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# Drying kinetics of self-compacting concrete

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#### Abstract

A comparative study of the drying kinetics of self-compacting concrete (SCC) with those of an ordinary concrete and sand concrete (SC) shows a monomodal porometry structure of the SCC with a peak located in the vicinity of 1000 Å (macropores), with, however, for SC a peak located in the vicinity of 250 Å (mesopores), whereas ordinary concrete (OC) presents a bimodal structure with 2 peaks: the first corresponds to micropores and the second to macropores.

Another important and unexpected result is that SCC with macropores has lower drying kinetics than SC with mesopores. Furthermore, the SCC pores are more tortuous and less oriented with a lower degree of connectivity than SC.

Concrete with limestone filler as SCC and SC has finer pores than conventional concrete.

Key Words: Self-compacting concrete, Porometry, Gammadensimetry, Drying kinetics, Sand concrete, Ordinary concrete.

# Introduction

Self-compacting concrete (SCC) was first developed 35 years ago in Japan. Since then, various investigations have been carried out in Japan, Germany, France, USA, and others countries. Research work on SCC is focussed on establishing a rational mix design method and test methods (Ozawa et al., 1986; Chiara et al., 2000; Dirk, 2001; Dehn, 2002). However, many questions have been asked about its durability, whereas few studies have been carried out on it as noted by Stephen (2003). The studies concerning this field usually reported in the literature do not take into account the porometry (porosity and pore distribution) or the gammadensimetry of the material. Pore structure is among the main factors influencing the transport properties and the durability of concrete (sulfatic attack, acid attack, freeze-thaw, water absorption, chloride ions penetration etc.). The

drying kinetics has a deep influence on the delayed deformations (shrinkage and creep), which are important criteria for durability. Since transport properties of concrete and drying kinetics strongly depend on the pore structure, the purpose of the present investigation is to study the porometry and drying kinetics of SCC. To better understand the behaviour of SCC towards drying, the results were compared with the corresponding properties (porometry and drying kinetics) of ordinary concrete (OC) and sand concrete (SC) with the same water/cement ratio, and compressive strength at 28 days. Taking into account SC in the comparison could be an interesting way to highlight the very close links that exist between porometry and the use of a large amount of filler because the SC as the SCC requires an important quantity of filler in its composition.

# **Principles of Composition**

SCC consists basically of the same components as ordinary concrete (OC). However, there exist differences regarding the composition. Content of fine material is higher in SCC than in OC, and a larger quantity of superplasticizer and/or stabilizer should be used; if necessary. SCC should not only flow under its own weight but should also fill the entire form and achieve uniform consolidation without segregation. Studies to develop SCC have been carried out by Ozawa et al. (1986) at the University of Tokyo.

SC is distinguished from OC by its composition. Theoretically, SC is constituted only by cement, sand, and water. However, various components are added such as water reducing agents (superplasticiser) because of material fineness. To improve the performance of SC, fillers are added to increase the compacity and strength of the material.

For the mix design of OC, the experimental method recommended by Lesage (1974) and Gorisse (1974) was used to obtain the optimum sand-aggregate ratio.

## **Properties of Tested Concretes' Constituents**

Properties of the limestone, cement, and sand are illustrated in Tables 1-3.

**Table 1.** Chemical composition of limestone [%].

$SiO_2$	CaO	MgO	$Fe_2O_3$	$CO_2$	$H_2O$
15.5	45.4	0.4	0.5	36	2.2

Table 2. Chemical composition of cement (CEM II A-32.5) [%].

$SiO_2$	CaO	MgO	$Fe_2O_3$	$Al_2O_3$	$SO_3$	loss on ignition
21.9	63.8	O.2	3.9	6.6	1.5	1.9

Table 3. Physical properties of sand.

Nature	Bulk density	Specific density	% of fines	Sand equivalent	
Calcareous	1.4	2.6	1.4	95.5	

Viscosity agent: VISCOCRETE 20 HE, acrylic copolymer following NF EN 934-2, pH =  $4.5 \pm 1$ , cl<sup>|</sup>  $\leq 0.1\%$ Superplasticiser: MEDAFLUID SFR following NF EN 934-2, pH =  $6.7 \pm 1$ , cl<sup>|</sup>  $\leq 0.1\%$  and density =  $1.18 \pm 0.01$ 

Table 4 gives the compositions of the tested concretes.

Constituents	Siliceous	CPJ- CEM II	Calcareous	Water	Super	Calcareous	Viscosity	Agg []	regate kg]
	sand [kg]	A-32,5	[kg]	[1]	[1]	sand [kg]	agent [1]		
Concretes		[Kg]						3/8	8/15
OC	680	320	-	170	-	-	-	450	770
SC	1500	350	200	190	7	-	-	-	-
SCC	578	400	78.95	206	-	249	6	333	492.2

**Table 4.** Mix proportions of tested concretes (for  $1 \text{ m}^3$ ).

# **Properties of Fresh Concrete**

The material properties were evaluated using recommendations according to the AFGC (Association Française de Génie Civil, 2000). It is important to appreciate that none of the test methods for SCC has yet been standardised, and these tests are not yet definitive. Commonly used tests are the slump flow, L-box, and sieve stability test.

• Slump flow test and T<sub>50</sub> test:

Flow test consists of the determination of concrete sample diameter spread on a plate base after realising a slump test without any compaction in obstructions absence. This test gives an appreciation about filling ability of concrete and also some indication of its resistance to segregation.

The  $T_{50}$  test indicates the time necessary to reach a spread of 500 mm. This second indication is about flow: a lower time indicates greater flow ability. The AFGC suggested that a time of 2 to 5 s is appropriate.

• L-Box test:

This test, based on a Japanese design for underwater concrete, has been described by Petersson et al. (1990). With this widely used test it is possible to measure the filling and passing ability of SCC. It also detects any serious lack of stability.

• Sieve test stability:

This test has been developed in France to assess segregation resistance (stability). It consists of taking a sample of 10 L of concrete and pouring half of it into a 5 mm sieve of 350 mm diameter. After 2 min, the mortar that passed through the sieve is weighed, and expressed as a percentage of weight of the original sample on the sieve. Table 5 presents the mechanical characteristics of the SCC.

Test	Slump flow (cm)	T <sub>50</sub> (s)	L. Box (H <sub>2</sub> /H <sub>1</sub> ) (%)	Sieve test stability (%)	
Results	sults 73.6 2.9 0.9		7 %		
Acceptance criteria	60 ÷ 75	≤7	≥ 0.8	$0 \le \pi \le 15\%$	

Table 5.Properties of fresh SCC.

The OC is characterised by

- Slump cone test: 8-10 cm;
- Plastic consistency.

The sand concrete is characterised by

- Slump cone test: 6 cm;
- LCPC maniabilimeter: 15-20 s;
- Thixotropic behaviour.

# Mechanical properties of hardened concretes

The relevant material properties are given in Table 6 and each value included in it is obtained from 3 cylinders (11 cm diameter and 22 cm high). Procedures followed the French norm (NFP 18-406 for compressive strength, and standard method of LCPC / France for modulus of elasticity).

Regarding tensile strength, a cylinder splitting test (Brazilian test) was used according to NFP 18-408 (see Figure 1).

Mechanical	Concretes	Age (days)						
(MPa)		3	7	14	28	60	90	
	OC	14	20	25	26	26	29	
Compressive strength	SC	11	17	21.5	23.6	23.6	28.8	
	SCC	20.2	25.4	31.6	31.7	32	34.6	
	OC	2	2.1	2.1	2.5	2.5	2.9	
Tensile strength	SC	1.2	1.9	2.2	2.3	2.4	2.5	
	SCC	2.7	2.8	2.9	3.1	3.1	3.3	
	OC	21,000	27,900	27,900	31,000	31,000	32,500	
Modulus of elasticity	SC	17,000	19,700	19,700	23,000	23,000	23,000	
	SCC	22,000	29,000	29,000	32,000	32,000	33,500	

 Table 6.
 Mechanical characteristics of tested concretes.



Figure 1. Tensile splitting test.

# Drying Kinetics by Gammadensimetry Method

This non-destructive method is based on gamma rays' absorption, which enables monitoring the evolution of density according to depth and time. Experiments were performed on a disk specimen (16 cm in diameter and 10-cm thick) whose faces were made tight, thus undergoing radial drying. The specimen is placed on a plate allowing rotation and vertical displacement. The specimen is crossed by a collimated beam of gamma rays, which are measured (Figure 2). The position of the test tube makes possible the monitoring of each crown density (Figure 3). Figure 4 shows that SCC is affected by fast and uniform drying. Amplitude of density variation in the heart of the specimen at the end of 90 days represents nearly 65% of that measured at the external envelope of the specimen during the same period. Thus tensile gradients are not very significant and resulting density of cracking is weak.



Figure 2. Test set-up of gammadensimetry.



Figure 3. Cross section of disk specimen illustrating studied crowns.

Figure 4. Density change of SCC.

To understand better drying kinetics of SCC, a comparison was made with an OC and a SC to verify if the kinetics differ from each other and analyse the influence of calcareous filler on drying, since both SCC and SC are formulated with limestone filler in contrast to OC.

## Comparative Analysis of Drying Kinetics for Tested Concretes

Figure 5 illustrates that OC drying affects only one peripheral crown of a few millimetre thickness during the first months. This leads to hydraulic gradients and consequently to an increase in the cracking density of the specimen. These results have been explained by Benaissa et al. (1992). This drying process of OC as it is illustrated by Figure 5 is very different from one of SCC, where the drying mechanism is relatively fast and uniform (Figure 4).

Figure 6 shows that drying kinetics of sand concrete is extremely fast and uniform; indeed the heart undergoes drying from the fifth day after demoulding of the specimen, at 48 h.



Figure 5. Density change of OC.

Figure 6. Density change of SC.

Amplitude of variation of density in the heart of the specimen at the end of 90 days represents more than 80% of that measured at peripheral drying during the same period. Thus the hydraulic and tensile gradients are weak and density of cracking is consequently weak. This behaviour of SCC is not very different from that of SC. The difference in kinetics of the 2 concretes is about 15% after 4 months and uniformity of drying of SCC is lower than that of SC. Such behaviour of SCC drying (more different from that of OC and not too different from that of SC) could be explained by the fact that compositions of SCC and SC include a large amount of filler. Filler is used to complete granulometric distribution in order to enhance its granular packing factor and to modify poromety.

The porometry seems to play a central role in drying kinetics. Thus the difference in desiccation observed between the tested concretes should be related to this factor. To verify this assumption, we analysed 3 fragments of hardened SCC and SC at 90 days by Hg porosimetry. Obtained results of each series of concrete (SCC and SC) were very close, and that is why we have taken into account only 1 amongst the 3 results. These results were also compared to the curves obtained by Verbeck and Helmuth (1968).

# Porometry of SCC

Concrete is a porous material of gel pores and capillary pores, which include mesopores and macropores. According to categorisation of pore size given by the International Union of Pure and Applied Chemistry (IUPAC, 1972), gel pores are smaller than 20 Å, mesopores are included in the range of 20 Å to 500 Å, and macropores are larger than 500 Å. We consider also that the maximum limit of macropores is 10,000 Å, as suggested by Brandt (1995).

The main goal of this study is to compare the mercury intrusion porosimetry (MIP) of SCC, OC, and SC. MIP was conducted on a specimen excluding coarse aggregate. Thus samples of SCC and OC consisted of only hydrated cement paste. This was due to the practical restrictions of the apparatus. Three replicates were used in each concrete (SCC and SC). Samples were dried in an oven at 110 °C for 48 h after 28 days of curing and stored in a desiccator of silica-gel before testing. Surface tension of mercury is 0.48 N/mm and the contact angle adopted is 141.1°. Maximum mercury pressure is 2000 bars and covering pore radius is 20 Å to 60  $\mu$ m.

Figures 7 and 8 represent respectively pore size distribution of SCC and SC. Figure 9 represents pore size distribution of ordinary concrete cement paste according to Verbeck and Helmut (1968).



Figure 7. Pore size distribution of SCC.

Figure 8. Pore size distribution of SC.

As can be seen in Figure 7, SCC's porometry structure is monomodal as is the SC one (Figure 8). However, OC presents a bimodal structure (Figure 9).

SCC shows a single peak in the vicinity of 1000 Å, around where are located more than 80% of the pores. Thus these pores are of the type "macro", whereas in OC hardened cement paste presents 2 peaks: the first one corresponds to micropores and is located in the range 50 Å to 100 Å according to water/cement ratio. The second peak corresponds to macropores (600 Å to 1300 Å).

Particular monomodal structure of SCC and SC is due to amount of filler used in their compositions and consequently we have a refinement of pores by an optimisation of granular skeleton. Baroghel et al. (2004) and Carrasco et al. (2004) studied in this direction. The water/cement ratio is kept equal at about 0.5 for all tested concretes to avoid any influence of this parameter on porometry.



Figure 9. Porosity size distribution curves of cement pastes for different E/C from Verbeck and Helmuth.

The influence on transport properties is attributed to the fact that paste is the main criterion of porometry and thus durability. In SCC and SC, a part of aggregates' volume is replaced by paste; therefore, volume of micropores (intrahydrate pores) is increased while the volume of macropores and capillary pores is decreased.

A large amount of calcareous filler incorporated in SCC and SC completes the granulometric distribution, leading to a reduction in the volume of capillary pores and enhancing the packing factor. It seems also that the use of a smaller amount of limestone filler combined with a viscosity agent in SCC reduces less greatly the volume of pores than in SC, where the quantity of limestone filler, 2.5 times higher than in SCC, is combined with superplasticiser. Tests will be performed in order to verify the difference in impact introduced by the viscosity agent and superplasticiser combined with limestone filler on porometry. Limestone powder used in SCC and SC contributes to hydration reactions instead of being only inert filler as it is often supposed. Limestone reacts with  $C_3A$  of cement to form calcium monocarboaluminates, which contribute to the densify matrix of SCC and SC. Kakali et al (2000) approach this topic in their study of  $C_3A$  and  $C_3S$  hydration in the presence of  $CaCO_3$ . These are the main factors explaining difference of the porometry between SCC and SC on one side and between SCC and OC on the other side. The lower amount of limestone (40% of those used in SC) in SCC leads to larger pores than in SC. Another factor explaining the difference is that SC is mixed without coarse aggregate.

An important and unexpected result is that SCC with macropores has much lower drying kinetics than SC with mesopores. This is an astonishing result because it is stated that only capillary pores that include mesopores and macropores contribute to the movement of water through concrete as reported by Reinhardt (1990) and Pradhan et al. (2005). Thus the drying kinetics of SCC should be faster than that of SC because of the higher impact of macropores on drying than mesopores, but this is not the case. Indeed Figure 10 shows that the magnitude of weight loss of SCC is at least 20% lower than that of SC at 160 days.

Curves have been obtained by testing 3 prismatic specimens (7 cm  $\times$  7 cm  $\times$  28 cm) of each concrete mix and each curve illustrated in Figure 10 is the average of 3 specimens.

The curve pattern of SC is similar to those of SCC and OC, but the amplitude is higher. SCC weight loss is 10% higher than that of OC because the capillary pores' structure is undoubtedly finer in SCC. This is related to the use of filler, which results in refinement of capillary pores.



Figure 10. Weight change over time of tested concretes.

Difference in structure explains the drying kinetics of SCC and SC compared to OC: rapid and uniform between the heart of the sample and its periphery whereas the OC drying kinetics is slow and not uniform, i.e. accelerated in the skin and slow in the heart of the specimen. This established fact leads to the conclusion that hydrous gradient causes a weak tensile gradient for SCC and SC, and an important tensile gradient in OC. Therefore the cracking density of OC is higher than that of SCC. Previous research by Benaissa et al. (1993) showed that the SC cracking density is lower than that in OC. OC drying kinetics is also similar to that of SCC and SC, but with lower magnitude according to their porometric structure.

Moreover, curves of Figure 10 show a contrary phenomenon, with weight loss amplitude of SCC (macroporous concrete) much less marked than that of SC (mesoporous concrete).

Results are the averages of 3 specimens for each kind of concrete (7 cm  $\times$  7 cm  $\times$  28 cm).

This is an extremely interesting and unexpected result that calls into question the generally established idea, according to which the larger the pore the faster the weight loss, especially when one passes from a category of pores to another (micropores, mesopores, and macropores).

However, the macropores of SCC, compared with the mesopores of SC, should have resulted in an amplitude of weight loss more important than in SC, but that was not the case.

This phenomenon, contrary to the SC, could be attributed to more tortuous and randomly oriented tubes of complex pores of SCC. Indeed in SC pores are certainly less tortuous and more oriented, thus privileging drying.

Lower weight loss of SCC compared to SC is related to a previously described phenomenon. In comparison to SC, the lower drying kinetics of SCC is undoubtedly related to the lower degree of connectivity of pores. This assumption is confirmed by the fact that the permeability of SCC, which needs 7.3 MPa to have duct drainage at 24 h, is lower than that of SC, which needs 5.8 MPa at the same age. Baroghel et al. (2004) pointed out that permeability increases notably with the degree of connectivity of pores. This fact has a drastic influence on hydrous transfer. This lower degree of pore connectivity in SCC should be related to the wider granulometry (0/15) of SCC compared to SC (0/5). We estimate too that the presence of coarse aggregates constitutes also an introducing factor of additional tortuosity of SCC pores compared to SC, which is deprived of coarse aggregates. The additional tortuosity leads to slow kinetics of hydrous transfer.

Another assumption would be that necks of SCC pores are smaller in dimension than the pores themselves, consequently explaining a difficult departure of water and thus slow drying.

Compared to OC, confrontation of SCC results remains quite logical. Indeed, the bimodal structure of OC and the presence especially of "macro-peak" explain the more accelerated drying in SCC than in OC. Benaissa et al. (2008) underlined factors at the origin of SC behaviour with respect to OC and which remain valid in the analysis of SCC behaviour compared to OC.

These explanations remain at the stage of assumptions given the difficulty in reasoning on the infinitely small scale and require to be confirmed by further analysis. Study of microstructure, evolution of porosity according to degree of hydration, and analysis by imagery can strongly contribute to a more rigorous approach to porometry and thus comprehension of mechanisms of desiccation of SCC compared to other types of concretes.

# Conclusion

From the present research, the following conclusions can be drawn:

- Porometric structure of SCC is of monomodal type.
- Porous mode of SCC is of "macro" type, while SC is mesoporous and OC is both microporous and macroporous.
- Drying kinetics of SCC between internal and external faces of specimens is relatively uniform.
- Drying kinetics of SCC is accelerated compared to OC of the same range.
- In spite of a macroporous mode of SCC and mesoporous mode of SC, drying is less pronounced in the SCC than SC, contrary to what is reported.
- Degree of connectivity of SCC pores is certainly less developed than that of SC, thus a more slowing down hydrous transfer.
- Quantity of filler in SCC (80 kg/m<sup>3</sup>) lower than in SC (200 kg/m<sup>3</sup>) can also reduce the degree of pore connectivity.
- Pores are more tortuous and less oriented in SCC than those of SC and OC, thus slow drying in SCC.
- SCC structure is finer than that of OC.
- Parameters with a large impact on porometry seem to be the pore size distribution, the degree of pore connectivity, and the tortuosity and orientation of pores. All these factors are related to the amount of filler in the mix composition combined with superplasticiser and/or viscosity agent.

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#### References

Baroghel-Bouny, V;, Ammouche, A;, Hornain, H. and Gawsewitch, J., "Vieillissement des bétons en milieu naturel. Une expérimentation pour le XXI<sup>e</sup>siècle", Bulletin des Laboratoires des Ponts et Chaussées, 228, Septembre-Octobre, 4328, 71-86, 2004.

Benaissa, A., Morlier, P., Viguier, C. and Chauvin, J.J., "*Cinétique de Dessiccation et Retrait du Béton de* Sable ". Revue de l'ITBTP, France, n° 504, juin 1992.

Benaissa, A., Morlier, P. and Viguier, C., "Microstructure du Béton de Sable " Cement and Concrete Research, 23, 663-674, 1993.

Benaissa, A., Boutaleb, A., Boudraa, S.E. and Malab, S., "The Sand Concrete: A Very Little Cracked Material" -11dbmc. Durability of Building Materials and Components, 11dbmc, Istanbul, Turkey, 2008.

Brandt, A.M., "Cement Based Composites: Materials, Mechanical Properties and Performance", E and FN Spon, UK, 1995.

Carrasco, M.F., Menédez, G., Bonavetti, V. and Irassar, E.F., "Strength Optimization of Tailor-Made Cement with Limestone Filler and Blast Furnace Slag". Cement and Concrete Research, 2004.

International Journal of Pure Application Chemistry, IUPAC, n° 31, 1972.

Chiara, F., Brower, L., Ozyildirim, C. and Daczko, J., "Workability of Self-Compacting Concrete" The Economical Solution for Durable Bridges and Transportation Structures", International Symposium on High Performance Concrete. Proceedings, PCI/FHWA/FIB, September 25-27, Orlando, Florida, 398-407, 2000.

Dehn, F., Holschemacher, K. and Dirk, W., "Self-Compacting Concrete (SCC): Time Development of the Material Properties and the Bond Behaviour". LACER n<sup>°</sup>. 5, 2002.

Dirk, L., "Segregation Resistance of Self Compacting Concrete", Scientific Report, University of Munich, Center for Building Materials (cbm), Munich, Germany, 2001.

Document Scientifique et Technique de l'Association Française de Génie Civil [AFGC], "Recommandations provisoires", 2000.

Gorisse, F., "Essais et Contrôles du Béton", Edition Eyrolles, Paris, 1974.

Manual of Symbols and Terminology, Appendix 2, part 1, "Colloid and Surface", 1972.

Kakali, G., Tsivilis, S., Aggeli, E. and Bati, M., "Hydration Products of C<sub>3</sub>A, C<sub>3</sub>S and Portland Cement in the Presence of CaCO<sub>3</sub>", Cement and Concrete Research, n°30, 1073-1077, 2000.

Lesage, R., " Etude Expérimentale De La Mise En Place Du Béton Frais", Rapport de Recherché n°37, Laboratoire Central des Ponts et Chaussées, 1974.

Ozawa, K., Mackawa, K., Kunishima, L. and Okamura, H., "Development of High Performance Concrete Based on the Durability of Concrete Structures", Proceedings of the Second East-Asia and Pacific Conference on Structural Engineering and Construction (EASEC-2), vol. 1, 445-450, January 1986.

Petersson, O., Billbery, P. and Van, B. K., "A Model of Self Compacting Concrete", Proceedings of RILEM Conferences on "Production Methods and Workability of Concrete. Edited by P. J. M. BARROS et al (Prisley, 1996), 483-490, 1990.

Pradhan, B., Nagesh, M. and Bhattacharjee, B., "Prediction of the Hydraulic Diffusivity from Pore Size Distribution of Concrete", Cement and Concrete Research, N°35; 2005.

Reinhardt, H.W. and Gaber, K., "From Pore Size Distribution to an Equivalent Pore Size of Cement Mortar", Material and Structure 23, 3-15, 1990.

Stephen, A., Escadeilas, G. and Marchese, G., "Durability of Self Compacting Concrete", 3<sup>rd</sup> International Symposium on Self Compacting Concrete 17-20, August, Reykjavik, Iceland, 2003.

Verbeck, G.J. and Helmuth, R.H., "Structures and Physical Properties of Cement Paste", Proc. of the  $5^{th}$  Int. Symp. on Chemistry of Cement, Session III-1, Tokyo, 1968.