

## CHEMICAL SHRINKAGE OF CEMENT PASTES WITH PLASTICIZING ADMIXTURES



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### Abstract

Total and external chemical shrinkage have been followed for a number of cement pastes until 48 h. Total chemical shrinkage is believed to roughly be proportional to the degree of hydration, while the differences in external chemical shrinkage give an impression on how prone the mixture may be to micro-cracking. The difference between total and external shrinkage results in contraction pores. The study is part of a larger on-going study focusing on the early volume change of binders of high performance concrete.

Variables in the present part of the study have been 5 cement types, plasticizer (sodium lignosulphonate denoted LS) and 2 super-plasticizer types (sodium salts of sulphonated melamine - formaldehyde condensate named SMF and naphthalene sulphonate - formaldehyde condensate called SNF) admixture dosage (0.0, 0.7, 1.0 and 1.4 %) and water-to-cement ratio (0.30 and 0.40). However, only 25 of the 100 possible combinations were made.

Total chemical shrinkage was used to monitor the retardation of cement hydration by e.g. LS and the acceleration of hydration rate thereafter. LS retard early hydration (until 10 h) more than the super-plasticizers SNF and SMF, which accelerated hydration for some cements for the whole period of measurement. LS often accelerated hydration from 24 to 48 h, probably due to improved dispersion of cement grains. The flattening-out-level of the external chemical shrinkage was independent of admixture type, and the same as the reference without admixtures, when the samples were rotated (undisturbed by "bleeding").

The admixtures did not directly influence the chemical shrinkage in a way that should indicate a higher risk of cracking. However, the retardation of early hydration and setting of cement by lignosulphonate could allow a longer period of evaporation of water from floors and bridge decks in practice. For this reason mixes with lignosulphonate might be more prone to form drying shrinkage cracks.

**Keywords:** Cement paste, Chemical shrinkage, plasticizers, super-plasticizers, and w/c.

## 1. INTRODUCTION

Practical experience has shown that high performance concrete is sensitive to cracking at early ages (from placing to a few hours after finishing), even when great care is taken to avoid evaporation from the surface that initiates plastic shrinkage /1, 2/. The main reason for this early shrinkage is considered to be early volume change producing stresses under restraint conditions, coupled with low stress/strain capacity of the concrete at this stage. The present study is part of a larger work. Earlier papers have been focusing on the influence of cement type /3/, water-to-cement ratio /4/ and mineral additives /5, 6, 7/ on chemical shrinkage. The influence of the last component, the plasticizers, is treated at present and comparison is done for a number of Portland cements. A second paper on the effect of plasticizers at different w/c on chemical shrinkage is recently published /8/.

The total chemical shrinkage during the hydration of cement is caused by the smaller volume of products (e.g. calcium silicate hydrate gel and calcium hydroxide) compared with the reactants (e.g. alite and water). As a rule of thumb, the total chemical shrinkage at 100 % hydration is about 6.25 ml/100 g cement (i.e. 25 % of the chemical bound water to cement ratio of 0.25) /9/. The total chemical shrinkage equals the external chemical shrinkage until the net-work of hydration products bridging the unreacted cement grains is strong enough to resist the contracting forces. At this point (5-9 h depending on cement composition, fineness, w/c etc.), the external chemical shrinkage rate slows down drastically and the shrinkage vs. time curve flattens out. Thereafter, the second manifestation of total chemical shrinkage; the formation of internal contraction pores, is dominating. The relationship between the two is considered important in the cracking problem. Both quantities are measured in parallel in the present work.

The terminology in the present paper is total chemical shrinkage, which is the sum of external chemical shrinkage and the volume of empty contraction pores at all stages. In literature, there is a terminology confusion: Total chemical shrinkage may be named *chemical shrinkage*, *water absorption*, *volume contraction*, or *Le Chatelier shrinkage* after the first scientist who examined the shrinkage of cement paste. External chemical shrinkage is also named *external volume change*, *bulk shrinkage* and *autogeneous shrinkage*.

The methods for measuring total chemical shrinkage all have in common that the sample has to be kept water saturated and that the water needed to replace the volume decrease is measured, while the common methods for measurement of external chemical shrinkage are characterised by sealed curing of the cement paste. There are mainly three techniques for measuring chemical shrinkage; 1) dilatometry, 2) gravimetry and 3) pycnometry.

Dilatometry is based on direct measurements of length or volume change.

The most common dilatometry technique for total chemical shrinkage measurement is to put the paste in a recipient (e.g. glass tube), filling the rest of the recipient with water and plugging the recipient with a stopper with a water filled pipette stuck through. Reading the fall of the water level in the pipette versus time gives the total volume change. Knudsen and Geiker /10/ have pioneered a variety of applications for this method since the 1980's. This method is used in the present paper.

A dilatometry technique for external shrinkage measurement has been utilised by measuring the uniaxial length change of a flexible, sealed tube by inductive sensors /11/.

Gravimetry is based on indirect measurement of volume change by recording reduced buoyancy under water by weighing (i.e. the law of Archimedes). If the weight change of a container filled with paste and excess water having at least one flexible wall is recorded, the total chemical shrinkage is measured /12/. If the weight change of a sealed elastic bag filled with paste only is registered, the external chemical shrinkage is measured. The latter method is used in the present paper. Note that the samples for measurement of external chemical shrinkage should be continuously rotated in order to avoid artificially high values caused by imbibition of "bleeding" water /13/.

Pycnometry is only applicable for total chemical shrinkage measurements, and is carried out by filling a pycnometer with paste and topping it with water. Water is added to refill the pycnometer at different ages, and the weight increase relates to the total volume change.

## **2. EXPERIMENTAL**

### **2.1 Chemicals**

The cements were all Portland cements produced by Norcem A/S, Norway, over a period of time. The cement characteristics are given in Table 1.

P30 = Standard Portland cement produced at the Dalen plant until 1993.

P30c = Standard Portland cement produced at the Dalen plant after 1993.

P30k = Standard Portland cement produced at the Kjølpsvik plant.

SR = Sulphate Resistant Portland cement produced at the Dalen plant.

G = API Class G Oil well Cement produced at the Dalen Plant.

The mixing water was distilled before use.

The plasticizer sodium lignosulphonate (LS) was of the type Borresperse Na from Borregaard, Sarpsborg, Norway, a solution of 40% solids with some minor antifoaming agent.

The super-plasticizer sodium naphthalene sulphonate - formaldehyde condensate (SNF) was of the type Mighty 150 produced by Kao, Japan, a solution with 42 % solids.

The super-plasticizer sodium salt of sulphonated melamine - formaldehyde condensate was of the type Melment from SKW, Germany, a solution with 40 % solids.

## 2.2 Methods

### 2.2.1 Mixing procedure

Cement and water was mixed in a Hobart mixer of 5 litre capacity. Mixing times were 2 min at gear 1 and 1 min at gear 2. The bowl with paste was put on a vibrating table to remove most of the entrained air. The starting time of the experiments was the first contact between cement and water (time = 0), and the first measurements started at 1 h.

Table 1 Cement characteristics

Cement	P30	P30c	P30k	SR	G
<u>Oxides (%)</u> :					
CaO (C)	63.16	63.44	63.61	64.62	64.82
SiO <sub>2</sub> (S)	20.28	20.92	20.84	21.98	22.0
Al <sub>2</sub> O <sub>3</sub> (A)	4.89	4.60	5.04	3.48	3.53
Fe <sub>2</sub> O <sub>3</sub> (F)	3.61	3.54	3.30	4.88	4.78
MgO (M)	2.21	1.80	-	1.45	1.42
SO <sub>3</sub> (')	2.98	3.06	2.70	2.18	1.75
Alkalis	1.13	0.94	1.06	0.69	0.60
Blaine (m <sup>2</sup> /kg)	309	358	361	290	303
<u>Minerals (%)</u> (Bogue)					
C <sub>3</sub> S	57	55	54	60	61
C <sub>2</sub> S	16	19	19	18	17
C <sub>3</sub> A	6.9	6.2	7.8	1.0	1.3
C <sub>4</sub> AF	11.0	10.8	10.0	14.9	14.6
C'	5.1	5.2	4.6	3.7	3.0

### 2.2.2 Total chemical shrinkage

Cement paste was put into three Erlenmeyer flasks for parallel experiments. The weights of the empty and filled recipients were measured in order to determine the amount of paste. The recipients were then carefully filled with distilled water at room temperature in a manner to avoid turbulence. A silicon rubber stopper was used to plug each recipient, taking care not to enclose any air bubbles. A pipette was filled with water and stuck through a hole in the stopper. A graded pipette of 0.2, 0.5 or 1 ml was chosen depending on the expected volume change. The recipients were put in a water bath at 20±1°C. Every hour until 48 h, the position of the meniscus in the pipettes was read. The decrease of the water column in ml is directly the total chemical shrinkage (as a drop of liquid paraffin on top prevented evaporation), which were expressed as ml/100 g cement after a calculation of the mean value from the three parallel measurements with a typical standard deviation of ±2%. The method relies on the assumption that all contraction pores are filled with water. A photo of the experimental set-up is reproduced in Fig. 1.

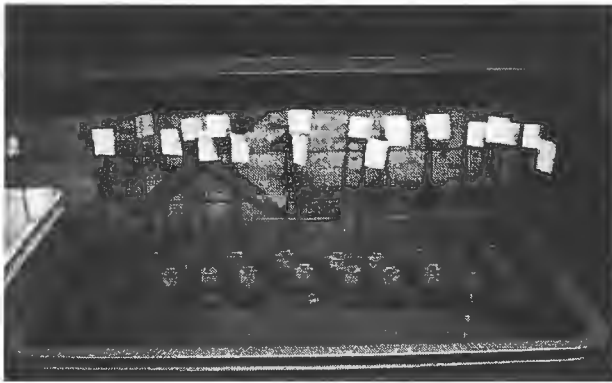


Fig. 1. Picture showing experimental set-up for measuring total chemical shrinkage. Erlenmeyer flasks are filled with cement paste and excess water. The pipette on top is to read the total chemical shrinkage. The flasks are placed in a tray with water in a temperature controlled room or alternatively in a temperature regulated water bath.

### 2.2.3 External chemical shrinkage

Three elastic rubber bags (i.e. condoms) were filled with cement slurry while the condoms were inserted in a 100 mm plastic tube of 50 mm inner diameter. Each condom was closed by twisting the upper part and tying it with a thin copper wire, and sealed by spraying silicon glue into the open end. The excess end part was cut off and the total mass determined.

The filled and sealed condoms in their tubes were kept in a water bath of  $20 \pm 1^\circ\text{C}$  after the tubes had been turned perpendicularly to their axes in order to let all air bubbles escape. The tubes were kept on an ordinary rotating table modified to function under water (i.e. a chain transfer between the motor and the rollers placed under water). During the first 10 h, the condom was weighed every hour under water. According to Archimedes principle, an external shrinkage will lead to a reduction in buoyancy, which will be registered as a weight increase. Each condom was weighed in a basket under water hanging on a scale in a separate water bath with no stirring to avoid turbulence. The transfer between the bath for rotation and the bath for weighing took place under water at all times by placing the tube in a water filled glass under water in one bath and taking it out of the glass under water in the second bath. This was done to avoid trapping of any air bubbles in the transfer process.

After the last weighing under water at 48 h, the condoms were wiped dry and weighed in air. Finally, the condom, including copper wire and silicon glue, were stripped off and weighed in order to calculate the net weight of the cement slurry after subtracting the weight of the tube. The external shrinkage is presented as the mean value of three parallel measurements and given in ml/100 g cement with a standard deviation of about  $\pm 5\%$ . In the present study, the samples with  $w/c = 0.30$  were not rotated since they were not prone to "bleeding". In such a case the tube in the preceding procedure was excluded and the condoms carefully weighed as such.

Photos of the rotating table with cement filled condoms in tubes under water and the weighing arrangement with a cement filled condom in a tube are reproduced in Fig. 2.

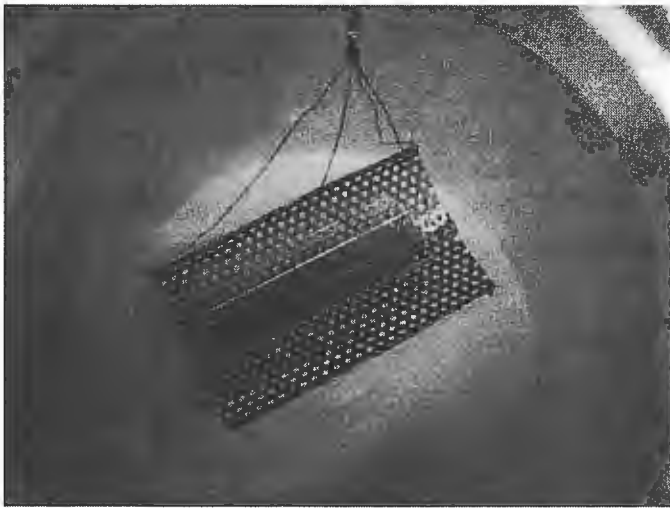
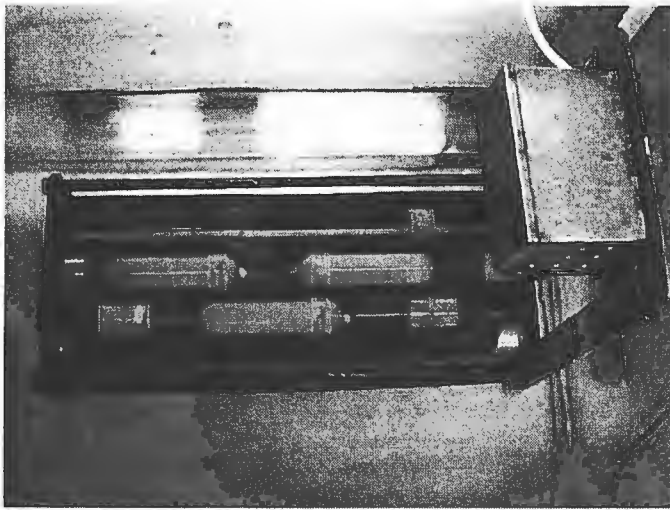


Fig. 2 Upper photo illustrate three parallels of cement filled condoms in tubes on a rotating table under water as described in the procedure for external chemical shrinkage in the preceding text. The lower photo shows one of the cement filled condoms in its tube in the weighing basket under water attached to a scale. The reduced buoyancy as a function of time is proportional to the external volume reduction of the condom according to the principle of Archimedes.

### 3. RESULTS AND DISCUSSION

The total and external chemical shrinkage versus time profiles of P30 cement paste with  $w/c = 0.30$  are shown in Figs. 3 and 4, respectively. The effect of 0.7 % additions of the plasticizer LS (lignosulphonate), super-plasticizer SNF (naphthalene sulphonate – formaldehyde condensate) and super-plasticizer SMF (sulphonated melamine - formaldehyde condensate) are compared with the reference without admixtures. It is evident from the total chemical shrinkage that all admixtures retard the early hydration (4 – 12 h) of the cement in the increasing order  $SMF < SNF < LS$ , assuming that chemical shrinkage approximately is proportional with degree of hydration,  $\alpha$ . From 24 to 48 h (last data point), the degree of hydration is higher than the reference for all admixtures and in fact the highest

for LS. The reason for this effect is probably better dispersion and breaking of possible aggregates of cement grains. The external chemical shrinkage curves in Fig. 4 show the same trend of early hydration retardation as in Fig. 3. The knee-points leading into the flattening out levels (FOL) is quite gradual (6-8 h) for all pastes but the one with LS (rather sharp transition at 9 h). The FOLs are equal for all pastes, except for the paste with 0.7 % LS reaching a significantly higher FOL ( $\approx + 30\%$ ). LS is known to interact strongly with the cement mineral  $C_3A$ , otherwise known to form ettringite in reaction with gypsum. If a more amorphous  $C_3A$ -LS complex is formed on the expense of needle shaped ettringite crystals, it is possible that more hydration is needed to make a strong enough hydration network to resist the contraction forces. This could explain the higher FOL. Since the samples for external chemical shrinkage is not rotated in this study, any tendency of separation (“bleeding”) will lead to a higher FOL when the “bleeding” water is sucked into the sample after contraction pores are formed. However, it is unlikely that LS should lead to more “bleeding” than SNF or SMF for the P30 cement.

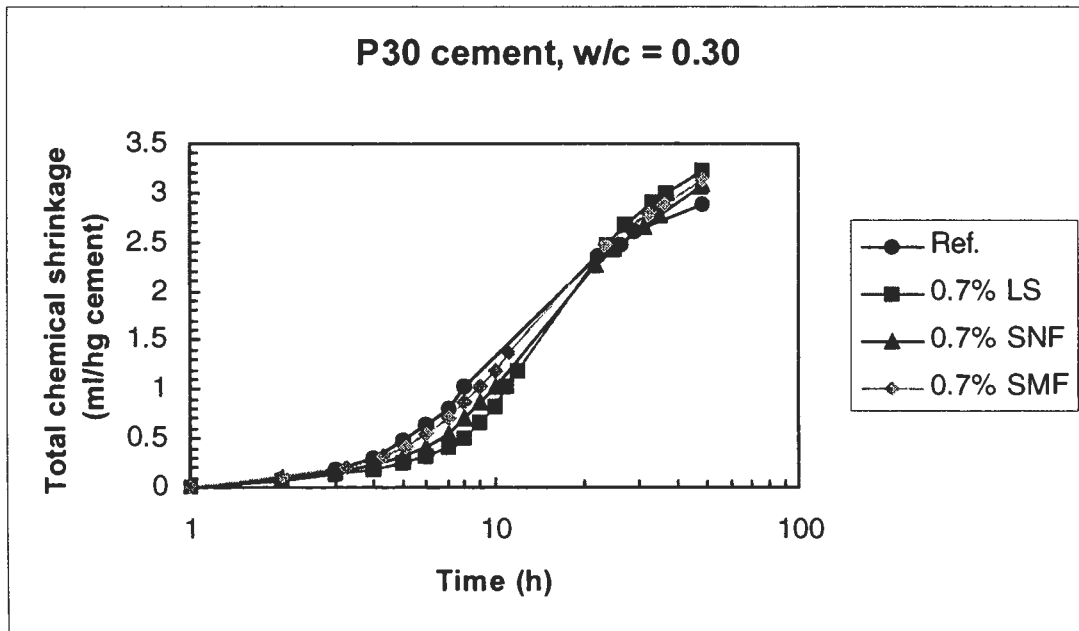


Fig. 3 Total chemical shrinkage versus time curves for P30 cement paste with  $w/c = 0.30$  and 0.7 % LS, SNF and SMF as compared with the reference without admixtures.

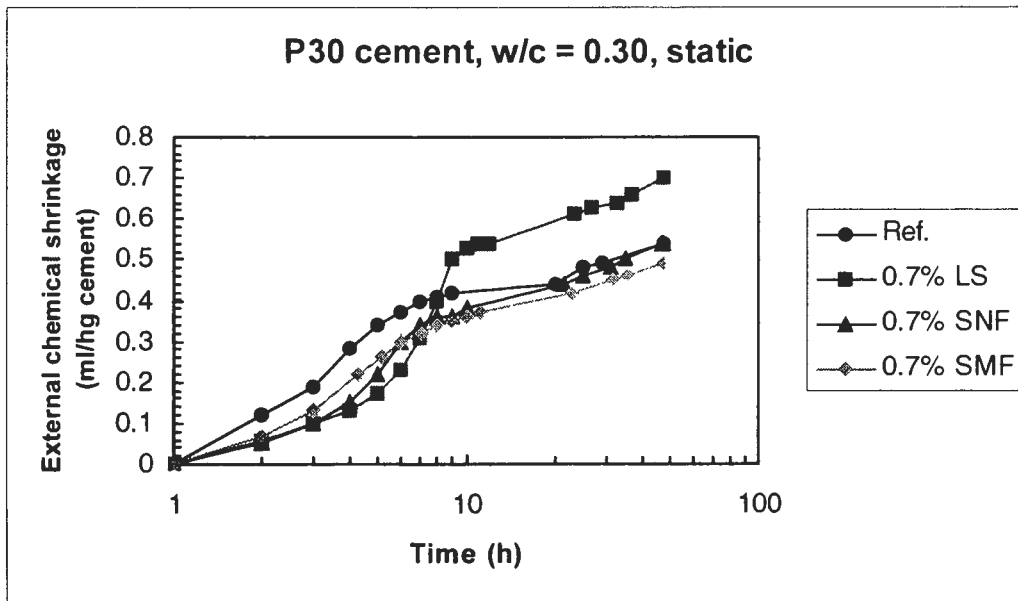


Fig. 4 External chemical shrinkage versus time curves for P30 cement paste with w/c = 0.30 and 0.7 % LS, SNF and SMF as compared with the reference without admixtures. Samples are not rotated.

The total and external shrinkage versus time profiles for SR cement pastes with w/c = 0.30 are plotted in Figs. 5 and 6, respectively, for the different admixtures. Unlike for the P30 cement, only 0.7% LS weakly retard the hydration (as seen by comparing Figs. 3 and 5), while 0.7% SNF or SMF actually accelerates the hydration all the way to 48 h compared with the reference. The degree of hydration for the paste with 0.7% LS is also higher than the reference between 24 and 48 h, which again may be subscribed to a better dispersion of cement grains and breaking of possible agglomerates.

The flattening out levels (FOLs) for the external chemical shrinkage curves for the SR cement pastes in Fig. 6 are very different with an increasing level in the order SNF > SMF > reference > LS. SR cement is more prone to “bleeding” than the P30 cement, even though the surface is only marginally lower (290 versus 309 m<sup>2</sup>/kg), as demonstrated by loss of hydrostatic pressure during separation /1/. This may be due to the low C<sub>3</sub>A content of SR cement and thereby lower water binding by ettringite formation on the surface. The reason why paste with 0.7% LS actually has a lower FOL than the reference paste may be explained by a weak bleeding tendency of the reference that is absent when LS is added since LS causes a gelling (i.e. thixotropy) of the SR paste. This gelling tendency increases with decreasing C<sub>3</sub>A content of different cements /14/.

The total and external chemical shrinkage versus time curves for P30k cement paste with w/c = 0.40 are shown for 0.7 % LS, SNF and SMF in Figs. 7 and 8, respectively, and for 1.0 % LS, SNF and SMF together with 1.4 % SMF in Figs. 9 and 10, respectively. All are compared with the curves for reference paste without admixtures. Note that all the samples in the w/c = 0.40 series are rotated for the external shrinkage measurements.



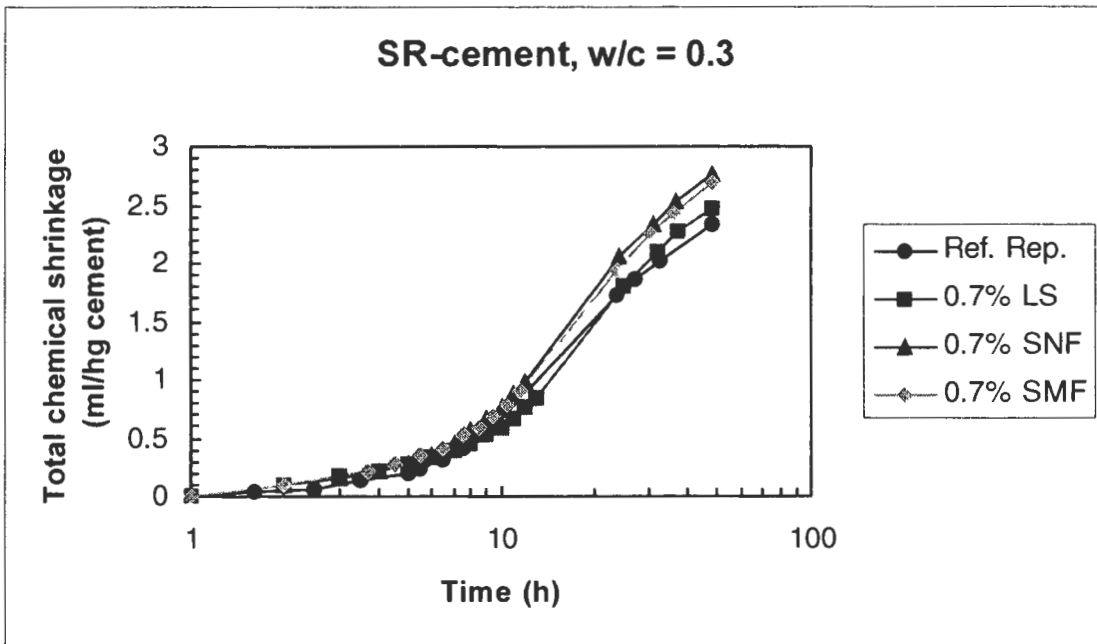


Fig. 5 Total chemical shrinkage versus time curves for SR cement paste with w/c = 0.30 and 0.7 % LS, SNF and SMF as compared with the reference without admixtures.

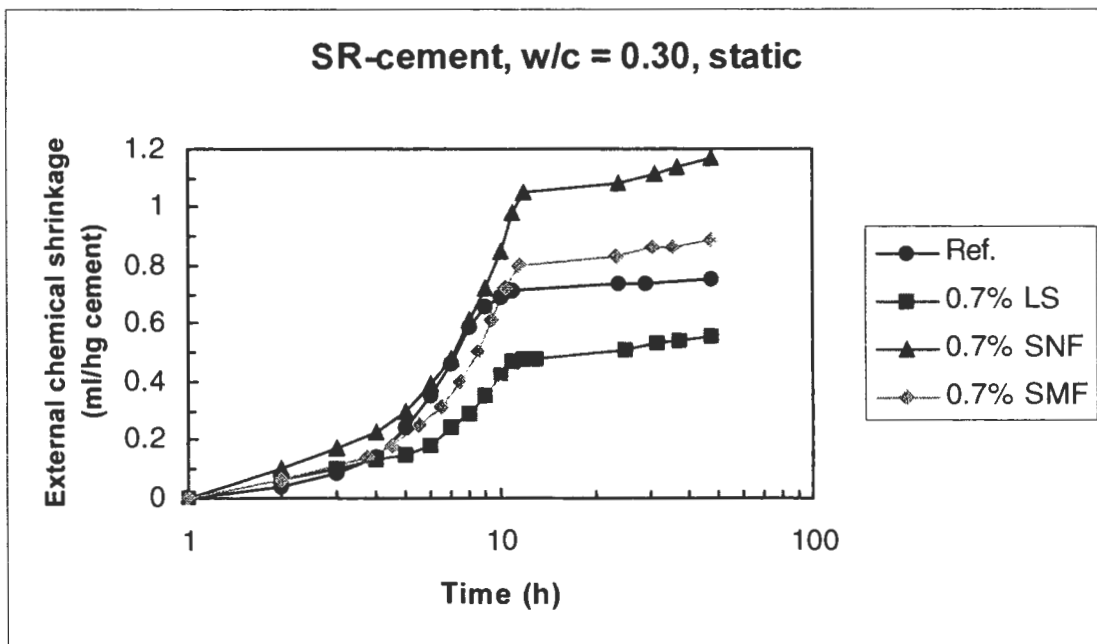


Fig. 6 External chemical shrinkage versus time curves for SR cement paste with w/c = 0.30 and 0.7 % LS, SNF and SMF as compared with the reference without admixtures. Samples not rotated.

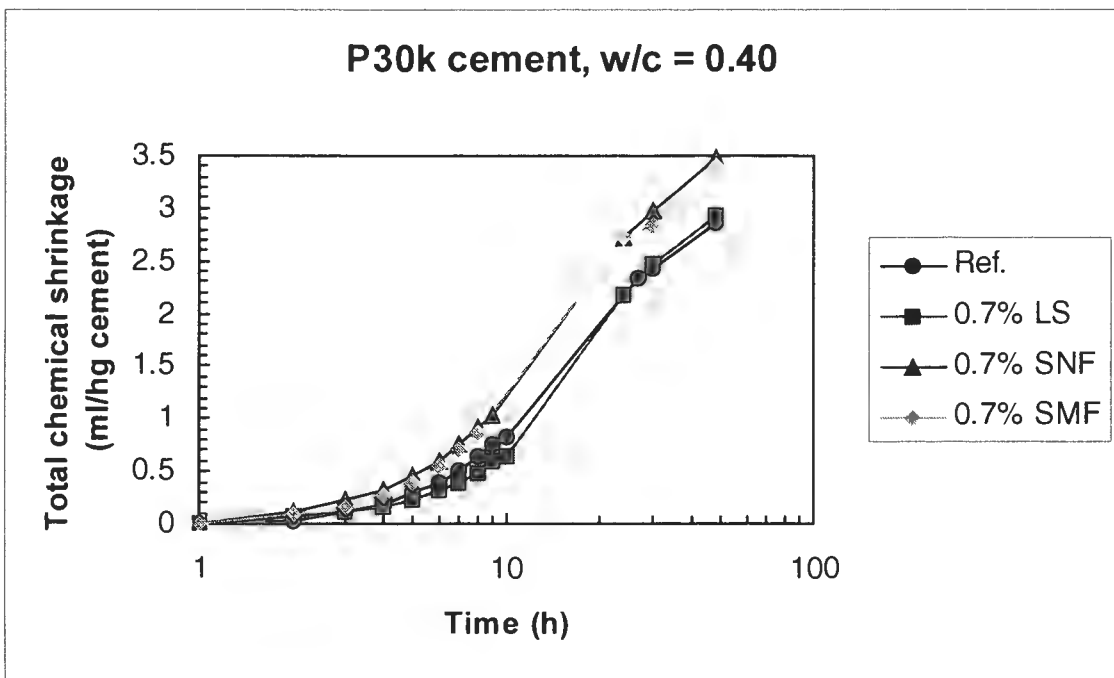


Fig. 7 Total chemical shrinkage versus time curves for P30k cement paste with w/c = 0.40 and 0.7 % LS, SNF and SMF as compared with the reference without admixtures.

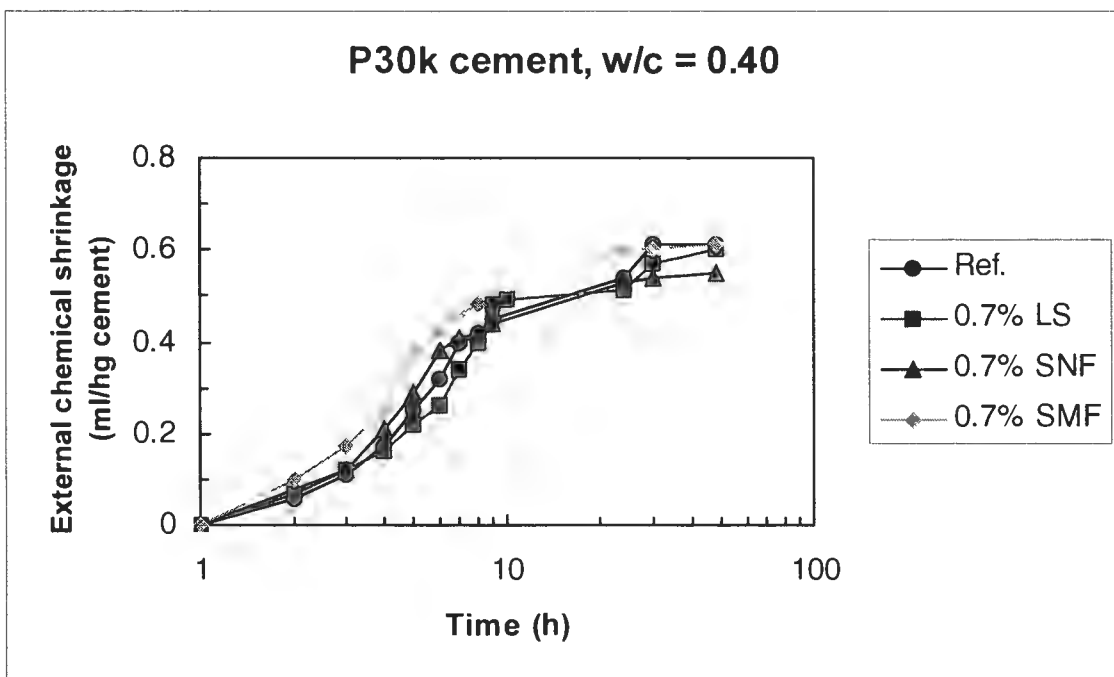


Fig. 8 External chemical shrinkage versus time curves for P30k cement paste with w/c = 0.40 and 0.7 % LS, SNF and SMF as compared with the reference without admixtures.

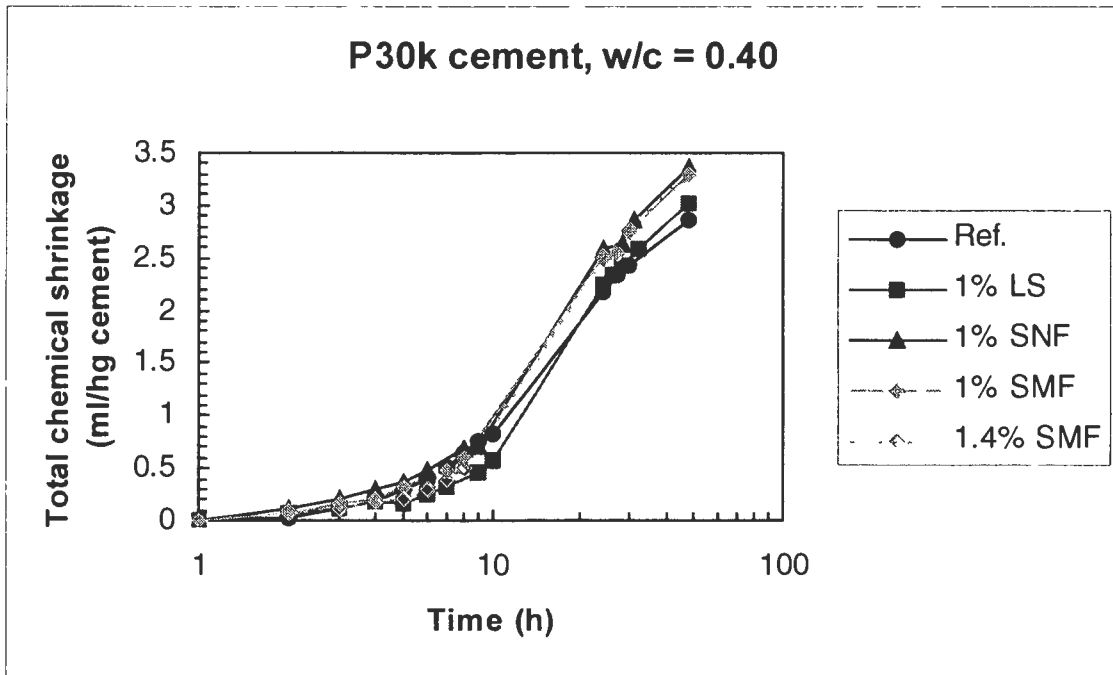


Fig.9 Total chemical shrinkage versus time curves for P30k cement paste with w/c = 0.40 and 1.0 % LS, SNF and SMF, and 1.4 % SMF, as compared with the reference without admixtures.

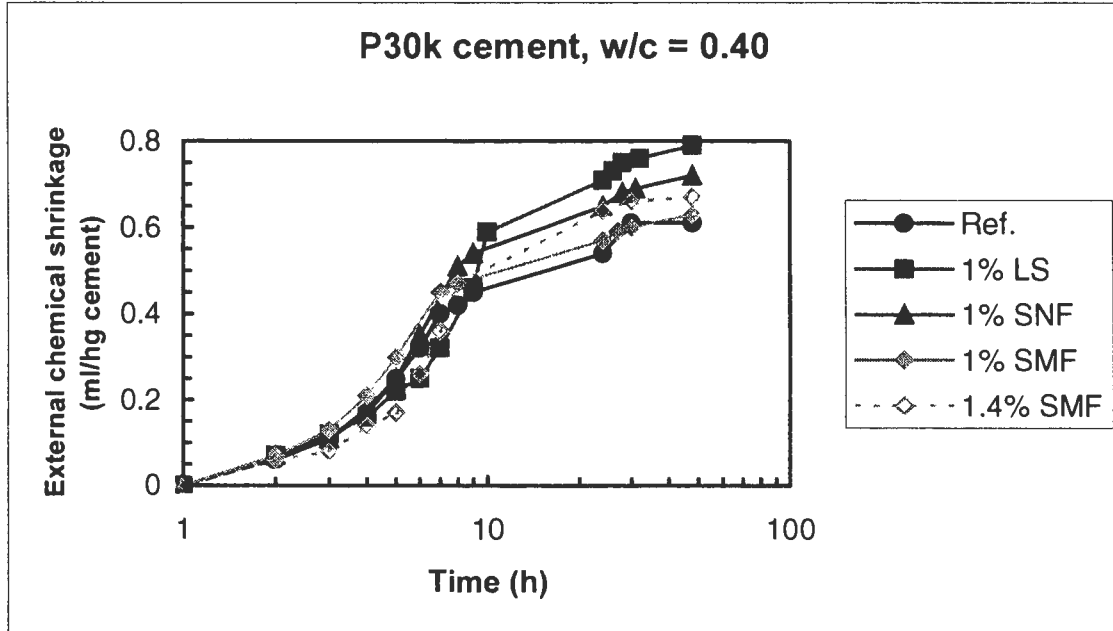


Fig. 10 External chemical shrinkage versus time curves for P30k cement paste with w/c = 0.40 and 1.0 % LS, SNF and SMF, and 1.4 % SMF, as compared with the reference without admixtures.

The results in Fig. 7 show that 0.7 % SNF and SMF have equal influence on hydration and that both accelerate the hydration for the whole 48 h period of measurement compared with the reference. 0.7% LS marginally retards the hydration in the early period from 4 - 10 h, but are otherwise equal to the reference. The external chemical shrinkage curves, including the FOLs, are for all practical purposes equal (Fig. 8) when admixtures are dosed 0.7 % of cement weight. Fig. 9 reveals that even with a 1 % dosage the SNF and SMF accelerate hydration between 10 and 48 h (about equal to reference before 10 h), and that 1.4 % SMF behaves about equal (marginally extra retardation between 4 – 8 h) to 1.0 % SMF. The hydration acceleration by SNF and SMF is less at 1.0 % than at 0.7 % dosage. LS retards somewhat more in the early period from 4 to 10 h when dosed at 1.0 % compared with 0.7 %, but the degree of hydration after 24 h is higher for the highest dosage. The external chemical shrinkage curves for the higher dosages in Fig. 10 is about equal until the flattening out level, which is marginally increasing in the order Ref.  $\approx$  1 % SMF < 1.4 % SMF  $\approx$  1 % SNF < 1 % LS. The difference between the highest and lowest FOL is however not more than 0.2 ml/100 g cement.

The total and external shrinkage versus time profiles for P30c cement pastes with w/c = 0.40 are plotted in Figs. 11 and 12, respectively, for the different admixtures at 1 % dosage. 1 % LS retards the P30c cement hydration in the period 5 - 8 h, while it is marginally accelerated by the other admixtures in the same period. The degree of hydration is higher than the reference and equal for the three admixtures in the period 24 - 48 h. The external shrinkage curves in Fig. 12 for all three admixtures changes gradually from plastic to rigid state and reaches an equal flattening out level (about 0.7 ml/100 g cement) that is much lower than the reference (about 1.2 ml/100 g cement). The latter difference is probably due to “bleeding” since the reference sample was rotated by hand (180° every 15 min, which was probably insufficient).

The total and external shrinkage versus time profiles for G cement pastes with w/c = 0.40 are plotted in Figs. 13 and 14, respectively, for the different admixtures at 1 % dosage. Fig. 13 shows that the hydration of G cement is virtually unaffected by the admixtures until 7 h. Thereafter the G cement hydration is retarded by LS and SNF, but accelerated by SMF. However, at 48 h the hydration with LS is equal to the reference. The external chemical shrinkage profiles for G cement in Fig. 14 are equal for 1% SNF and SMF until the flattening out level, which is marginally higher for SNF (+ 0.1 ml/100 g cement). The curve for 1 % LS is retarded relative to the others, but reaches the same FOL as 1% SNF.

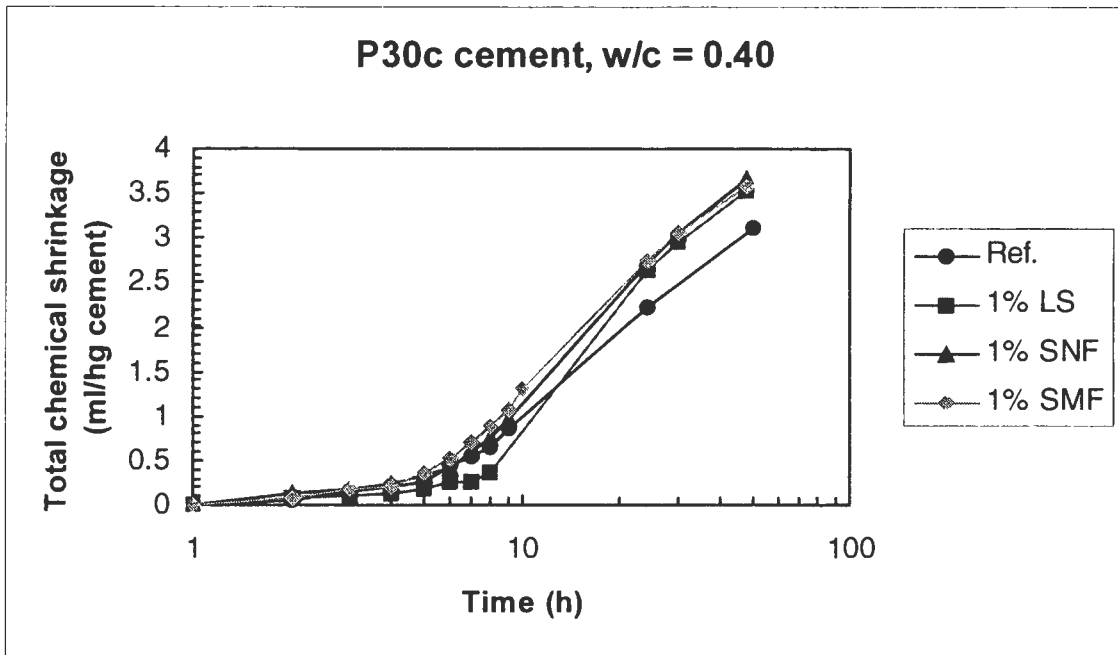


Fig. 11 Total chemical shrinkage versus time curves for P30C cement paste with w/c = 0.40 and 1.0 % LS, SNF and SMF as compared with the reference without admixtures.

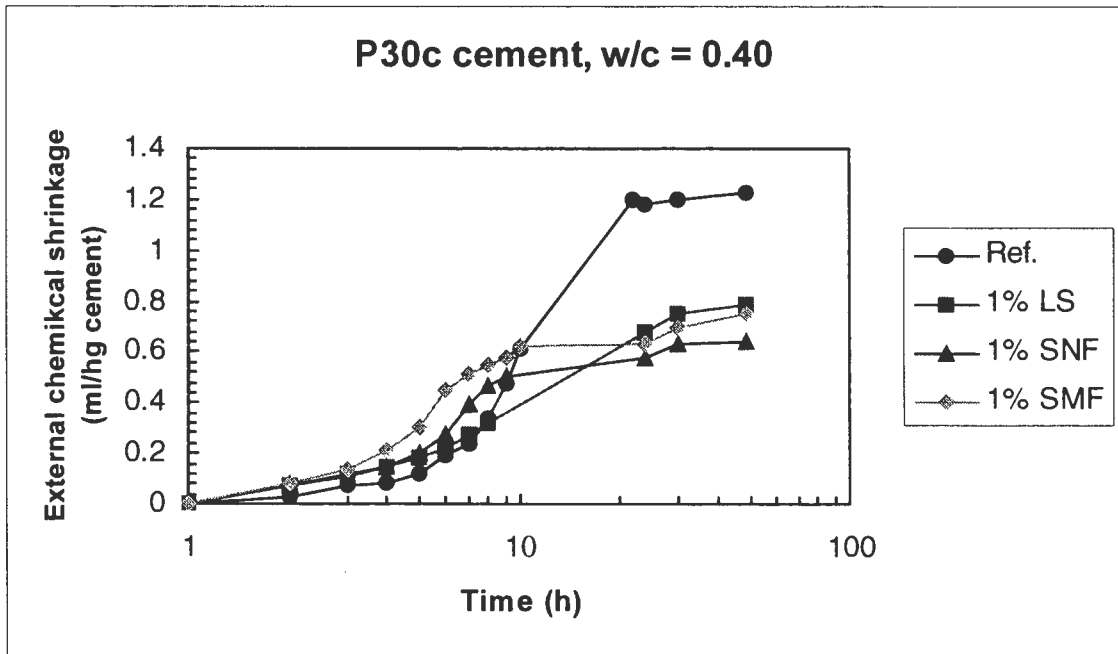


Fig. 12 External chemical shrinkage versus time curves for P30c cement paste with w/c = 0.40 and 1.0 % LS, SNF and SMF as compared with the reference without admixtures. Reference rotated by hand.

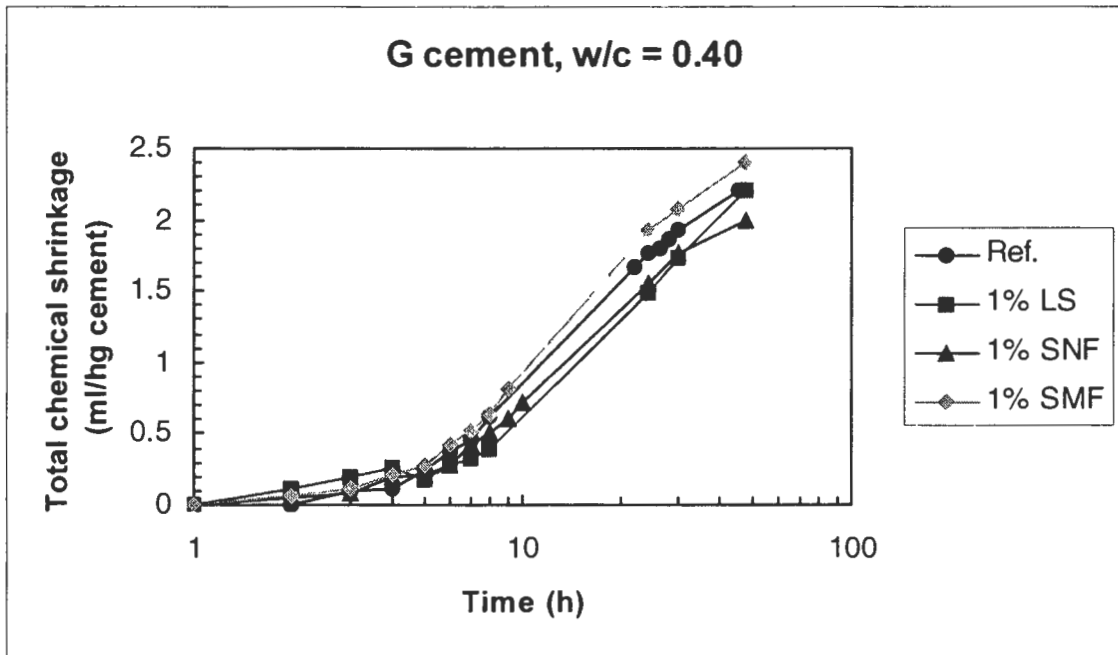


Fig. 13 Total chemical shrinkage versus time curves for G cement paste with w/c = 0.40 and 1.0 % LS, SNF and SMF as compared with the reference without admixtures.

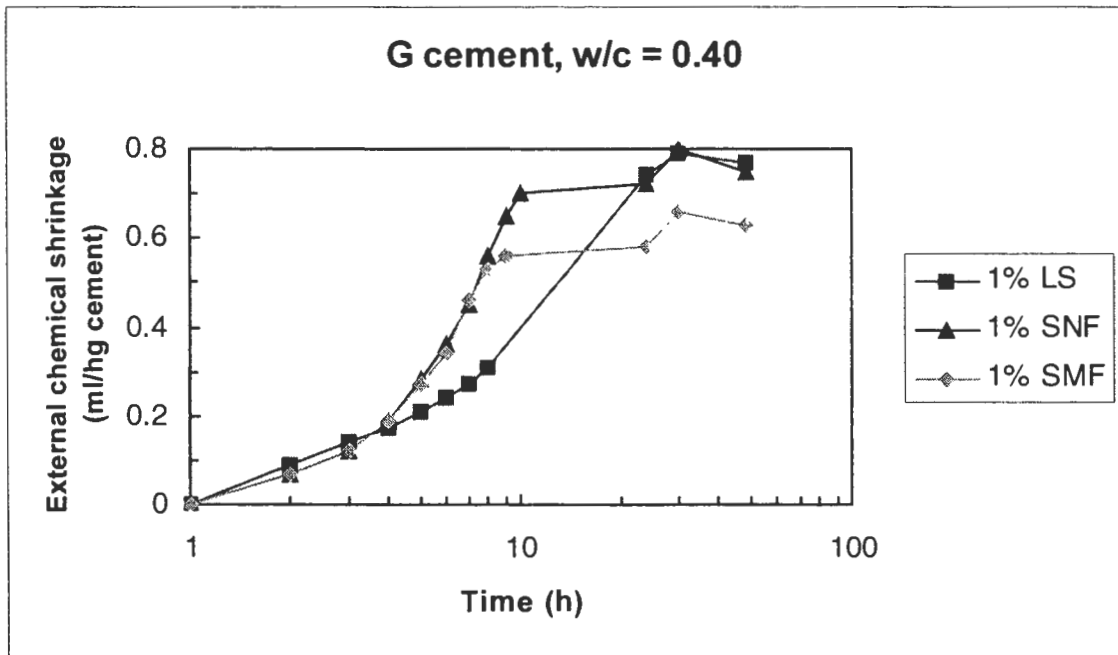


Fig. 14 External chemical shrinkage versus time curves for G cement paste with w/c = 0.40 and 1.0 % LS, SNF and SMF as compared with the reference without admixtures. Rotated reference not measured.

#### 4. CONCLUSION

As expected, LS (lignosulphonate) retards early hydration (until 10 h) more than the super-plasticizers SNF (naphthalene sulphonate - formaldehyde condensate) and SMF (sulphonated melamine - formaldehyde condensate).

In spite of retardation of early hydration and setting, LS may increase cement hydration in the period 24 to 48 h, probably due to improved dispersion and breaking of possible cement agglomerates.

SNF and SMF accelerated hydration for some cements for the whole period of measurement (48 h).

The flattening-out-level of the external chemical shrinkage is independent of admixture type, and the same as the reference without admixtures, when the samples were rotated (undisturbed by "bleeding").

The admixtures did not directly influence the chemical shrinkage in a way that should indicate a higher risk of cracking. However, the retardation of early hydration and setting of cement by lignosulphonate could allow a longer period of evaporation of water from floors and bridge decks in practice. For this reason mixes with lignosulphonate might be more prone to form drying shrinkage cracks.

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