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Influence of limestone filler and viscosity-modifying admixture on the shrinkage of self-compacting concrete

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ABSTRACT

The aim of this experimental work is to study shrinkage evolution with age in self-compacting concretes (SCC) made with w/c = 0.6 and different limestone filler and viscosity-modifying admixture (VMA) contents. The results show that limestone fillers speed up hydration reactions and provide a finer porous structure. As a result, when specimens are hardened under water, SCC made with limestone fillers tends to shrink, since the water only penetrates the outer layers of the specimen, while the interior is subject to self-desiccation. If the concrete contains substantial air content (3.2%) the water finds it easier to penetrate and the concrete swells. When hardening takes place in the open air, autogenous shrinkage in SCC is lower than in normally-vibrated concretes (NVC) and the higher the fines content, the lower the shrinkage. This is more than likely due to the use of limestone filler as addition, finer porous structure and higher amount of absorbed water from the aggregate which compensates for the auto-desiccation of the concrete. On the whole, drying shrinkage in SCC is greater than in NVC. However, when filler is replaced by VMA the porous structure is coarser and shrinkage is reduced by 33%, thanks to the lower capillary pressure. This means that due to the reduction in autogenous and drying shrinkage, SCC made solely with VMA presents 7.7% lower total shrinkage than NVC. In SCC made with limestone filler, total shrinkage is only slightly higher than in NVC, the differences being under 9.2%.

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1. Introduction

Total shrinkage is the sum of various types of shrinkage: chemical, autogenous, drying and carbonation. Even though changes of temperature and carbonation reactions have an effect on shrinkage, this phenomenon is mainly the result of water loss and the reduction of the volume of hydrated products compared with the reacting constituents (cement, active additions and water). During the hydration of cement after the start of setting, chemical shrinkage does not occur freely, since it is hindered by the stiffness of the paste itself, by the granular skeleton and possibly by the reinforcement. As a result, an increasing volume of very fine pores is formed inside the hydrated cement paste which drains water from the coarser capillaries. If water is not supplied from an external source, as hydration of the cement progresses, the volume of water in the pores decreases, a process which is known as self-desiccation, and water menisci are formed. Due to the water surface tension, as the water content in the capillary pores is reduced, the force of attraction between pore walls is increased, giving rise to increased shrinkage. This shrinkage, known as autogenous shrinkage, is higher at low w/c ratios and becomes especially significant when the w/c ratio falls below 0.4 [1,2]. In concrete with a w/c ratio over 0.60, autogenous shrinkage is usually between five and ten times less than that caused by drying, so that its effects are not normally taken into consideration [1]. In general, a low w/c ratio and a high cement content cause increased autogenous shrinkage, possibly due to the greater volume of small-diameter pores and the reduction of the available water [3]. Before initial setting of the concrete, capillary forces are almost negligible, so that chemical shrinkage is practically the only contributor to autogenous deformation.

When water is applied during curing, the water lost from the capillary pores in the concrete is replaced by water from the external source, so that self-desiccation does not occur. However, when the water/binder ratio is very low, even when there is an external water supply, the products formed by hydration may obstruct some capillaries and prevent the water from getting to certain zones in the concrete, in which autogenous shrinkage will also occur. The influence of this phenomenon on total shrinkage depends on the size of the specimen [4].

Drying shrinkage is a relatively slow process. The driving force behind it is the loss of water from the pores due to the lower humidity prevailing outside the concrete. The diffusion of water through the

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material mainly depends on its porous structure, the size and shape of the specimen, the duration of drying and the prevailing atmospheric conditions [5–7].

High fines and cement content and a higher paste volume than in NVC are normally used in making SCC. Since the cement paste is mainly responsible for changes of volume, the higher the paste content, the greater the shrinkage will be [5,8,9], so that, in theory, SCC is liable to suffer a considerable degree of shrinkage. However, significantly increasing the paste volume does not necessarily lead to greater shrinkage, since other factors also intervene in the process, such as the continuity of the capillary network and the density of the matrix [10]. Shrinkage may also be promoted by the retarding effect of the powerful superplasticizers used to make SCC [11–13]. In addition, the higher the dosage, the longer the delay in hydration reactions [13].

Many authors point out that shrinkage is higher in SCC than NVC [10,11,14–17]. However, there are also studies that did not find significant differences between both types of concrete [18–23], and even some that found lower shrinkage in SCC [24]. However, it should be pointed out that in many of these studies the SCC, unlike the NVC used as a reference, contained a high content of active additions, in order to meet this type of concrete's great need for fines. These additions may affect the concrete's fresh and hardened properties and therefore may also affect its shrinkage. Shrinkage may be increased by increasing the content of silica fume [25–27] or granulated blast furnace slag [28–30]. On the other hand, the use of fly ash or limestone filler may reduce both autogenous and drying shrinkage [8,9,28,31–33]. Some authors consider that the limestone fines content has no influence either on autogenous [10,19,23] or on total shrinkage if the same water/powder ratio is maintained [21].

Viscosity-modifying admixtures (VMA) may be used to increase the viscosity of SCC. Some studies have shown that by using VMA more stable concrete is obtained in view of water variations in the mixtures than that obtained with fines only [34,35]. This enhanced stability seems to have a bearing on the quality of the aggregate-paste interface, with a denser structure in the transition zone [34,36,37]. The major construction chemical industries are at present developing a new family of VMA that will allow SCC to be made without additional fines. With these new admixtures the aim is to obtain a

Table 1 Mixture proportions of concretes.

	N-300	S-300/125	N-350	S-350/0	S-350/45	S-350/90
Cement (kg/m³)	300	300	350	350	350	350
Limestone filler (kg/m³)	0	125	0	0	45	90
Water (l)	180	180	210	210	210	210
w/c	0.6	0.6	0.6	0.6	0.6	0.6
Superplasticizer (kg/m³)	3.60	5.70	1.05	5.60	5.25	5.08
VMA (kg/m³)	0	0	0	1.75	1.23	0
Coarse aggregate (kg/m ³)	865.9	733.6	883.9	642.4	684.3	726.4
Fine aggregate (kg/m³)	1070.6	1072.7	939.0	1164.9	1080.1	996.5
Total aggregates (kg/m³)	1936.5	1806.3	1822.9	1807.3	1764.4	1722.9
Total fine particles (kg/m ³)	493.5	613.6	524.6	548.7	582.7	616.9
Paste volume (dm³)	354.6	402.8	391.1	406.3	418.6	430.4
Water absorbed by aggregates and filler (1)	14.31	16.37	13.22	13.95	14.77	15.59

VMA: viscosity-modifying admixture.

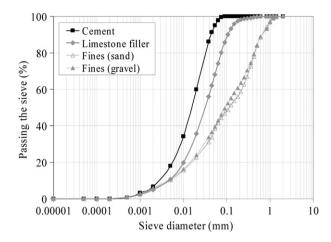


Fig. 1. Grading curves of cement, aggregate fines and limestone filler.

more robust concrete, although this could involve the loss of the benefits of fines, such as the better particle-packing which leads to a denser cementitious matrix.

Bearing in mind the foregoing, the aim of this experimental work was to study shrinkage in SCC made with differing limestone filler and VMA contents, comparing the results obtained with those obtained from traditionally vibrated concrete. Due to the fact that shrinkage is directly related to the porous structure of the material, the microstructure of the different types of concrete was analysed in order to explain the behaviour observed.

2. Experimental programme

2.1. Concrete mixtures, materials and mixing procedure

Six types of concrete were made: four self-compacting and two normally-vibrated concretes, using different cement, limestone filler and viscosity-modifying admixture contents. In order to obtain strengths approaching those normally used for building, all of them were manufactured with a 0.60 w/c ratio. The characteristics of each mix are shown in Table 1. The nomenclature used to identify each type of concrete refers to self-compacting (S) or normal (N) type concrete, cement content (300 or 350 kg/m³) and the limestone filler addition (0, 45, 90 or 125 kg/m³).

To produce the self-compacting concretes, CEM II/A-S 42.5 N cement was used with crushed limestone aggregates: 4/10 gravel and 0/3 sand. Additionally, limestone filler was also used. The grading curves of the cement, the fines of coarse and fine aggregates and the filler (particle size <0.063 mm) are determined by means of laser diffraction (Fig. 1). The properties of the aggregates are shown in Table 2 and the characteristics and composition of the cement are shown in Table 3. The admixtures used were a polycarboxylate-based superplasticizer (Viscocrete 3425) and a modified starch-based VMA (Stabilizer 4R). The properties of the mixtures are displayed in Table 4.

Taking as a reference the mixes established for SCC, the corresponding reference NVC were made with the same types and amounts of cement. The superplasticizer was in this case a calcium

Table 2 Properties of the aggregates.

Type of aggregate	Specific gravity (g/dm³)	Fines (%)	Water absorption (%)
Filler limestone	2.65	-	1.16
Fine sand 0/3	2.63	14.9	0.90
Gravel 4/12	2.70	3.9	0.54

Table 3Characteristics and chemical composition of the cement.

Type of cement Blaine fineness (cm²/g)		Strength (28 days)	Composition (%)											
	(MPa)	SiO ₂	Al_2O_3	Fe ₂ O ₃	CaO	MgO	SO ₃	K ₂ O	Na ₂ O	Ti ₂ O ₅	$P_{2}O_{5}$	P ₂ O ₅ CO ₂ LOI		
CEM II/A-S 42.5N	3850	51.1	23.02	4.93	3.70	60.41	2.79	2.46	0.60	0.51	0.33	0.11	0.55	1.76

lignosulfonate (Sikament 175). Its content was adjusted to achieve a slump of 90 ± 10 mm in the Abrams cone for the two mixtures.

The mixing sequence of the concrete was carried out by firstly adding the aggregates, the filler and the cement. Once a homogenous mixture was obtained, 3/4 of the water was added and after mixing for a further minute, the superplasticizer was added with the remaining water. Total mixing time was 6 min for NVC and 8 min for SCC made with limestone filler. In the specific case of SCC with VMA, it was observed that a longer mixing time was required since the initial viscosity of the mixture was much higher, but then gradually reduced until it stabilised. More specifically, 3 min after adding the superplasticizer, the VMA was added and mixing continued until completing the total time of 12 min.

2.2. Test programme and methodology

2.2.1. Shrinkage test

To determine autogenous shrinkage, total shrinkage and swelling, prismatic specimens measuring $100 \times 100 \times 400$ mm were used. To obtain further information on this phenomenon, setting time (ASTM C 403-08), temperature and weight loss were also measured. Thermocouples were inserted into the centre of the specimens immediately after casting (Fig. 2) to determine the evolution of temperature rise due to the hydration reaction.

In order to measure shrinkage of the specimens until mould removal (48 h), the specimens were made in accordance with the recommendations of the Technical Committee on Autogenous Shrinkage of Concrete of the Japan Concrete Institute [38]. A polystyrene sheet (thickness 3 mm) was placed on the bottom and on both sides of the mould so that free movement of the specimen was not restricted by the mould. A polyester film was also placed on the polystyrene sheet on all sides of the mould and on the surface of the specimen. At the time of initial setting, the end plates of the mould were removed and two dial gauges were put into place (Fig. 3). Later, at the age of 48 h, the specimens to be used for measuring autogenous shrinkage, drying shrinkage and weight loss were stored in a vertical position inside a climatically controlled chamber for more than one year at 20 °C and 50% RH (Fig. 4). To determine autogenous shrinkage, half of the specimens were sealed with various layers of plastic film to avoid moisture loss. Those used to measure total shrinkage were not sealed. In order to study swelling, some specimens were submerged in water at 20 °C in containers for a period of 90 days.

Three batches were made from each mix. One specimen for each type of test was made from each batch, the result of each test being the arithmetic mean of the three values obtained. Two 150 mm

Table 4 Results of the mixture properties.

Mix	Air content	Slump flo	ow test	Compressive strength (28 days)
	(%)	Ø _f (cm)	T ₅₀ (s)	(Mpa)
N-300	1.1	No test	No test	39.4
N-350	1.5	No test	No test	39.0
S-300/125	3.2	62	3.5	40.4
S-350/0	1.1	67	1.5	39.6
S-350/45	0.9	69	1.2	36.9
S-350/90	0.9	71	1.0	38.7

diameter \times 300 mm high cylindrical specimens were also made from each batch for compressive strength tests at 28 days (EN 12390-3:2003). The compressive strength results are shown in Table 4.

2.2.2. Mercury intrusion porosimetry (MIP) test

At the age of 28 days, pore size distribution was determined using a Micromeritics AutoPore IV-9500 mercury porosimeter with a maximum pressure of 60,000 psia (414 MPa). At this pressure, the smallest pore size into which mercury can be introduced is 3 nm. This test was carried out on small drilled cores (12 mm diameter × 23 mm high), weighing approximately 6 g. The cored samples were obtained from $100 \times 100 \times 100$ mm cubic specimens. The samples were first dried in an oven at 110 °C and then immersed in mercury under gradually increasing pressure. Using this technique, a measure of the total porosity of the sample may also be obtained, as well as the surface area of the pore network. However the MIP technique has certain limitations. First, the assumption that the intruded pores are cylindrical is not fully in agreement with the characteristics of real pore structure. Second, the sample must be dried prior to intrusion and this can cause microstructural damage. Due to surface tension, when the water evaporates the tensile stress generated by the water menisci in the capillaries causes the collapse of some of the fine pores [39]. Moreover, microcracking may be induced in concrete as result of cement hydrates desiccation and as result of differential thermal expansion of the aggregates and hardened cement paste. As a



Fig. 2. Measurement of the temperature inside concrete.

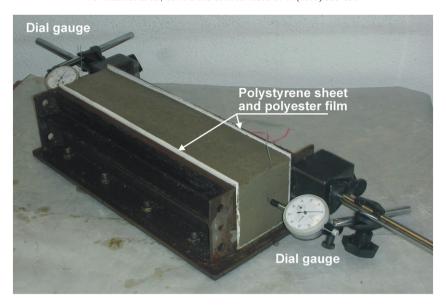


Fig. 3. Measurement of shrinkage before mould removal (age less than 48 h).



Fig. 4. Shrinkage test (age more than 48 h).

result, capillary porosity increases [39,40] and specific surface area of the pores is reduced [41]. However, total mercury porosity values are not so much influenced by the drying technique [39]. A third problem is known as the ink-bottle effect, in which a larger pore is preceded in the intrusion path of the mercury by a smaller neck. This may produce pore size distribution curves with an exaggeratedly high volume of smaller pores and a small volume of larger pores. Despite these limitations, the MIP technique is still a very effective aid for comparing the pore structure and pore network characteristics of different types of cement-based materials. The main characteristics of the pore structure of the concretes under study are given in Table 5. A sample was taken from each of the three batches of each type of concrete. The results shown in Table 5 are the arithmetic mean of the three values obtained.

3. Results and discussion

Shrinkage strain and thermal strain due to heat generation during cement hydration are generated simultaneously. The temperature of the concrete was monitored by thermocouples from casting up to the age of 72 h (Fig. 5). During this period, the temperature change was not very significant (of the order of 4.4 K), due to the chemical composition of the cement (low C₃A content) and to the small size of the concrete specimen. In Fig. 5 it can be seen that the temperature rise occurs during the first day (from 5 to 25 h), after which it falls to ambient temperature approximately one day later. In order to eliminate the temperature effect, the shrinkage values were corrected considering that $\Delta \varepsilon = \alpha \cdot \Delta T$, where $\Delta \varepsilon$ is the thermal strain, α is the coefficient of thermal expansion (1/K) and ΔT is the temperature change (K). In order to determine temperature distribution across the specimen, a $100 \times 100 \times 400$ mm specimen was also made from each mix, which was broken into four pieces after 24 h. Temperature distribution was recorded in each of the three resulting cross-sections by means of a thermographic camera with a resolution of 0.1 K (Fig. 6). The results show a fairly homogenous distribution, with temperature differences throughout the cross-section of less than 0.4 K. The strain/time curve corrected for the temperature effect corresponds to the volumetric change of concrete under isothermal conditions. As in other similar studies [4], $\alpha = 10^{-5}$ /K was selected. After 60 h the temperature variation in the mixtures studied was quite small, so this effect was neglected.

The cement hydration rate tended to be a little faster in S-350/90, due to the accelerating effect of limestone filler on cement hydration

Table 5Pore characteristics of concrete mixtures.

	N- 300	S-300/ 125	N- 350	S-350/ 0	S-350/ 45	S-350/ 90
Total cumulative volume (mm ³ /g)	53.4	48.5	63.9	64.3	68.9	66.1
Total specific surface area (m^2/g)	7.89	8.27	8.76	9.66	10.94	10.78
Average pore diameter (nm)	28.2	23.5	29.2	26.9	25.3	24.6
Threshold diameter (µm)	0.4	0.15	0.15	0.15	0.15	0.1
Total porosity (%)	12.57	11.54	14.52	14.64	15.49	15.76

[42–44] (Fig. 7). As fines content is reduced in the mixture, initial setting time increases. Thus, for example, there is a difference of 135 min between S-350/90 and S-350/45 and between S-350/45 and S-350/00 the difference is 90 min. NVC initial setting time was slightly less than SCC made without additional fines (S-350/0), probably due to a reduced content of superplasticizer in the NVC mix. As polycarboxylate-based superplasticizers can have a strong retarding effect, a reduction in superplasticizer content leads to faster setting [11–13].

3.1. Concrete pore size distribution

Figs. 8 and 9 show the increase in mercury intrusion volume according to the equivalent pore diameter. Pore volume distribution follows a similar pattern in the six mixes. Total pore volume was very similar in all the concretes made with the same cement content (Table 5). The most significant differences between the different mixes are seen to be in the capillary pore volume $(0.01-1 \, \mu m)$. This is an important aspect, since, according to Kumar and Bhattacharjee [45] although the smallest pores have no effect on the concrete's strength properties, they are directly related to shrinkage and creep.

3.1.1. Comparison of SCC and NVC mixes

SCCs show a finer porous structure than NVCs. In N-300 the volume of pore size between 0.15 and 0.4 µm is far higher than in S-300/125 (around 130%), with the below-0.15 µm pore volume being very similar in both mixes (Fig. 8). For the concrete made using 350 kg/m³ (Fig. 9), the differences between SCC and NVC are lower, which is logical because the volume of paste in both types of concrete is similar (Table 1). In N-350 the maximum concentration of pores tends to be around larger pore sizes than in SCCs (mixes S-350/0, S-350/45 and S-350/90), with a lower volume of smaller capillary pores and greater volume of large capillary pores. In Figs. 8

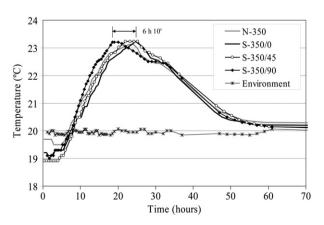


Fig. 5. Temperature evolution in the first 72 h.

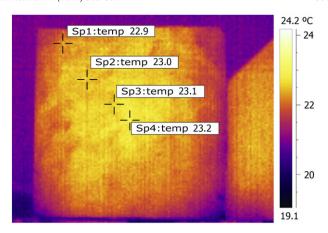


Fig. 6. Temperature distribution throughout the specimen (thermography test).

and 9 and Table 5 it can also be seen that the threshold diameter is lower for SCCs than in the corresponding reference NVCs. This diameter marks the limit from which the highest number of pores is concentrated and therefore is a good indicator of the fineness of the porous structure. Furthermore, average pore diameter is smaller in SCCs and the specific surface area of the SCCs is greater than that of the NVCs. These results agree with those obtained in previous work [46]. SCCs can therefore be said to show a finer porous structure than NVCs, probably due to the higher content of aggregates in NVCs (in the aggregate/paste interfacial transition zone (ITZ) the porosity is higher and the capillary pores are larger than those in cement paste [47]) and to the addition of limestone filler in SCCs which produces the following effects: a) filler effect, b) creation of more nucleation sites (since there are more nucleation sites, the size of portlandite crystals is smaller and therefore porosity is lower since, on the one hand the ITZ is not so thick [48] and on the other, the crystals behave like microcracks that create additional porosity [49]), and c) a small part of the CaCO₃ present in the limestone filler promotes the reconversion of monosulfoaluminate to ettringite, leading to an increase of the total volume of the hydrate phase [50-53] (as ettringite has a low density and thus a relatively large volume per formula unit). Besides, by using a more powerful superplasticizer in SCC than in NVC, cement dispersion is enhanced, thus reducing the formation of floccules that leave pores on the inside [54]. On the other hand, the vibration involved in NVC enhances bleeding, creating an interconnected network of capillary pores.

3.1.2. Influence of fines and VMA content

The composition of the three SCC types made with 350 kg/m³ of cement differs in the limestone fines and VMA contents. As can be

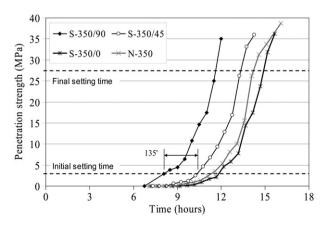


Fig. 7. Setting time.

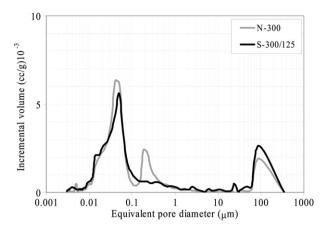


Fig. 8. Pore size distribution.

seen in Fig. 9, in S-350/90 the threshold diameter is lower than for the other two types of SCCs (S-350/0 and S-350/45): 0.10 μm compared to 0.15 μm . Moreover, the volume of large capillary pores (larger than 0.1 μm) and macropores (larger than 100 μm) is 35.2% less than in S-350/0 and 31.7% less than in S-350/45. In other words, the porous structure is finer. This is because of better particle packing afforded by the limestone fines, which broaden the grain-size distribution and provide a denser cementitious matrix, since they occupy the spaces between cement particles. Also, since the S-350/90 concrete has a lower aggregate content (Table 1), the aggregate-paste ITZ is smaller and therefore, in accordance with Section 3.1.1, there are fewer large capillary pores.

3.2. Compressive strength

The compressive strength results are shown in Table 4. Although SCC pore structure is finer than NVC, total pore volume is quite similar, which means that the compressive strength values of all the concretes are also similar, with no statistically significant differences between SCC and NVC.

3.3. Swelling

As shown in Fig. 10, all the specimens undergo an increase in volume due to water absorption after being submerged in water. However, after the third day, the SCC types containing limestone filler (S-350/45 and S-350/90) change their behaviour and start to shrink. This is probably due to the greater fineness of their porous structure,

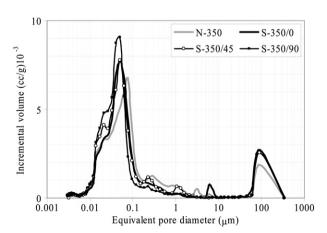


Fig. 9. Pore size distribution.

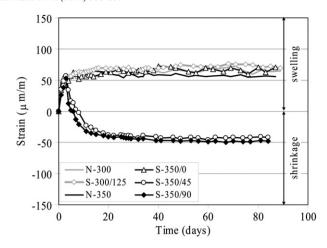


Fig. 10. Concrete strain under water.

as shown by the MIP tests, so that water is only absorbed by the outer layer of the specimen, while the interior undergoes self-desiccation. In this case, the mean change in specimen length is influenced by both swelling and autogenous shrinkage, which occur simultaneously in the cross section. This behaviour was also observed in other studies [55] involving concrete with a low w/c ratio.

However, an exception was observed in the case of S-300/125, which did not behave like S-350/45 and S-350/90, in spite of having smaller total pore volume, smaller mean pore diameter and similar pore threshold diameter. This could have been due to the considerable content of trapped air in S-300/125 (Table 4) caused by the greater viscosity and lower fluidity of the mixture in fresh state [56]. The presence of these macropores facilitates water absorption and thus the swelling of the specimen. The rest of the concrete types all show very similar swelling values with no significant differences. Similarly to S-350/45 and S-350/90, deformation progresses rapidly and tends to stabilise after approximately 20 days of age.

3.4. Autogenous shrinkage

Figs. 11 and 12 give the evolution of autogenous shrinkage with time. As expected from concrete made with a w/c ratio of 0.6, this shrinkage is only a small percentage of the total shrinkage in the first few days (Fig. 13). However, in the longer term, autogenous shrinkage is not as insignificant as some other authors have found [4]. As the specimens lose water and approach hygrometric equilibrium with the atmosphere, drying shrinkage slows down and stabilises. However, as cement hydration continues, autogenous shrinkage carries

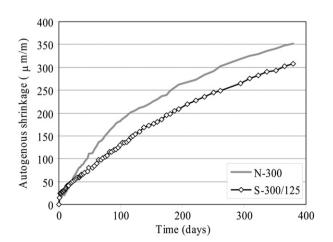


Fig. 11. Autogenous shrinkage.

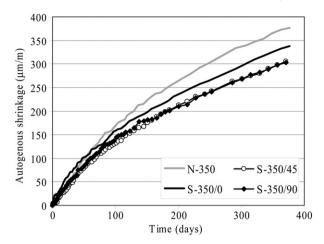


Fig. 12. Autogenous shrinkage.

on due to self-desiccation within the specimen, so that after one year the autogenous/total shrinkage ratio increases to between 0.4 and 0.55, depending on the type of concrete (Fig. 13).

3.4.1. Comparison of SCC and NVC mixes

Since SCC is made with a higher volume of paste (Table 1) and has a finer porous structure, in theory it could be expected to undergo greater shrinkage than NVC, given that cement paste is the principal material responsible for changes of volume [5,8,9] and the finer the capillaries the greater the tensile stress generated by the water menisci in the capillaries. However, this did not turn out as expected; the SCC presented less shrinkage than the corresponding reference NVC, the differences being on average between 14.3% for concrete made with 300 kg of cement and 19.8% for those made with 350 kg. This could be due to various causes. The calcium carbonate present in the limestone filler promotes the reconversion of monosulfoaluminate to ettringite (see Section 3.1.1), leading to an increase of the total volume of the hydrate phase [52,53]. Also, the filler not involved in the reaction (i.e. most of the filler) behaves like a normal aggregate, hindering shrinkage of the cement paste. On the other hand, as SCC has a finer porous structure, water diffusion from the capillary pore network to the gel pores formed during cement hydration is also slower and this slows down the self-desiccation process in the larger capillary pores, which means that less autogenous shrinkage takes place.

Another factor exists which, as far as we are aware, has not been considered in other studies, which is the water absorbed by the

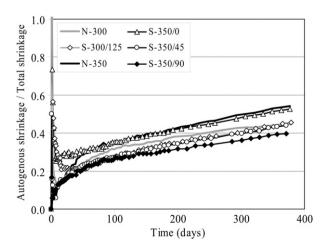


Fig. 13. Ratio between autogenous shrinkage and total shrinkage.

aggregates. Since mixes are designed assuming that aggregates are saturated with a dry surface, in the course of concrete self-desiccation the aggregates compensate for the loss of water from the paste with the water they have absorbed. Table 1 indicates the quantity of water absorbed by aggregates and filler. It can be seen that the capacity to provide extra water is greater in the SCC mixes than in the corresponding reference NVC mixes and thus the self-desiccation is lower in SCC. The SCC types therefore show less long-term autogenous shrinkage than the NVC. This also explains why in S-350/45 and S-350/90 water loss in unsealed specimens is higher than in N-350 (Fig. 14) in spite of the finer porous structure of the former (slower water diffusion), due to the fact that total water content is higher (mix water + water absorbed by aggregates). This can also be verified by comparing S-300/125 and N-300.

3.4.2. Influence of fines and VMA content

Taking mixes S-350/0, S-350/45 and S-350/90 as reference, in Fig. 12 it can be seen that concrete made with VMA only (S-350/0) tends to present higher shrinkage than those made with limestone filler addition: 10.7% more than mix S-350/45 and 11.6% more than S-350/90. According to the information given in the preceding section, this can be explained by the high water absorption coefficient of the filler, since the extra water from the filler and aggregates was less in S-350/0 than S-350/45 and 350/90 (Table 1). Also, when VMA is used as a substitute for limestone filler, the favourable effect of the filler on hindering shrinkage disappears, as also does its filler effect, which helps to speed up internal water diffusion and thus also autogenous shrinkage.

3.5. Drying shrinkage

Drying shrinkage cannot be measured. As many authors have pointed out, this shrinkage is obtained by subtracting autogenous from total shrinkage. However, this process involves a small error, which can be seen in Figs. 15 and 16 in the form of a descent in the curve at advanced ages. This, of course, is impossible, since it would mean that the concrete stops shrinking and starts to swell. The error is due to the fact that in unsealed specimens self-desiccation is less important than in sealed specimens, since when the ambient RH is higher than that in the concrete, a small quantity of water vapour is absorbed by the concrete, which compensates for its self-desiccation.

3.5.1. Influence of fines and VMA content

As shown in Fig. 16, the higher the fines content in SCC mixes (0, 45 and 90 kg/m^3), the higher the shrinkage. At the age of one year

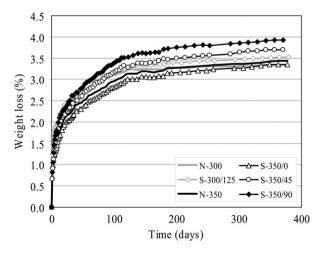
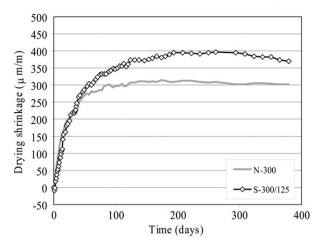


Fig. 14. Weight loss.





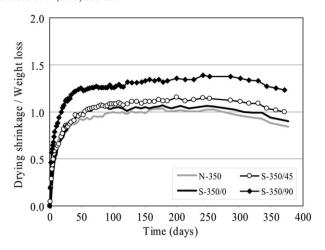


Fig. 17. Ratio between drying shrinkage and weight loss.

shrinkage in S-350/90 was 18.1% higher than S-350/45 and 50% higher than S-350/0. Given that the volume of cement paste (cement, water and additives) was the same in all three, the differences observed can be explained by the fineness of their porous structure, since the finer the capillaries, the greater the force of attraction exerted by the capillary walls. In addition, due to the water surface tension, since total specific pore surface area is greater (Table 5), the total force of attraction exerted is also greater. In fact, in the figure drawn to illustrate the relationship between shrinkage and water loss (Fig. 17), it can be seen that the concrete with the finest porous structure (S-350/90) shows the greatest shrinkage for the same quantity of evaporated water and vice-versa. The use of VMA to replace limestone filler therefore can be said to produce concrete with reduced drying shrinkage.

3.5.2. Comparison of SCC and NVC mixes

In general, SCC presents greater drying shrinkage than NVC (Figs. 15, 16). This can also be explained by the influence of the limestone filler, which gives SCC a finer porous structure and a larger total specific pore surface area and thus greater capillary forces. In fact, the smallest differences between SCC mixes and the corresponding reference NVC mixes were recorded between N-350 and S-350/0 (4.6%), which have a very similar porous structure. In this particular case, the lower shrinkage shown by the SCC in relation to NVC could be due to the action of the VMA in limiting water migration by bonding with the hydrogen in the water [57,58] (in other tests carried out in

parallel with this research, we found that water evaporation is reduced when mixed with VMA.). On the other hand, the biggest differences were obtained between N-300 and S-300/125 (22.7%) and N-350 and S-350/90 (43.4%); i.e. when a considerable amount of filler was added to the SCC.

3.6. Total shrinkage

Drying shrinkage composed the most important part of the total shrinkage of the concrete mixes analysed in this work, especially at an early age. Thus, in conformity with the contents of the previous section, SCC in general suffers greater deformation than NVC, and the higher the fines content the greater the total shrinkage (Figs. 18, 19). However, the differences between individual concretes are not as significant as in drying shrinkage, due to the lesser autogenous shrinkage of concrete made with higher fines contents. For example, the differences between S-350/90 and mixes S-350/45 and S-350/0 are 9.8% and 18.3%, respectively. Also, the differences between NVC mixes (N-300 and N-350) and the corresponding SCC mixes made with high fines contents (S-300/125 and S-350/90) are below 9.2%. Even the SCC made with VMA only (S-350/0) shows 7.7% less shrinkage than N-350, not only due to lower autogenous shrinkage but also to its lower drying shrinkage. The use of VMA as a substitute for limestone filler therefore definitely produces SCC with lower total shrinkage.

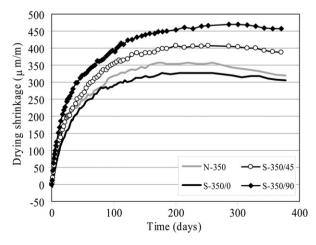


Fig. 16. Drying shrinkage.

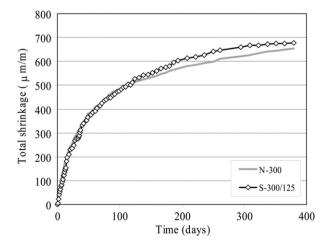


Fig. 18. Total shrinkage.

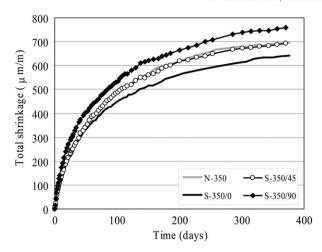


Fig. 19. Total shrinkage.

4. Conclusions

In accordance with the results of the experimental work carried out, the following conclusions can be drawn:

- The higher the limestone fines content in the mix, the shorter the initial and final concrete setting times.
- SCCs have a finer pore structure than NVCs, because of the use of lower amount of aggregates, higher fines content and powerful superplasticizers that enhance dispersion of the finest particles.
- When submerged in water, SCC made with limestone filler and low air content (0.9%) do not suffer swelling but shrinkage, due to their finer porous structure, so that water only penetrates the outer layers of the specimen, while the interior is subject to self-desiccation. However, if the air content is significant (3.2%) water finds it easier to enter and so the concrete swells.
- Even though at early ages the autogenous/total shrinkage ratio is small, as time passes it increases. After one year this ratio reaches values ranging between 0.4 and 0.55, depending on the type of concrete.
- Autogenous shrinkage in SCC was lower than in NVC and the higher the fines content the lower the shrinkage. This is probably due to a combination of factors: the use of limestone filler as addition, a finer porous structure, which slows down water diffusion within the specimen, and a higher amount of absorbed water from the aggregate, which compensates for concrete self-desiccation. The average differences between both types of concrete were 14.3% for those made with 300 kg of cement and 19.8% for those made with 350 kg of cement.
- The use of VMA to replace limestone filler produces SCC with lower drying shrinkage, being about 33% less than SCC made with limestone filler without VMA.
- Drying shrinkage in SCC is higher than NVC, except in the case of those made with VMA without additional fines.
- Total shrinkage in SCC made with limestone filler is slightly higher than in NVC (differences below 9.2%). However, SCC made with VMA shows 7.7% less shrinkage than NVC, due to lower autogenous and drying shrinkage.

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References

- [1] P.C. Aïtcin, Demystifyig autogenous shrinkage, Concr. Int. 21 (11) (1999) 54-56.
- [2] M.D. D'Ambrosia, D.A. Lange, A.J. Brinks, Restrained shrinkage and creep of self-consolidating concrete, in: S.P. Shah (Ed.), Proceedings of 2nd North American Conference on the Design and Use of Self-Consolidating Concrete (SCC) and the 4th International RILEM Symposium on Self-Compacting Concrete, Chicago 2005 pp. 921–928
- [3] E. Tazawa, S. Miyazawa, Effect of constituents and curing conditions on autogenous shrinkage of concrete, in: E. Tazawa (Ed.), Autogenous Shrinkage of Concrete, Taylor & Francis, 1999, pp. 269–280.
- [4] P.C. Aïtcin, Autogenous shrinkage measurement, in: E. Tazawa (Ed.), Autogenous Shrinkage of Concrete, Taylor & Francis, 1999, pp. 257–268.
- [5] B. Bissonnette, P. Pierre, M. Pigeon, Influence of key parameters on drying shrinkage of cementitious materials, Cem. Concr. Res. 29 (10) (1999) 1655–1662.
- [6] S.A. Al-Saleh, R.Z. Al-Zaid, Effects of drying conditions, admixtures and specimen size on shrinkage strains. Cem. Concr. Res. 36 (10) (2006) 1985–1991.
- 7] A.M. Neville, Properties of Concrete, Longman Group, London, 1996.
- [8] E. Rozière, P. Turcry, A. Loukili, Influence of paste volume, addition content and addition type on shrinkage cracking of self-compacting concrete, in: S.P. Shah (Ed.), Proceedings of 2nd North American Conference on the Design and Use of Self-Consolidating Concrete (SCC) and the 4th International RILEM Symposium on Self-Compacting Concrete, Chicago, 2005, pp. 945–951.
- [9] T.A. Hammer, The influence of some mix design parameters on drying shrinkage of SCC, in: G. De Schutter, V. Boel (Eds.), Proceedings of 5th International RILEM Symposium on Self-Compacting Concrete, RILEM Publications S.A.R.L., Ghent, 2007, pp. 559–564.
- [10] D. Chopin, O. Francy, S. Lebourgeois, P. Rougeau, Creep and shrinkage of heat-cured self-compacting concrete (SCC), in: O. Wallevik, I. Nielsson (Eds.), Proceedings of the 3rd International RILEM Symposium on Self-Compacting Concrete, RILEM Publications S.A.R.L., Reykjavik, 2003, pp. 672–683.
- [11] P. Turcry, A. Loukili, A study of plastic shrinkage of self-compacting concrete, in: O. Wallevik, I. Nielsson (Eds.), Proceedings of the 3rd International RILEM Symposium on Self-Compacting Concrete, RILEM Publications S.A.R.L., Reykjavik, 2003, pp. 576–585.
- [12] J. Roncero, R. Gettu, Influencia de los superplastificantes en la microestructura de la pasta hidratada y en el comportamiento diferido de los morteros de cemento, Cem. Hormig. 832 (2002) 12–28.
- [13] F. Puertas, T. Vázquez, Early hydration cement. Effect of admixtures superplasticizers, Mater. Constr. 51 (262) (2001) 53–61.
- [14] J.K. Kim, S.H. Han, Y.D. Park, J.H. Noh, Material properties of self flowing concrete, J. Mater. Civ. Eng. 10 (4) (1998) 244–249.
- [15] S. Rols, J. Ambroise, J. Péra, Effects of different viscosity agents on the properties of self-leveling concrete, Cem. Concr. Res. 29 (2) (1999) 261–266.
- [16] R. Loser, A. Leemann, Effects of curing time and drying behaviour of SCC in case of restrained shrinkage deformations, in: G. De Schutter, V. Boel (Eds.), Proceedings of 5th International RILEM Symposium on Self-Compacting Concrete, RILEM Publications S.A.R.L., Ghent, 2007, pp. 539–544.
- [17] G. Heirman, L. Vandewalle, The Influence of Fillers on the Properties of Self-Compacting Concrete in Fresh and Hardened State, in: O. Wallevik, I. Nielsson (Eds.), Proceedings of the 3rd International RILEM Symposium on self-compacting concrete, RILEM Publications S.A.R.L., Reykjavik, 2003, pp. 606–618.
- [18] B. Persson, A comparison between mechanical properties of self-compacting concrete and the corresponding properties of normal concrete, Cem. Concr. Res. 31 (2) (2001) 193–198.
- [19] M. Vieira, A. Bettencourt, Deformability of hardened SCC, in: O. Wallevik, I. Nielsson (Eds.), Proceedings of the 3rd International RILEM Symposium on Self-Compacting Concrete, RILEM Publications S.A.R.L., Reykjavik, 2003, pp. 637–644.
- [20] G. Pons, E. Proust, S. Assié, Creep and Shrinkage of Self-Compacting Concrete: A Different Behaviour Compared with Vibrated Concrete? in: O. Wallevik, I. Nielsson (Eds.), Proceedings of the 3rd international RILEM symposium on self-compacting concrete, RILEM Publications S.A.R.L., Reykjavik, 2003, pp. 645–654.
- [21] V. Seng, H. Shima, Creep and Shrinkage of Self-Compacting Concrete with Different Limestone Powder Contents, in: S.P. Shah (Ed.), Proceedings of 2nd North American conference on the design and use of self-consolidating concrete (SCC) and the 4th international RILEM symposium on self-compacting concrete, Chicago, 2005, pp. 981–985.
- [22] M. Collepardi, A. Borsoi, S. Collepardi, R. Troli, Strength, shrinkage and creep of SCC and flowing concrete, in: S.P. Shah (Ed.), Proceedings of 2nd North American Conference on the Design And Use of Self-Consolidating Concrete (SCC) and the 4th International RILEM Symposium on Self-Compacting Concrete, Chicago, 2005, pp. 911–919.
- [23] J. Piérard, V. Dieryck, J. Desmyter, Autogenous shrinkage of self-compacting concrete, in: S.P. Shah (Ed.), Proceedings of 2nd North American Conference on the Design and Use of Self-Consolidating Concrete (SCC) and the 4th International RILEM Symposium on Self-Compacting Concrete, Chicago, 2005, pp. 1013–1021.
- [24] G. Heirman, L. Vandewalle, D. Van Gemert, Influence of Mineral Additions and Chemical Admixtures on Setting and Volumetric Autogenous Shrinkage of SCC-Equivalent-Mortars. in: G. De Schutter, V. Boel (Eds.), Proceedings of 5th

- international RILEM symposium on self-compacting concrete, RILEM Publications S.A.R.L., Ghent, 2007, pp. 553–558.
- [25] O.M. Jensen, P.F. Hansen, Autogeneous deformation and change of the relative humidity in silica fume-modified cement paste, ACI Mater. J. 93 (6) (1996) 539–543.
- [26] J.J. Brooks, J.G. Cabrera, M.A. Megat Johari, Factors affecting the autogenous shrinkage of silica fume high-strength concrete, in: E. Tazawa (Ed.), Autogenous Shrinkage of Concrete, Taylor & Francis, 1999, pp. 195–202.
- [27] G.A. Rao, Long-term drying shrinkage of mortar influence of silica fume and size of fine aggregate, Cem. Concr. Res. 31 (2) (2001) 171–175.
- [28] Y.W. Chan, C.Y. Liu, Y.S. Lu, Effects of slag and fly ash on the autogenous shrinkage of high performance concrete, in: E. Tazawa (Ed.), Autogenous Shrinkage of Concrete, Taylor & Francis, 1999, pp. 221–228.
- [29] B. Persson, Creep, shrinkage and elastic modulus of self-compacting concrete, in: A. Skarendhal, Ö. Petersson (Eds.), Proceedings of 1st International RILEM Symposium on Self-Compacting Concrete, Stockholm, 1999, pp. 239–250.
- [30] H.W. Song, K.J. Byun, S.H. Kim, D.H. Choi, Early creep and shrinkage in self compacting concrete. Incorporating GGBFS, in: K. Ozawa, M. Ouchi (Eds.), Proceedings of Second International Symposium on Self-Compacting Concrete, Tokyo, 2001, pp. 413–422.
- [31] T.A. Hammer, K. Johansen, Ø. Bjøntegaard, Volume changes as driving forces to self-induced cracking of Norwegian SCC, in: K. Ozawa, M. Ouchi (Eds.), Proceedings of Second International Symposium on Self-Compacting Concrete, Tokyo, 2001, pp. 423–432.
- [32] S.N. Lim, T.H. Wee, Autogenous shrinkage of ground-granulated blast-furnace slag concrete, ACI Mater. J. 97 (5) (2000) 587–593.
- [33] S. Tangtermsirikul, Effect of chemical composition and particle size of fly ash on autogenous shrinkage of paste, in: E. Tazawa (Ed.), Autogenous Shrinkage of Concrete, Taylor & Francis, 1999, pp. 175–186.
- [34] N. Petrov, K.H. Khayat, A. Tagnit-Hamou, Effect of stability of self-consolidating concrete on the distribution of steel corrosion characteristics along experimental wall elements, in: K. Ozawa, M. Ouchi (Eds.), Proceedings of Second International Symposium on Self-Compacting Concrete, Tokyo, 2001, pp. 441–450.
- [35] D. Lowke, K. Wiegrink, P. Schiessl, A simple and significant segregation test for SCC, in: O. Wallevik, I. Nielsson (Eds.), Proceedings of the 3rd International RILEM Symposium on Self-Compacting Concrete, RILEM Publications S.A.R.L., Reykjavik, 2003, pp. 358–366.
- [36] J. Trägardh, Microstructural features and related properties of self-compacting concrete, in: A. Skarendhal, Ö. Petersson (Eds.), Proceedings of 1st International RILEM Symposium on Self-Compacting Concrete, Stockholm, 1999, pp. 175–186.
- [37] W. Zhu, P.J.M. Bartos, Microstructure and properties of interfacial transition zone in SCC, in: Z. Yu, C. Shi, K.H. Khayat, Y. Xie (Eds.), Proceedings of 1st International Symposium on Design, Performance and Use of Self-Consolidating Concrete, Changsha, China, 2005, pp. 319–327.
- [38] Japan Concrete Institute, Technical Committee on Autogenous Shrinkage of Concrete, Taylor & Francis, 1999.
- [39] C. Gallé, Effect of drying on cement-based materials pore structure as identified by mercury intrusion porosimetry. A comparative study between oven-, vacuum-, and freeze-drying, Cem. Concr. Res. 31 (10) (2001) 1467–1477.

- [40] M. Moukwa, P.C. Aitcin, The effect of drying on cement pastes pore structure as determined by mercury porosimetry, Cem. Concr. Res. 18 (5) (1988) 745–752.
- [41] A. Korpa, R. Trettin, The influence of different drying methods on cement paste microstructures as reflected by gas adsorption: comparison between freeze-drying (F-drying), D-drying, P-drying and oven-drying methods, Cem. Concr. Res. 36 (4) (2006) 634-649
- [42] G. Ye, X. Liu, G. De Schutter, A.M. Poppe, L. Taerwe, Influence of limestone powder used as filler in SCC on hydration and microstructure of cement pastes, Cem. Concr. Compos. 29 (2) (2007) 94–102.
- [43] M. Bouasker, P. Mounanga, P. Turcry, A. Loukili, A. Khelidj, Chemical shrinkage of cement pastes and mortars at very early age: effect of limestone filler and granular inclusions, Cem. Concr. Compos. 30 (1) (2008) 13–22.
- [44] B. Craeye, G. De Schutter, B. Desmet, J. Vantomme, G. Heirman, L. Vandewalle, Ö. Cizer, S. Aggoun, E.H. Kadri, Effect of mineral filler type on autogenous shrinkage of self-compacting concrete, Cem. Concr. Res. 40 (6) (2010) 908–913.
- [45] R. Kumar, B. Bhatttarcharjee, Porosity, pore size distribution and in situ strength of concrete. Cem. Concr. Res. 33 (1) (2003) 155–164.
- [46] M. Valcuende, C. Parra, Natural carbonation of self-compacting concretes, Constr. Build. Mater. 24 (5) (2010) 848–853.
- [47] J.P. Ollivier, J.C. Maso, B. Bourdette, Interfacial transition zone, Adv. Cem. Based Mater. 2 (1) (1995) 30–38.
- [48] A. Leemann, B. Münch, P. Gasser, L. Holzer, Influence of compaction on the interfacial transition zone and the permeability of concrete, Cem. Concr. Res. 36 (8) (2006) 1425–1433.
- [49] C. Vernet, Mécanismes chimiques d'interactions ciment-adjuvants, GTG Spa, Guerville, Service Physico-Chimie du Ciment, January 1995.
- [50] G. Kakali, S. Tsivilis, E. Aggeli, M. Bati, Hydration products of C3A, C3S and Portland cement in the presence of CaCO3, Cem. Concr. Res. 30 (7) (2000) 1073–1077.
- [51] V.L. Bonavetti, V.F. Rahal, E.F. Irassar, Studies on the carboaluminate formation in limestone filler-blended cements, Cem. Concr. Res. 31 (6) (2001) 853–859.
- [52] T. Matschei, B. Lothenbach, F.P. Glasser, The role of calcium carbonate in cement hydration, Cem. Concr. Res. 37 (4) (2007) 551–558.
- [53] B. Lothenbach, G. Le Saout, E. Gallucci, K. Scrivener, Influence of limestone on the hydration of Portland cements, Cem. Concr. Res. 38 (6) (2008) 848–860.
- [54] C. Legrand, E. Wirkin, Study of the strength of very young concrete as a function of the amount of hydrates formed — influence of superplasticizer, Mater. Struct. 27 (2) (1994) 106–109.
- [55] E. Tazawa, S. Miyazawa, Influence of constituent and composition on autogenous shrinkage of cementitious materials, Mag. Concr. Res. 49 (178) (1997) 15–22
- [56] M. Valcuende, C. Parra, E. Marco, A. Garrido, E. Martínez, J. Cánoves, Influence of limestone filler and viscosity-modifying admixture on the porous structure of self-compacting concrete, Constr. Build. Mater. 28 (1) (2012) 122–128.
- [57] M.R. Rixom, N.P. Mailvaganam, Chemical admixtures for concrete, E & FN Spon (Ed.), London, 1999.
- [58] D.P. Bentz, M.A. Peltz, K.A. Snyder, J.M. Davis, Verdict: viscosity enhancers reducing diffusion in concrete, Concr. Int. 31 (1) (2009) 31–36.