the CAE copolymer on the surface of cement particles seems by itself to be responsible for the dispersion of the cement grains and then for the fluidizing effect.

The slump-loss behaviour of the CAE polymer seems to be related to a more promptly ettringite formation and then to a more effective set regulation of the cement mixture.

In the presence of CAE superplasticizer higher strengths can be obtained at equal initial slump level for concretes cured at different temperatures: from cold weather curing (5°C) to steam-curing. If the comparison is done at the time of placing, the CAE superplasticized concrete appears to be more flowable (for the lower slump-loss) and stronger (for the lower  $w/c$ ) than the corresponding concrete mixtures with the SMF or SNF based admixtures.

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**Table 1 - Physical Properties and Chemical Analysis of Cement**

	Portland Cement*
<b>Physical Tests</b>	
Fineness - Passing 45 $\mu\text{m}$ (%)	87
- Blaine ( $\text{m}^2/\text{kg}$ )	380
Specific Gravity ( $\text{kg/l}$ )	3.15
<b>Chemical Analysis (%)</b>	
SiO <sub>2</sub>	21.23
Al <sub>2</sub> O <sub>3</sub>	6.89
Fe <sub>2</sub> O <sub>3</sub>	5.94
CaO	60.83
MgO	2.16
Na <sub>2</sub> O	0.10
K <sub>2</sub> O	0.63
SO <sub>3</sub>	1.62
Loss on ignition	0.60

\* Compressive strength (MPa) mortar specimens (w/c = 0.50): 3 day = 32 MPa; 7 day = 44 MPa; 28 day = 50 MPa.

**Table 2 - Composition of Normal Portland Cement Concrete Mixtures Used to Study the Effect of the Way of Addition of Superplasticizers on the Concrete Workability**

INGREDIENT:	BATCH QUANTITIES ( $\text{kg/m}^3$ )		
	Portland Cement	410	410
Water	162	168	164
Sand (F.M. = 2.7)	747	752	750
Gravel (19 mm max)	1121	1128	1125
Superplasticizer:			
- CAE	4.15	-	-
- SNF	-	4.90	-
- SMF	-	-	4.90
w/c	0.39	0.41	0.40

**Table 3 - Composition of Normal Portland Cement Concrete Mixtures for Laboratory and Field Tests on Slump-loss and Compressive Strength**

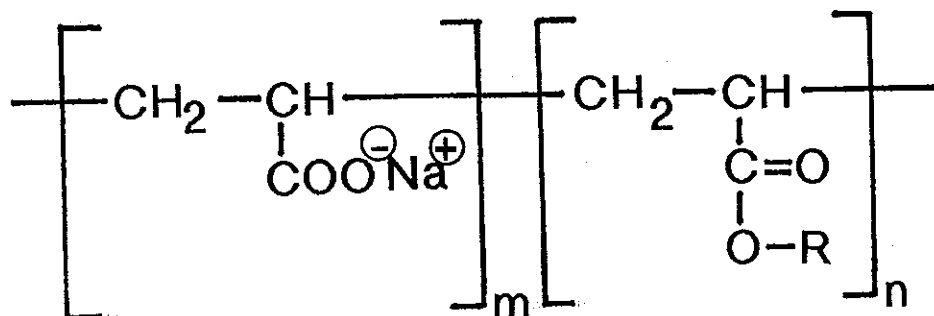
INGREDIENT:	BATCH QUANTITIES (kg/m <sup>3</sup> )		
	Portland Cement	352	355
Water	150	166	168
Sand (F.M. = 2.7)	964	952	950
Gravel (25 mm max)	965	946	940
Superplasticizer			
- CAE	3.52	-	-
- SNF	-	3.55	-
- SMF	-	-	3.55
w/c	0.43	0.47	0.47

**Table 4 - Effect of way of addition of CAE, SMF and SNF superplasticizers on the slump of portland cement concrete mixtures**

ADMIXTURE			w/c	Slump (mm)
Type	Dosage (%)	Way of Addition*		
CAE	0.30	Immediate	0.39	230
CAE	0.30	Delayed	0.39	235
SMF	0.50	Immediate	0.41	100
SMF	0.50	Delayed	0.41	215
SNF	0.48	Immediate	0.40	100
SNF	0.48	Delayed	0.40	230

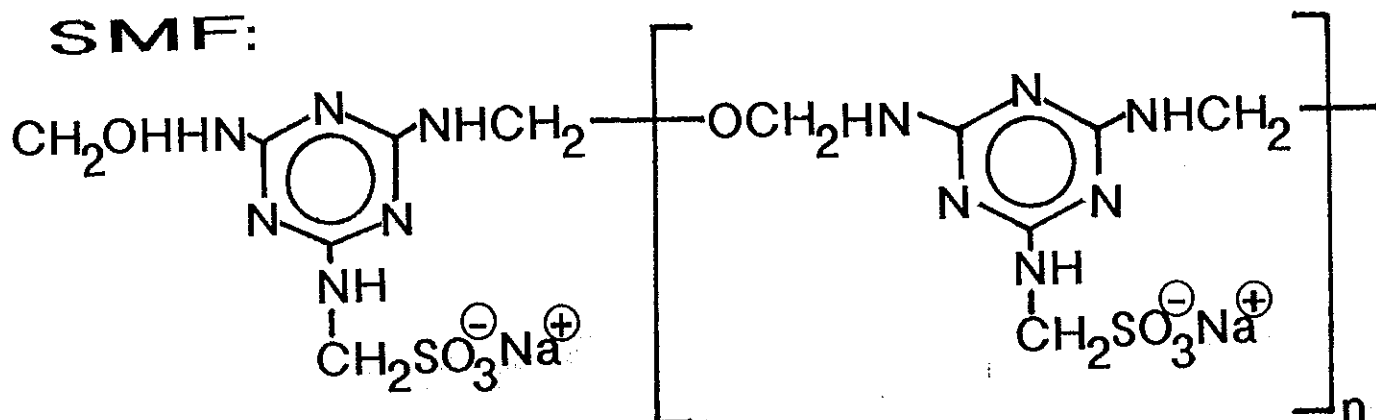
\* Immediate: admixture with mixing water. Delayed: admixture after 1 min of mixing.

**CAE:**



R = organic radical  
of ester group

**SMF:**



**SNF:**

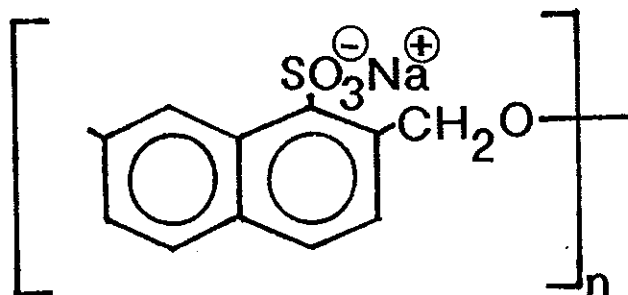


Fig. 1. Chemical composition of CAE copolymer and SMF or SNF polymers

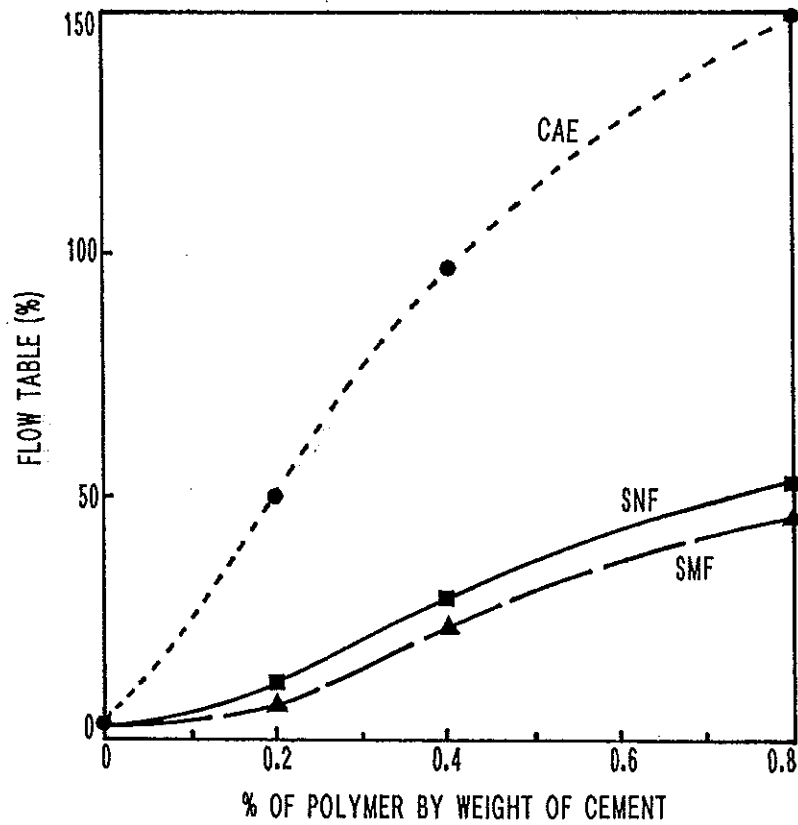


Fig. 2. Influence of CAE, SMF or SNF polymers on the fluidity of portland cement mortar mixtures ( $w/c = 0.40$ )

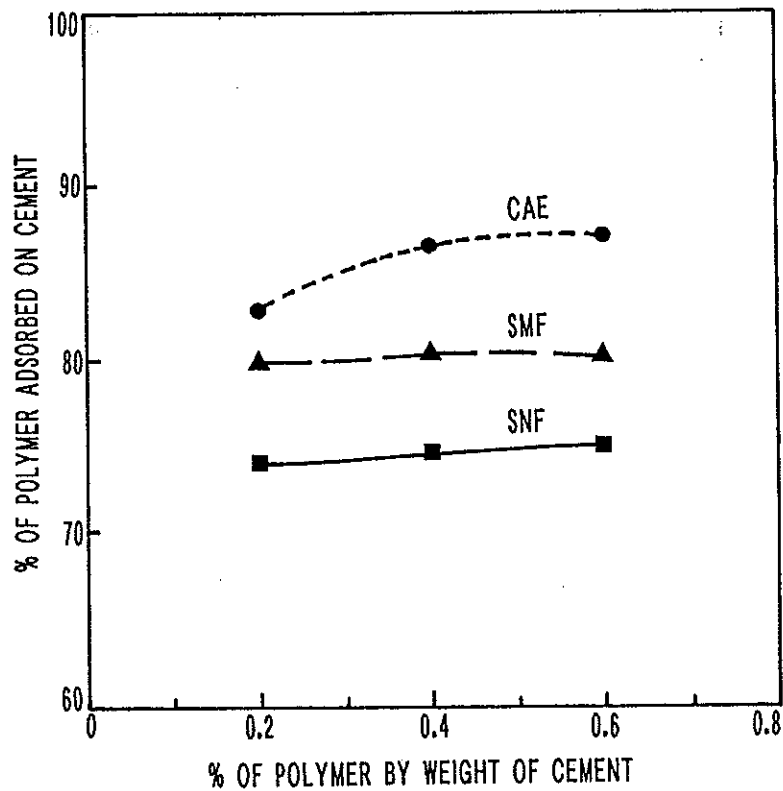


Fig. 3. Adsorption of CAE, SMF and SNF on cement as a function of polymer dosage by weight of cement

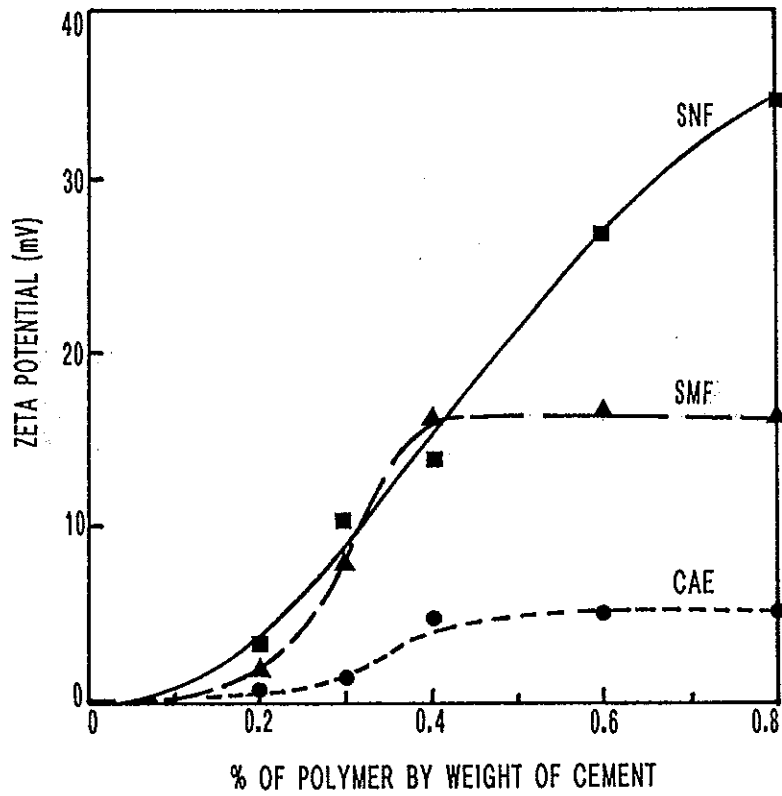


Fig. 4. Zeta potential of CAE, SMF and SNF cement pastes as a function of polymer dosage by weight of cement

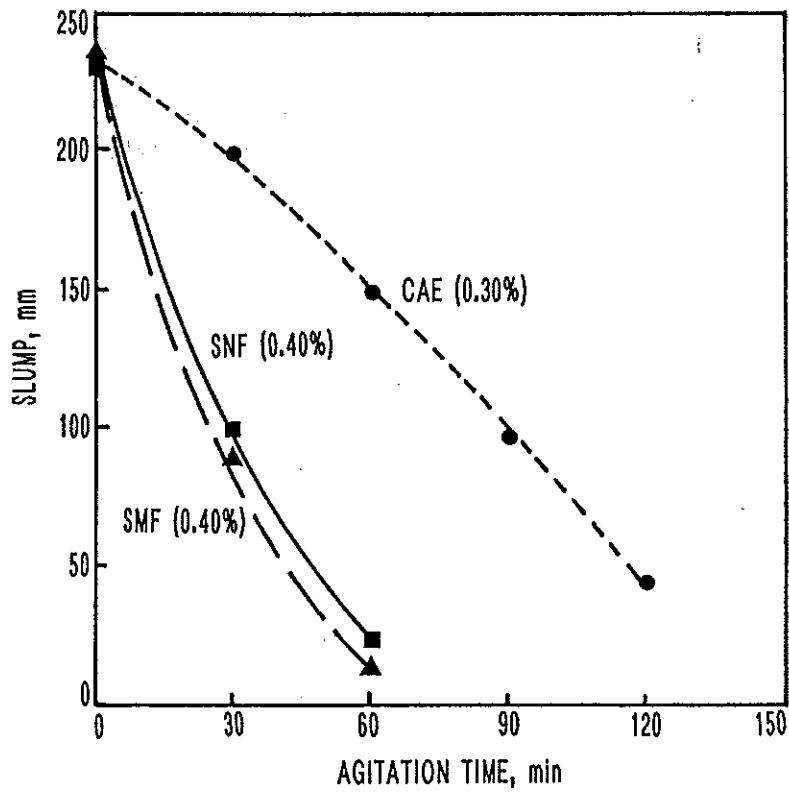


Fig. 5. Slump-loss of superplasticized concrete mixtures at 20°C with CAE, SMF or SNF polymers (laboratory tests)

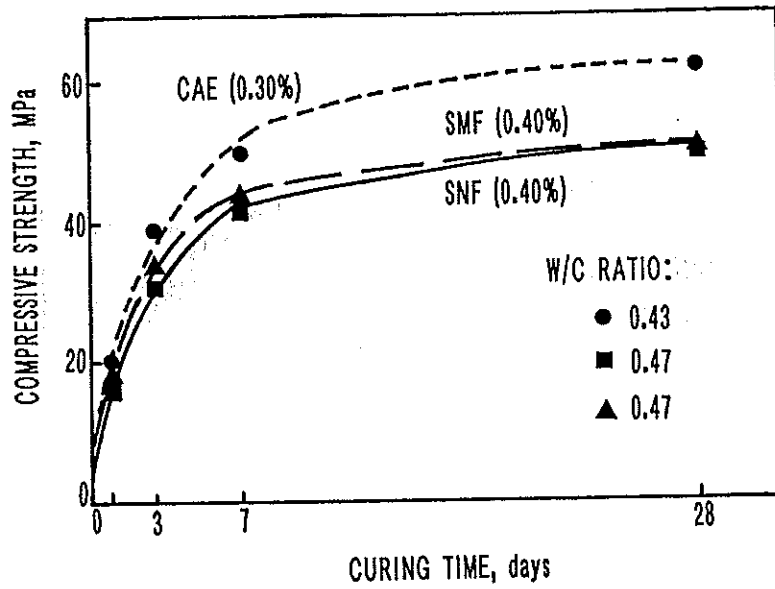


Fig. 6. Compressive strength of superplasticized concrete mixtures at 20°C with CAE, SMF or SNF polymers (laboratory tests)

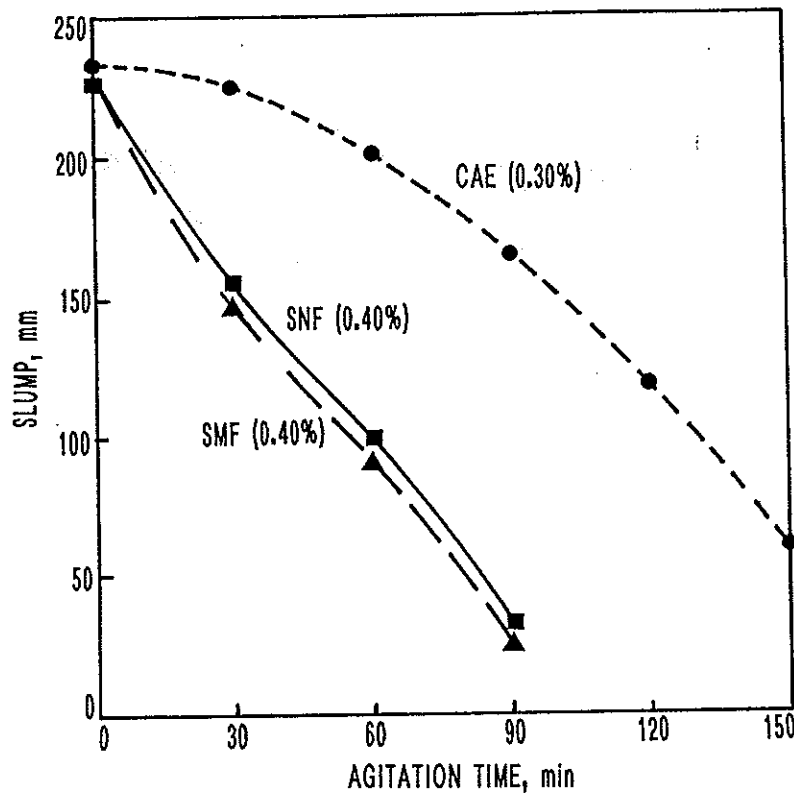


Fig. 7. Slump-loss of superplasticized concrete mixtures at 5°C with CAE, SMF or SNF polymers (laboratory tests)



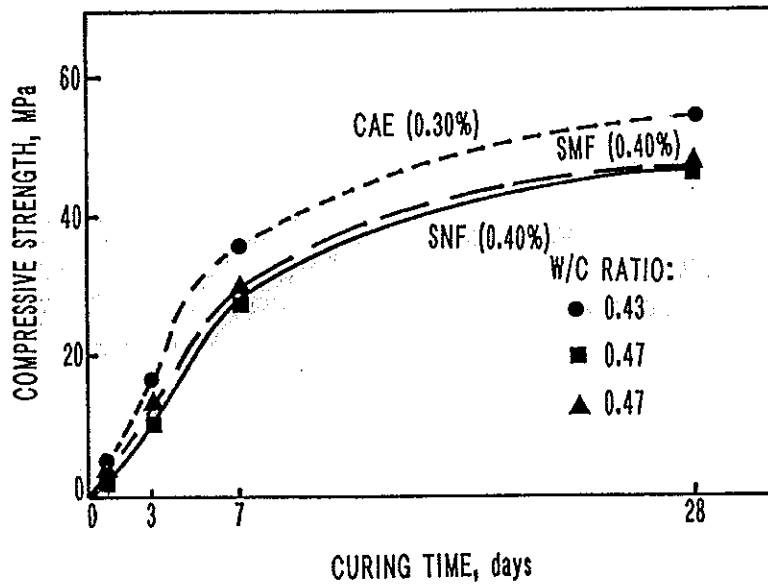


Fig. 8. Compressive strength of superplasticized concrete mixtures at 5°C with CAE, SMF or SNF polymers (laboratory tests)

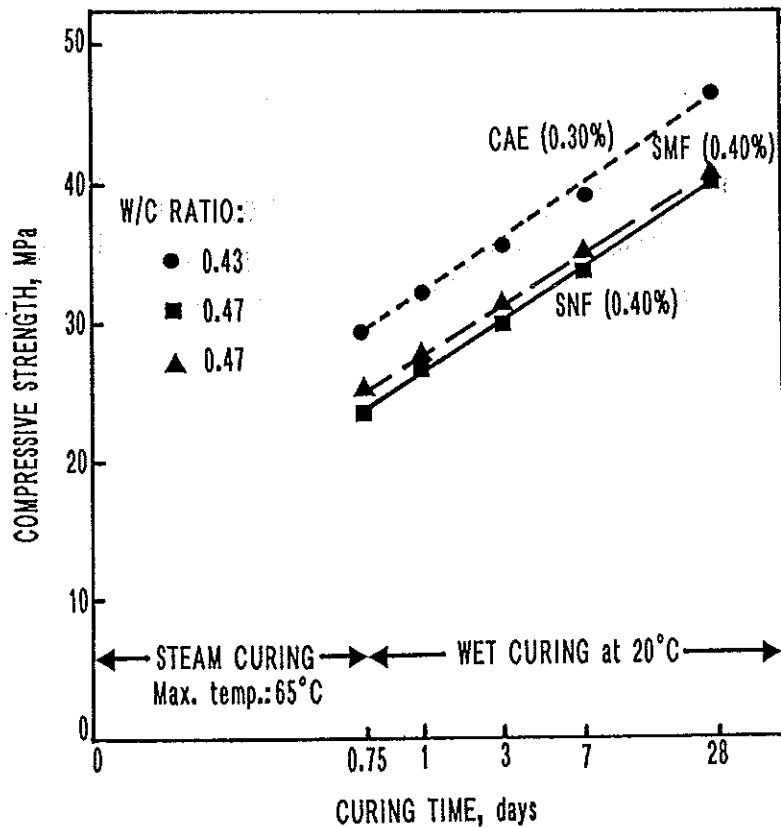


Fig. 9. Compressive strength of steam-cured superplasticized concretes with CAE, SMF or SNF polymers (laboratory tests)

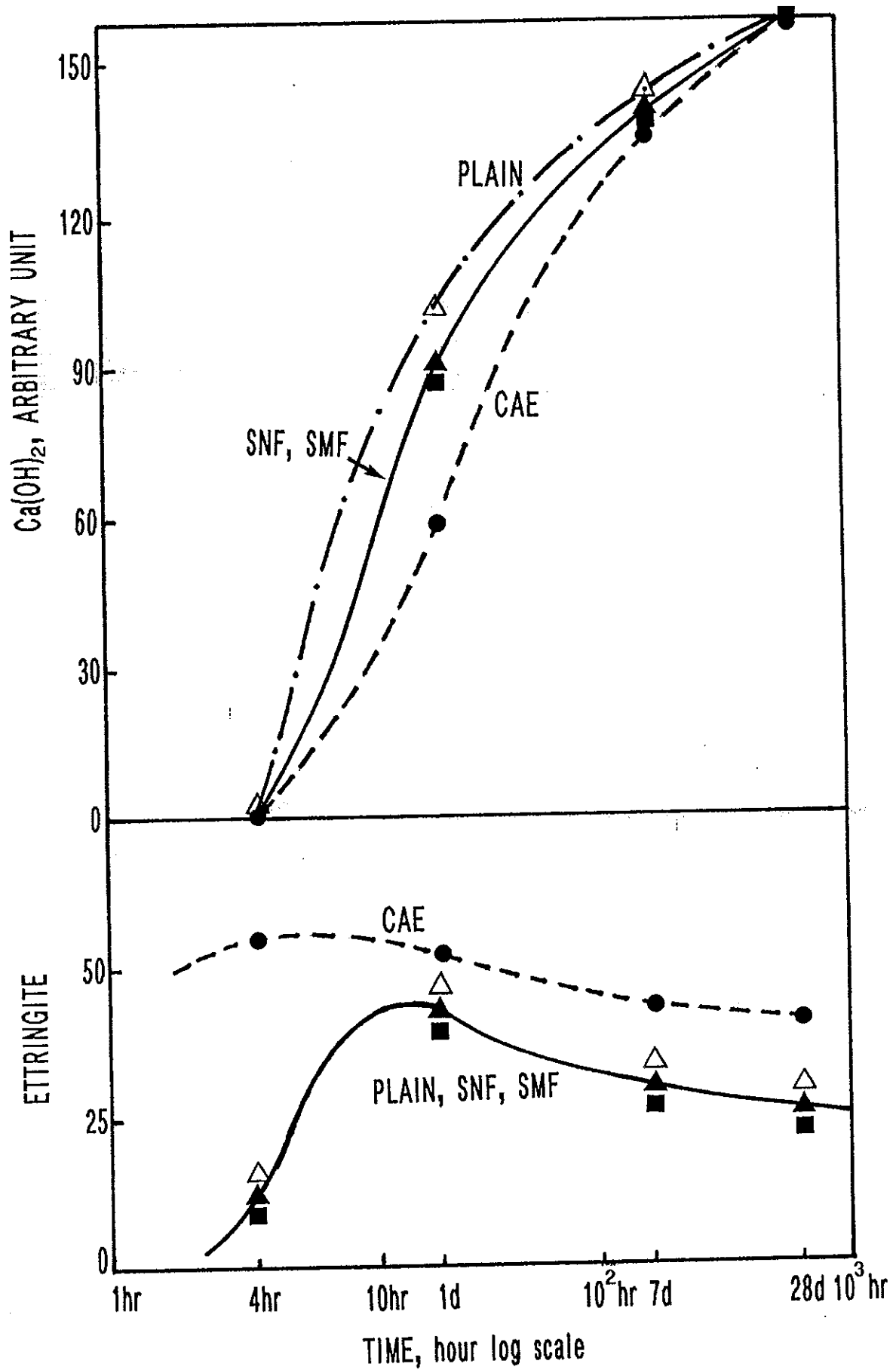


Fig. 10. XRD semiquantitative results for ettringite and  $\text{Ca(OH)}_2$  production in portland cement pastes with or without CAE, SMF or SNF

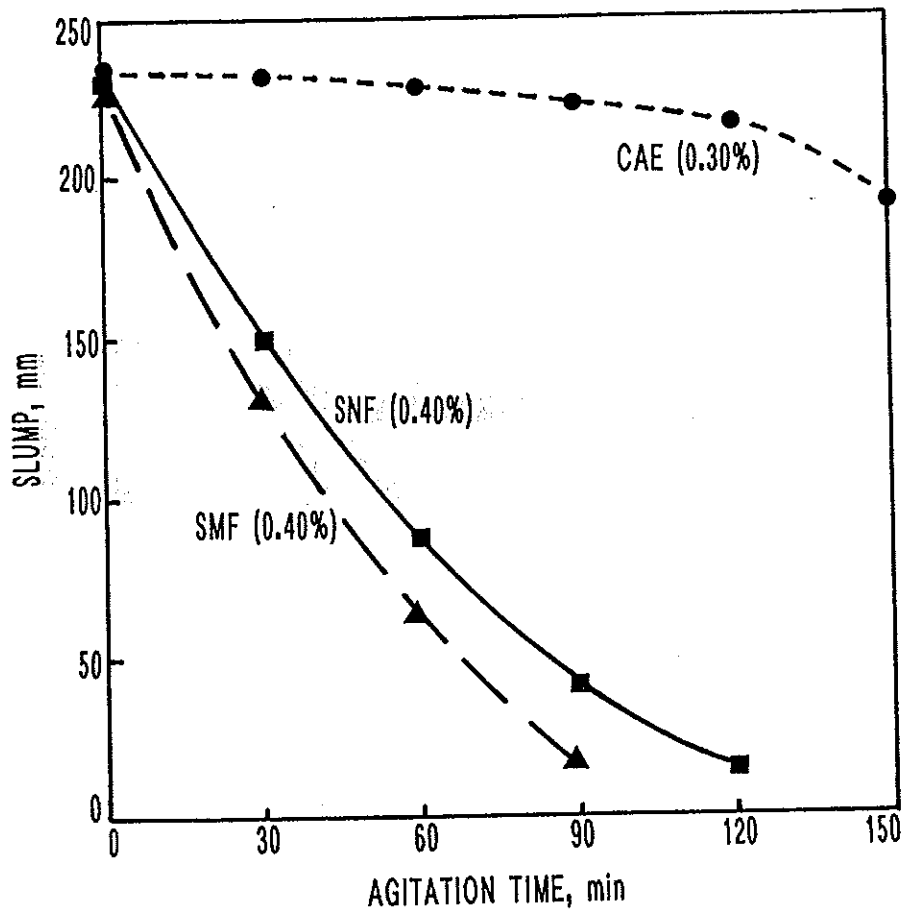


Fig. 11. Slump-loss of ready-mixed concretes with CAE, SNF or SMF superplasticizers at 21°C (field tests)

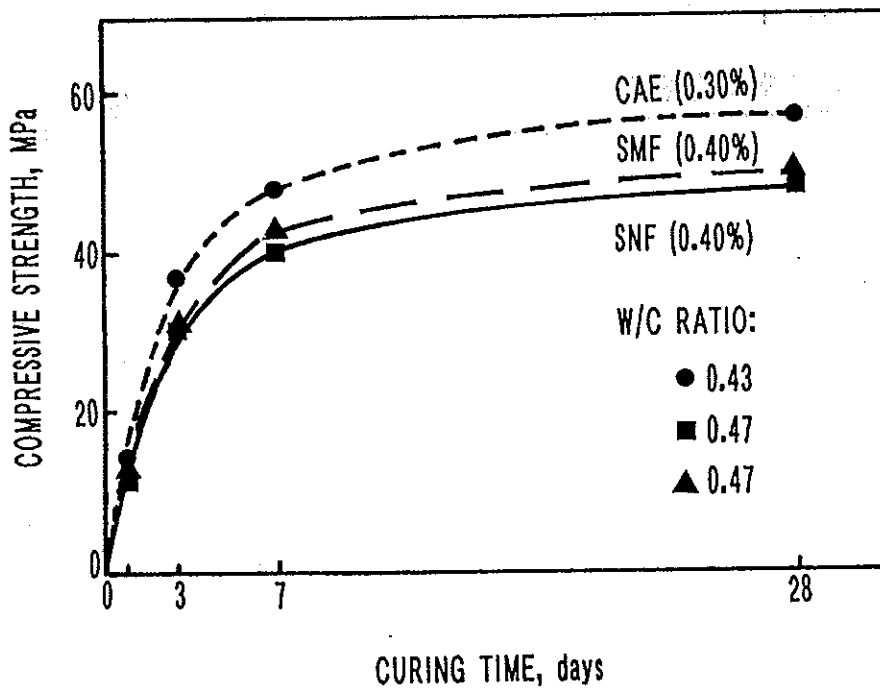


Fig. 12. Compressive strength of ready-mixed concretes with CAE, SNF or SMF superplasticizers cured at 21°C