EVALUATION OF POROSITY IN SELF-COMPACTING CONCRETE (SCC) PRODUCED WITH FLY ASH (FA) AND LIMESTONE FILLER (LF),

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ABSTRACT

SCC is increasingly used to improve specific construction aspects. In the near future, SCC can be expected to replace conventional concrete (CC) due to its many advantages.

Its main characteristics in the fresh state are achieved essentially due to changes in the content of the different components, namely the increase of the mortar's volume (more ultrafine material such as cement and additions) and the decrease of the coarse aggregates.

Nevertheless, the use of overly large volumes of additions such as FA and/or LF can substantially affect the concrete's pore structure and consequently its durability.

In this context, an experimental program was conducted to evaluate the effect, on the concrete's porosity, of incorporating FA and LF in binary and ternary mixes of self-compacting concrete.

With the results obtained, conclusions were established regarding the SCC durability considering its permeability and the microstructure of its pore structure.

Keywords: Self-compacting concrete; porosity; permeability, absorption

INTRODUCTION

The properties of SCC in the fresh state are what primarily set it apart from CC. Nevertheless, and due to the differences in the quantities of the constituent materials and in the casting process, the durability of SCC is different from that of CC. The differences mentioned derive from the reduction of the ratios between the quantities of course aggregate and mortar and the corresponding increase of the volume of binder and additions and from an adequate control of the aggregate's maximum dimension. Despite these reasons, both technical and economic, and even ecological (use of resources to ensure a sustainable growth) for the use of larger quantities of additions, if these are too high, that can substantially affect the SCC durability, in particular its permeability, which is strongly influenced by its pore structure, that is to say, by its porosity.

Considering that the conditions for the transport processes within the concrete's degradation mechanism strongly depend on its pore structure, it is important to study, in particular, the performance of SCC produced with the use of different combinations of additions, namely FA and LF, regarding the permeability of the SCC structure.

For that purpose, a total of 11 self-compacting batches, each with a volume of 65 litres, were produced using a mixer with a vertical axis: 1 with cement (C) only; 3 with C+FA in 30%, 60% and 70% replacement by volume (f_{ad}); 3 with C+LF in 30%, 60% and 70% f_{ad}; and finally 4 mixes with C+FA+LF in combinations of 10-20%, 20-10%, 20-40% and 40-20% f_{ad}. The evaluation of the porosity of the mixes produced was studied using the permeability coefficient (by water penetration under pressure test), water absorption test (by immersion), capillary water absorption, mercury intrusion porosimetry and the interpretation of images obtained by scanning electron microscopy.

EXPERIMENTAL PROGRAMME – MATERIALS AND MIX PROPORTIONS

The following materials were used: one type of cement complying with NP EN 197-1 (cement type I-42.5 R with specific gravity of 3.14; two mineral additions: fly ash complying with NP EN 450-1 and NP EN 450-2 with specific gravity of 2.30 and limestone filler complying with LNEC specification E 466 with specific gravity of 2.72; two limestone coarse aggregates complying with NP EN 12620, gravel 1 with specific gravity of 2.59, D_{max} of 11 mm and water absorption of 1.46% and gravel 2 with specific gravity of 2.64, D_{max} of 20 mm and water absorption of 0.78%; two siliceous sands complying with NP EN 12620, one coarse (0/4) with specific gravity of 2.55, fineness modulus of 3.70 and water absorption of 1.10% and one fine (0/1) with specific gravity of 2.58, fineness modulus of 2.03 and water absorption of 0.70%; a third-generation high-range/strong water-reducing admixture (S_p) complying with NP EN 934-1 and NP EN 934-2 (a modified polycarboxylic high-range water-reducing admixture in liquid form with a density of 1.07); tap water complying with NP EN 1008. In order to consider all variants of contents used in the mixes and the corresponding analysis of the binary and ternary mixes of FA and LF, the 11 SCC mixes were produced according to the NP EN 206-9. This data is shown in Tab. 1.

Table 1. Mix proportions and fresh properties of SCC

Mix proportions [kg/m^3]	**SCC1 100C**	**SCC2 30LF**	**SCC2 60LF**	**SCC2 70LF**	**SCC3 30FA**	**SCC3 60FA**	**SCC3 70FA**	**SCC4 10FA20LF**	**SCC4 20FA10LF**	**SCC5 20FA40LF**	**SCC5 40FA20LF**
CEM I 42,5 R (C)	707	512	297	222	503	290	218	506	506	297	293
Fly ash (FA)	---	---	---	---	158	318	373	53	106	109	215
Limestone filler (LF)	---	190	386	449	---	---	---	125	63	257	127
Superplasticizer (S_p)	7	5	3	3	5	4	3	5	5	3	3
Water	189	175	168	170	183	180	178	180	180	168	175
Fine aggregate ($0.6Fa_{0/1}+0.4Fa_{0/4}$)	723	747	758	756	735	741	743	740	740	759	748
Corse aggregate ($0.6Ca_1+0.4Ca_2$)	700	700	700	700	700	700	700	700	700	700	700
W/C	0.27	0.34	0.57	0.76	0.36	0.62	0,82	0.36	0.36	0.57	0.60
W/FM	0.27	0.25	0.25	0.25	0.28	0.30	0.30	0.26	0.27	0.25	0.28
Fresh properties											
Slump-flow (SF) [mm]	770	710	710	680	680	670	660	780	740	690	650
V-funnel (t_v) [s]	9.3	10.3	9.1	9.9	7.3	8.4	8.6	9.3	10.8	9.1	10.0
L-box (PL) [-]	0.91	0.89	0.85	0.82	0.84	0.81	0.80	0.91	0.90	0.89	0.83

In order to evaluate only the change in the unitary substitution ratios of cement by mineral additions (f_{ad} by volume), the following conditions were taken into account: the volumetric ratio between mortar and coarse aggregates' content (V_m/V_g=2.625), as well as the absolute volumes of coarse aggregate (V_g=0.268 m^3/m^3) and mortar (V_m=0.702 m^3/m^3), were kept constant; the volumetric ratio between the total powder content, cement and mineral additions, and fine aggregates in the mix (V_p/V_s=0.80) was kept constant; the volumetric ratio between water and fine material content in the mix (V_w/V_p), as well as the percentile ratio in mass between the high-range water reducing admixture (S_p) and the fine material content (S_p/p%) varied depending on the need for water and S_p of each mix in order to obtain the self-compacity parameters according to the works of Nepomuceno and Oliveira [1] and Silva et al. [2]. To ensure that the W/C and W/FM ratios remain as initially established, the properties (water absorption and moisture content) of the aggregates were dully controlled and, when necessary, the mix quantities of water and aggregates were corrected.

EXPERIMENTAL PROGRAMME – TEST METHODS AND SAMPLE PREPARATION

The permeability coefficients were determined resorting to the Valenta [3] equation, using the values for water penetration depth under pressure determined according to the NP EN 12390-8, at 91 days. For each mix studied, 3 saturated cubic moulds with 150 x 150 x 150 mm were used, to which water at 5 bar of pressure was applied during

72±hours. At the end, the moulds were split exactly into two halves, the water penetration front was marked and the maximum depth of penetration measured.

The capillary absorption coefficients were calculated as the first derivative of the equation for the linear regression of the values corresponding to the first 6 hours of capillary water absorption, as a function of the square root of time, determined according to the specification LNEC E 393, at 91 days, in 3 cylindrical moulds with 150 mm diameter and 100 mm height, for each reference. After the adequate time of wet curing (20 ± 2 ºC and RH ≥ 95%), the moulds were stored in a dry chamber at a temperature of 40 ± 5 ºC for 14 days until the test date. Later, the moulds were placed in a tray with water (5 ± 1 mm), dully supported. The water inflow was measured at pre-set times for the specific duration of de test (72 h).

The water absorption (total volume of penetrable pores) was determined according to the procedure described in the specification LNEC E 394, at 91 days, in 3 cubic moulds with 100 x 100 x 100 mm. The values mentioned were obtained from three masses: apparent mass of saturated samples after immersion to constant weight until the increase in mass was less than 0.1%, mass in the air while they were still soaked and mass of dry samples (dried in an oven at 105 ± 5 ºC to constant weight until the increase in mass was less than 0.1%).

The scanning electron microscopy (SEM) with backscattered electron imaging (BSE) of highly polished concrete surfaces allows that pores and anhydrous cement particles can be differentiated from the other phases present by their extreme grey levels. Using the image analysis software *ImageJ*, it was possible to obtain, for each sample, a grey level histogram from which porosity was determined by establishing an arbitrary pore threshold, from the inflection point of the cumulative brightness histogram of the BSE image in the magnification of 500× [4]. This test was performed at 91days.

Mercury intrusion porosimetry tests were conducted using an *AutoPore IV 9500* (*Micromeritics*) porosimeter capable of producing of up to $33x10^3$ psia. By modelling the pores as cylindrical channels, the test pressure was linked to the radius of these cylinders by the Washburn equation [5]. This test was performed at 91 days, in samples produced specifically for that purpose, i.e. without including coarse aggregate.

TESTS RESULTS AND DISCUSSION

The analysis of graph 3 of Fig.1 and Tab. 1 allows concluding that the W/C ratio has a significant influence in the variation of the water absorption (total volume of penetrable pores), confirmed by the similar results between the binary mixes with LF and FA. The same analogy can be made between the results of the binary and ternary mixes. Similarly, Assié [6] mentions that the concrete’s open porosity, evaluated by the water absorption by immersion, is a parameter which is directly linked to the concrete’s mechanical resistance and necessarily to its W/C ratio.

The influence mentioned may be associated to the fact that the concrete's porosity increases with the W/C ratio, i.e. the greater the W/C ratio the higher the volume of the cement matrix's pores will be, necessarily increasing the volume of accessible pores.

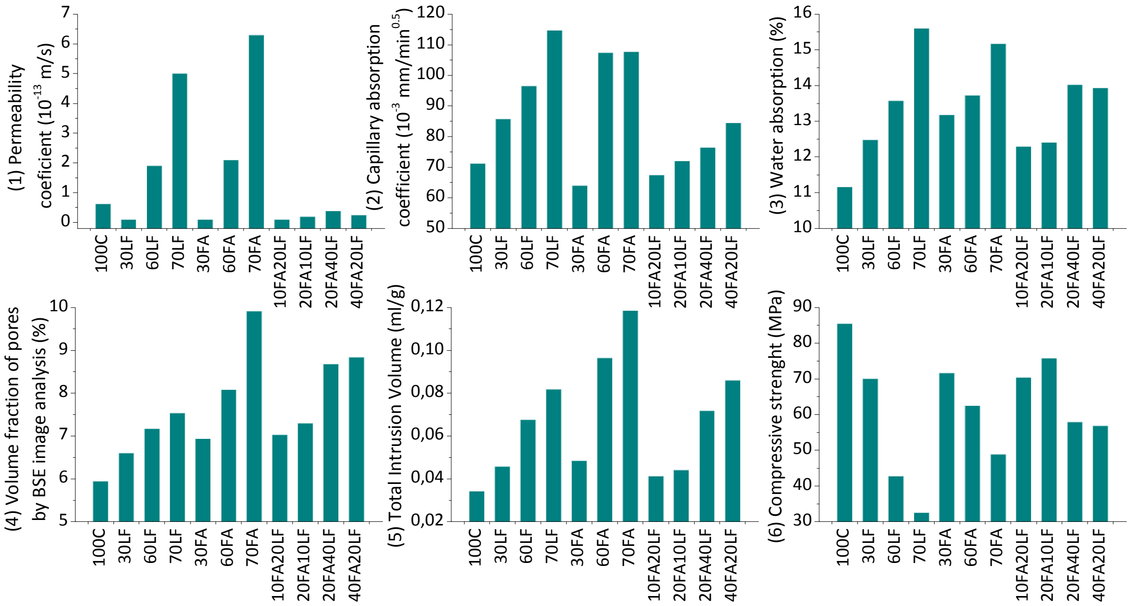
The evaluation of the porosity resorting to the analysis of the images (graph 4 of Fig. 1), considers, in addition to the open porosity, the closed pores. Comparing the porosity determined by water immersion with that obtained by the BSE analysis, it is clear that this last method consistently produces values which are lower than those obtained for immersion. This difference can be explained by the fact that the BSE analysis method only evaluates a relatively small range of pore diameters. This method essentially measures the porosity related to pores whose dimension is larger than 0.2 μm, possibly even also leaving aside pores of larger dimension due, in many cases, to a sample which is inadequate to the magnification used. As for the water immersion method, it covers a broader range of pore diameter, from the micropores to the empty spaces with millimetres, even with the known limitations associated with the difficulty of the water penetration at atmospheric pressure in concrete samples with a denser and more compact microstructure.

In a first analysis, it is possible to see that the capillary absorption (graph 2 of Fig.1) of all the mixes studied increases with the unitary substitution ratios of cement by mineral additions (f_{ad} by volume). As seen in these figures, there is no significant difference for the mixes with f_{ad} of 30% (for those with LF and with FA) when compared to the SCC without additions. The same happens with the ternary mixes, in which the behaviour mentioned is extended in the same way to the mixes with f_{ad} of 60%, which show, from that point of view, a very favourable behaviour. The main reason for the results mentioned (of the ternary mixes) is the synergy effect between FA and LF and consequently the refinement of the microstructure. The results obtained confirm that the cement with LF and FA makes it possible to obtain ternary mixes with improved performances, in comparison to binary mixes containing only cement and/or FA. The best reactivity of the SCC4 and SCC5 mixes could be mainly explained by the acceleration of the cement hydration, related to the presence of LF by nucleation site effect.

The analysis of the values in graph 1 from Fig. 1 shows that the smaller values of the permeability coefficient are obtained for the mixes SCC1.100C, SCC2.30LF, SCC3.30FA and all the ternary mixes, whose results are always lower than 10^{-13} m/s. The remaining mixes show permeability coefficients which are higher than the mentioned value but still with very small absolute values, always lower than 10^{-12} m/s. The results shown indicate a high compacity of the past matrix and a pore system hardly interlinked. In general, the results of the water permeability confirm the results of the water absorption and the capillarity coefficient and, as observed for the capillarity, the ternary mixes show extremely favourable permeability results. Considering the concrete's quality criteria, as a function of its permeability, presented in CEB [7], it can highlighted that all the mixes produced for this work can be considered of good quality, i.e. they have permeability coefficients smaller than 10^{-12} m/s.

According to what would be expected, it is possible to observe in graph 5 of Fig. 1 and in Fig. 2 that, with the increase of the f_{ad} value, an increase in the total porosity is obtained as well as in the corresponding average value of the pores dimension, for all the mixes studied.

Figure 1. Permeability, absorption, porosity, Hg total intrusion volume and compressive strength for all mixes.



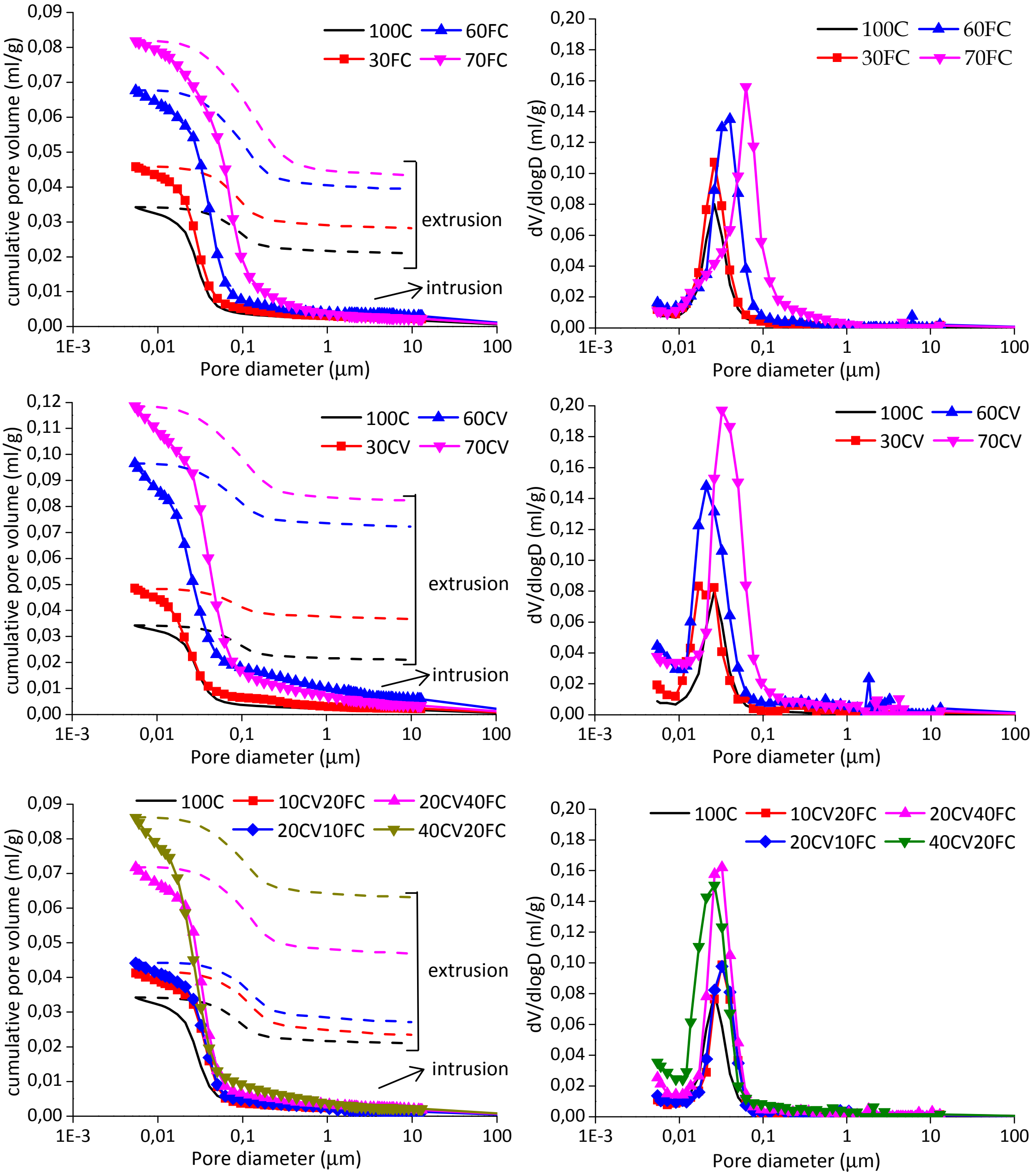
The porosity shown by the binary mixes SCC2 (with LF) is lower than that of the corresponding mixes with FA. The difference between the binary mixes mentioned is greater for higher f_{ad} values (equal to 70%). The porosity values showed by the ternary mixes are coherent with those observed for the binary ones, meaning that, for each of the f_{ad} values, the porosity of the ternary mixes is within the values obtained for the corresponding binary mixes.

From the analysis of the various figures, it is concluded that the distribution of the pores dimension does not follow the trend observed for total porosity. In that sense, the mixes with smaller diameters are the SCC1 (only with cement), the SCC3 (with FA) and the ternary SCC5 (40FA20LF) with the smallest pore dimension. As for the SCC2 mixes (with LF), for f_{ad} of 60 and 70%, they have the maximum pore diameter.

CONCLUDING REMARKS

With the elements shown, it is possible to state that the SCC produced with LF are those that exhibit a smaller total porosity but higher dimension pores, while in the SCC with FA the reverse can be observed, namely a higher porosity but lower dimension pores. The results obtained for the ternary mixes are within those obtained for the binary mixes with equivalent values of f_{ad} but nevertheless with very low water permeability levels, demonstrating, in general, an extremely favourable behaviour towards the studied mechanisms.

Figure 2. Cumulative pore volume and log differential intrusion for all mixes.



These conclusions can also be confirmed by the analysis of the capillary absorption coefficients through where, for the mixes with higher absorption coefficients (indicating a faster absorption), more capillary pores of greater dimension (binary mixes with LF) are expected to be found. As for the mixes with lower absorption coefficients (indicating a slower absorption), more capillary pores of smaller dimension are expected, that is, as initially mentioned, for the SCC3 mixes (with FA), a pore network characterised by a larger number of macropores linked to the exterior and also between themselves by a network of micropores or capillary pores of smaller dimension, relatively to the SCC2 mixes.

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LIST OF REFERENCES

1. Nepomuceno M., and Oliveira L., Parameters for Self-Compacting Concrete Mortar Phase, ACI Materials Journal, SP, Vol. 253, No. 21, July, 2008, pp. 323-340.

2. Silva P. M. S., Brito J. de, Costa J. M., Viability of two new mix design methodologies for SCC, ACI Materials Journal, Vol. 108, No 6, November-December, 2011, pp. 579-588.

3. Valenta O., Durability of Concrete, 2nd RILEM Symposium, in Prague, RILEM Bulletin, Matériaux et Constructions, Vol. 3 Issue 5, 1970 pp. 333–345.

4. Wong H. S., Buenfeld N. R., Head M. K., Estimating transport properties of mortars using image analysis on backscattered electron images, Cement and Concrete Research, Vol. 36, Issue 8, August, 2006, pp. 1556-1566.

5. Boel V., Audenaert K., Schutter G., Pore Size Distribution of Hardened Cement Paste in Self Compacting Concrete, ACI Materials Journal, SP, Vol. 234, No. 11, March, 2006, pp. 167–178.

6. Assié S., Durabilité des bétons autoplaçants, Toulouse, France, L'Institut National des Sciences Appliquées de Toulouse, Octobre, 2004, 254 p, Thèse de doctorat.

7. CEB (Comité Euro-International du Béton), Durable Concrete Structures (2nd Ed.), CEB Design Guide, Edition Thomas Telford, London, England, ISBN: 978-0-7277-3549-2, 1992, 112 p.