PORE-SIZE DETERMINATION FROM PENETRATION TESTS ON CONCRETE WITH N-DECANE

PORENGRÖSSENBESTIMMUNG AUS N-DECAN-EINDRING-VERSUCHEN IN BETON

DETERMINATION DES PORES DU A LA PENETRATION DE N-DECANE EN BETON

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SUMMARY

Absorption and infiltration tests on concrete mixes have been carried out with n-decane. The test results show a good agreement with theoretical predictions. The results indicate that the main parameter on the penetration is the water-cement ratio. Pore sizes which are reached are different for the absorption test and the infiltration test.

ZUSAMMENFASSUNG

Absorptions- und Infiltrationsversuche wurden an verschiedenen Betonen mit n-Decan durchgeführt. Die Ergebnisse haben eine gute Übereinstimmung mit theoretischen Vorhersagen gezeigt. Die Ergebnisse lassen den Schluss zu, dass der Wasserzementwert die maßgebliche Größe für das Eindringverhalten ist. Porengrößen, die erreicht wurden, sind bei Absorptions- und Infiltrationsversuchen verschieden.

RESUME

Des essais d'absorption et d'infiltration ont été réalisés sur des bétons de différentes compositions avec du n-décane. Les résultats montrent une bonne concordance avec des prévisions théoriques. Les résultats indiquent que le paramètre principal pour la pénétration dans le béton est le rapport eau-ciment. Les tailles des pores atteintes sont différentes pour les essais d'absorption et les essais d'infiltration.

KEYWORDS: Concrete, n-decane, penetration, absorption, infiltration, high-performance concrete

MOTIVE

Since several years, tests have been carried out on the penetration behaviour of organic fluids in concrete [1-5]. Two types of tests are being carried out: the capillary suction test and the infiltration test with a certain hydraulic head. The test results show that the capillary suction test satisfies usually technical requirements for the application of the material properties in assessing the behaviour of real structures. However, from a scientific point of view both tests reveal more than the material property only. Comparing the test results of both test methods one can calculate the pore radius of capillary pores in concrete. This will be shown in the following.

EXPERIMENTAL SET UP

For **suction tests,** specimens were placed into the test fluid. The samples rested on glass rods to allow free access of the testing fluid to the inflow surface. The fluid level was approx. 10 mm above the lower end of the specimen. Penetration occurred by capillary forces acting against gravity.

The experimental set-up for **infiltration tests** described in DAfStb guideline [6] was slightly modified. Preliminary tests had proven that the pressure head of 40 (+/- 5) cm specified there was too small to obtain measurable differences to capillary suction tests for high performance concretes. The required external pressure was estimated by calculation from the pore radius distributions of comparable concretes and fixed to 0,2 bar (20 kN/m^2) for all infiltration tests. This pressure was produced by a nitrogen bottle connected to the funnels on the samples by tubes.

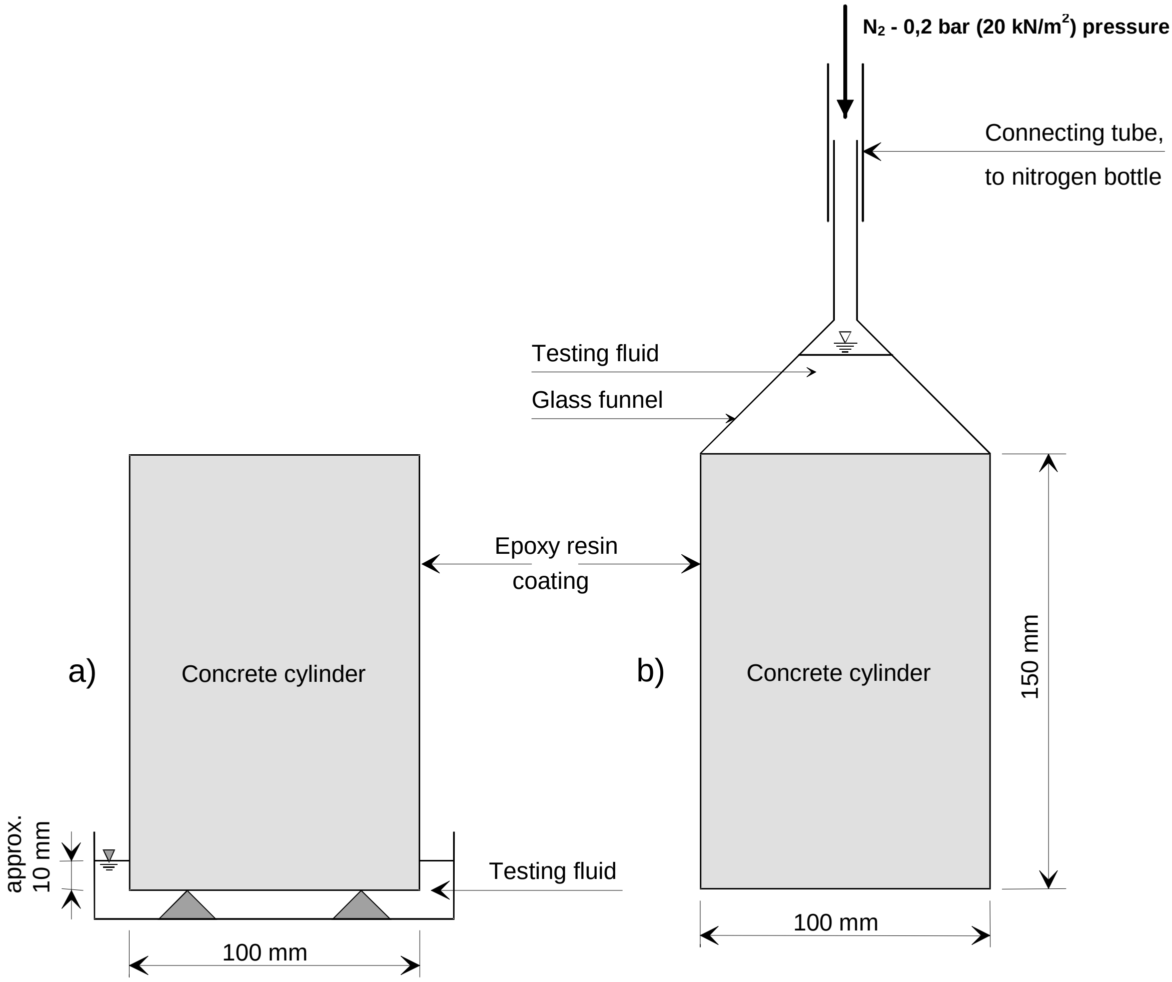

Fig. 1. Experimental set-up: a) suction test, b) infiltration test

Details on specimen preparation, storage and curing are given in [7].

THEORY

Modelling the pores as a single tube, capillary suction is governed by the square root of time law acc. to Eq. (1)

$$e_0 = \left(\frac{\sigma \cos \Theta \, r}{2\eta} t_0 \right)^{1/2} \tag{1}$$

with e_0 = penetration depth of capillary suction test, Θ = contact angle, r = pore radius, η = dynamic viscosity, t_0 = test duration.

If an external pressure p_a is applied the capillary pressure is increased by p_a and Eq. (1) reads

$$e_p = \left(\left(\frac{2\sigma \cos\Theta}{r} + p_a \right) \frac{r^2}{4\eta} t_p \right)^{1/2} \tag{2}$$

with e_p = penetration test of infiltration test, t_p = test duration. When Eqs. (1) and (2) are divided by each other one gets

$$r = \left(\left(\frac{e_p}{e_0} \right)^2 \frac{t_0}{t_p} - 1 \right) \frac{2\sigma \cos\Theta}{p_a} \tag{3}$$

Eq. (3) is an explicit equation for the effective pore radius. The effective pore radius is a fictitious pore size which is related to the single size pore model, i. e. concrete consists only of parallel pores of this size. Of course this model is a simplistic one since pores of many sizes take part in the capillary suction which depends on the pore size distribution of concrete. Studying the effective pore radius one should carry out experiments of short and long duration, during which readings are taken. However, in the following experiments only measurements after 72 hours are taken and the results explain the way of evaluation.

Eq. (3) can be written in a different way when the penetration coefficient $B = e\, t^{-1/2}$ has already been evaluated. Eq. (3) becomes

$$r = \left(\left(\frac{B_p}{B_0} \right)^2 - 1 \right) \frac{2\sigma \cos\Theta}{p_a} \tag{4}$$

with the index 0 referring to the suction test and p to the infiltration test.

A similar relation can be derived if the sorptivity S is used instead of the penetration coefficient B. S is linked to B via the porosity ε

$$S = B \cdot \varepsilon \tag{5}$$

with ε = const. Eq. (4) becomes

$$r = \left(\left(\frac{S_p}{S_0} \right)^2 - 1 \right) \frac{2\sigma \cos\Theta}{p_a} .$$

CONCRETES USED

The composition of the concretes used is given in Table 1. Table 2 shows some relevant properties. The air content has been measured in the fresh state.

Table 1. Composition of concretes

Concr.	Aggregates	Grading	Cement	Type of cement	W_{added}	SF (solid)	RE	SP	$W_{tot.}/(C+SF)$
	[kg/m³]		[kg/m³]		[kg/m³]	[kg/m³]	[kg/m³]	[kg/m³]	-
MR	1905	AB 16	320	CEM I 32,5 R	160	0	0	2.50	0.50
M1	1822	AB 16	338	CEM I 32,5 R	186	0	0	0.80	0.55
M2	1535	AB 2	467	CEM I 32,5 R	257	0	0	0	0.55
M3	1882	AB 32	309	CEM I 32,5 R	170	0	0	0	0.55
M4	1895	U 16	309	CEM I 32,5 R	170	0	0	0	0.55
M5	1677	C 16	405	CEM I 32,5 R	223	0	0	0.50	0.55
M6	1769	AB 16	485	CEM I 42,5 R	150	0	0	9.30	0.32
M7	1762	AB 16	465	CEM I 42,5 R	126	20	0	7.00	0.31
M8	1755	AB 16	445	CEM I 42,5 R	109	40	2.56	10.80	0.32
M9	1748	AB 16	425	CEM I 42,5 R	89	60	2.56	12.00	0.32
M10	1441	AB 2	615	CEM I 42,5 R	153	55	3.59	11.08	0.32
M11	1813	AB 16	338	CEM III/B	186	0	0	0	0.55
M11	1522	AB 2	467	CEM III/B	257	0	0	0	0.55

SF: silica fume RE: retarder SP: plasticizer C: cement W: water

Table 2. Some properties of the concretes tested, mean of three tests

Mix	Properties of fresh concrete			Compressive strength after 28 days		
	workability [1], flow	density of fresh concrete	air content	density of hard. concrete [2]	Compressive strength smallest value	Compressive strength mean value
	[cm]	[kg/dm³]	[%]	[kg/dm³]	[N/mm²]	[N/mm²]
MR/1	41.8	2.34	1.8	2.35	52.3	53.8
MR/2	44.8	2.33	1.2	2.36	51.5	53.2
M1/1	46.5	-	-	2.33	41.8	44.0
M1/2	46.5	2.35	1.5	2.33	45.4	46.2
M2	43.5	2.16	3.6	2.19	41.8	43.1
M3/1	44.5	2.39	0.9	2.37	42.5	43.6
M3/2	46.5	2.39	0.4	2.35	42.2	43.0
M4	46.5	2.40	0.3	2.38	47.5	49.0
M5	43.8	2.28	1.8	2.18	38.2	38.8
M6	43.0	2.40	1.75	2.39	75.2	77.2
M7	48.3	2.37	1.4	2.41	80.9	84.3
M8/1	42.0	2.37	1.5	2.40	88.0	90.5
M8/2	44.8	2.37	0.7	2.40	85.1	86.2
M9	44.0	2.38	1.6	2.38	85.4	88.9
M10	44.5	2.22	2.8	2.24	77.2	78.9
M11/1	47.5	2.36	0.55	2.35	40.9	43.0
M11/2	46.5	2.35	0.44	2.34	45.5	46.0
M12	51.0	2.36	1.4	2.19	33.8	35.6

[1] workability: average diameter of the spread concrete determined by the German flow table test
[2] determined on 100 mm cubes

The density is the dry density after 28 days. The compressive strength has been measured on 100 mm cubes (150 mm for M3, due to the maximum aggregate size of 32 mm) after 1 day kept in the mould, 6 days in the moist room and 21 days in the constant climate room at 20°C and 65% RH.

RESULTS

The results show typically the absorbed amount of liquid as function of time up to 72 hours generated in the capillary suction test and in the infiltration test. Fig. 2 to 4 show on the left the results of the capillary suction test and on the right of the infiltration test. The test results can be presented as a straight line of the absorbed amount vs. square root of time.

There are similar plots for the penetration depth which has been measured by visual observation at the epoxy resin covered surface of the specimens. The results are also shown in Fig. 5 to 7.

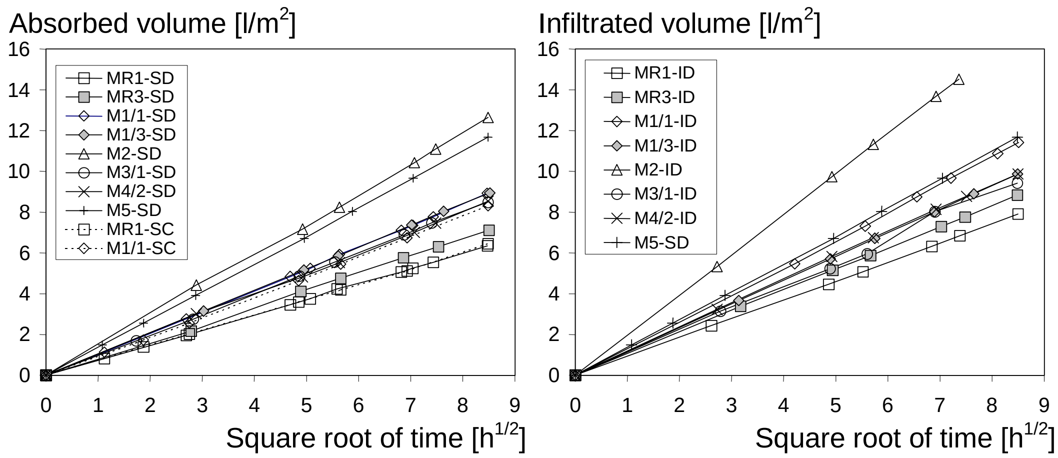

Fig. 2. *Absorbed volume (left) and infiltrated volume (right) as function of square root of time: Portland cement concretes with normal strength*

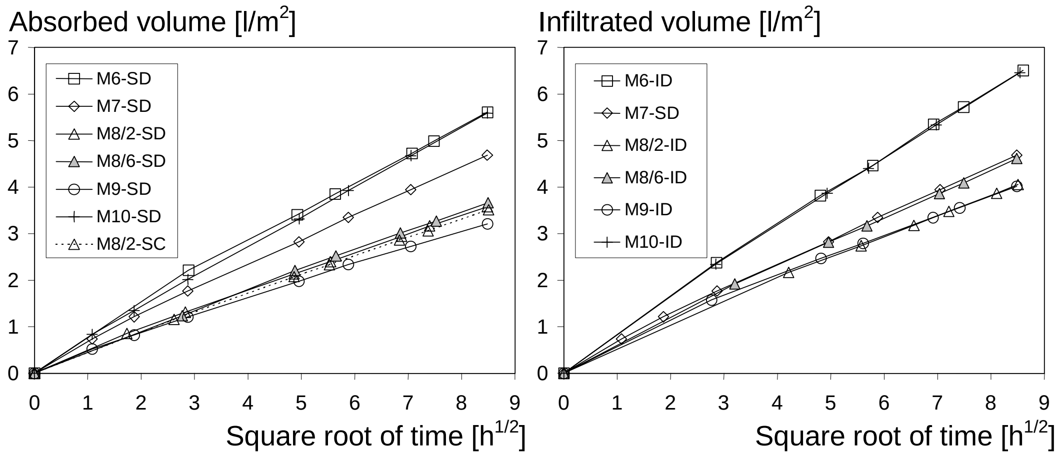

Fig. 3. *Absorbed volume (left) and infiltrated volume (right) as function of square root of time: Portland cement concretes with high strength*

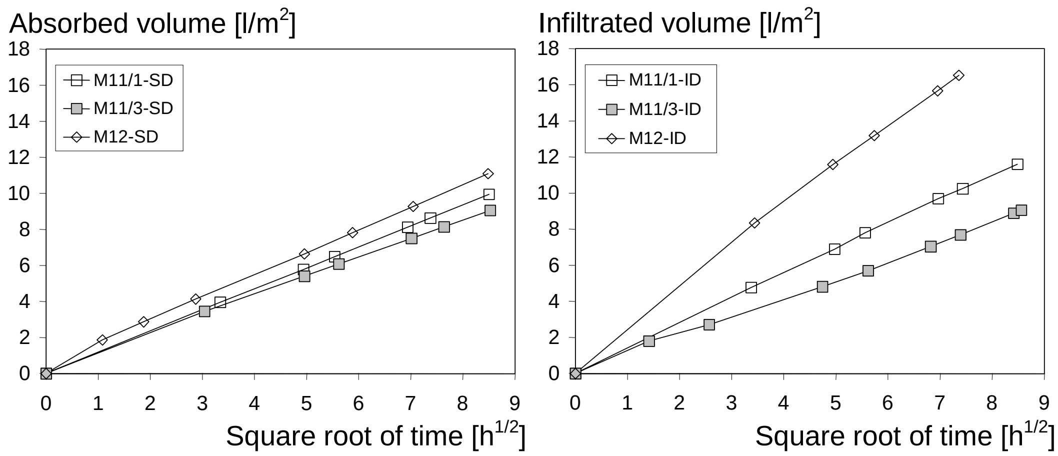

Fig. 4. *Absorbed volume (left) and infiltrated volume (right) as function of square root of time: Blast furnace slag cement concretes*

The properties of n-decane are given in Table 3.

Table 3. Physical values of n-decane at 20°C

Fluid	Formula	Density ρ [kg/dm^3]	Surface tension σ [mN/m]	Dynamic viscosity η [mN.s/m^2]	Ratio $(\sigma/\eta)^{0.5}$ [m$^{0.5}$/s$^{0.5}$]
n-decane	$C_{10}H_{22}$	0.73	23.9	0.88	5.21

With those values the results have been evaluated and are presented in Table 4.

Table 4. Pore parameters calculated from test results with n-decane

Concrete	Sorptivity $l\ m^{-2}\ h^{-1/2}$		penetration coefficient $mm\ h^{-1/2}$		r, from B µm cos θ =		r, from S µm cos θ =	
	S_o	S_p	B_o	B_p	1	2/π	1	2/π
MR	0.750	0.931	10.5	12.1	0.79	0.50	1.29	0.82
M1	1.054	1.342	12.2	14.7	1.10	0.70	1.48	0.94
M2	1.491	1.971	14.1	17.2	1.16	0.74	1.79	1.14
M3	1.001	1.111	12.5	13.7	0.47	0.30	0.55	0.35
M4	1.006	1.162	13.3	15.2	0.71	0.45	0.80	0.51
M5	1.378	1.567	13.7	15.2	0.58	0.37	0.70	0.45
M6	0.661	0.757	10.3	11.0	0.38	0.24	0.74	0.47
M7	0.552	0.626	9.2	10.0	0.44	0.28	0.68	0.43
M8	0.422	0.471	7.2	8.0	0.56	0.36	0.59	0.38
M9	0.378	0.474	7.4	9.0	1.09	0.69	1.37	0.87
M10	0.659	0.756	8.1	8.9	0.47	0.30	0.76	0.48
M11	1.169	1.366	13.5	15.2	0.63	0.40	0.87	0.55
M12	1.307	2.245	11.9	18.5	3.37	2.15	4.66	2.97

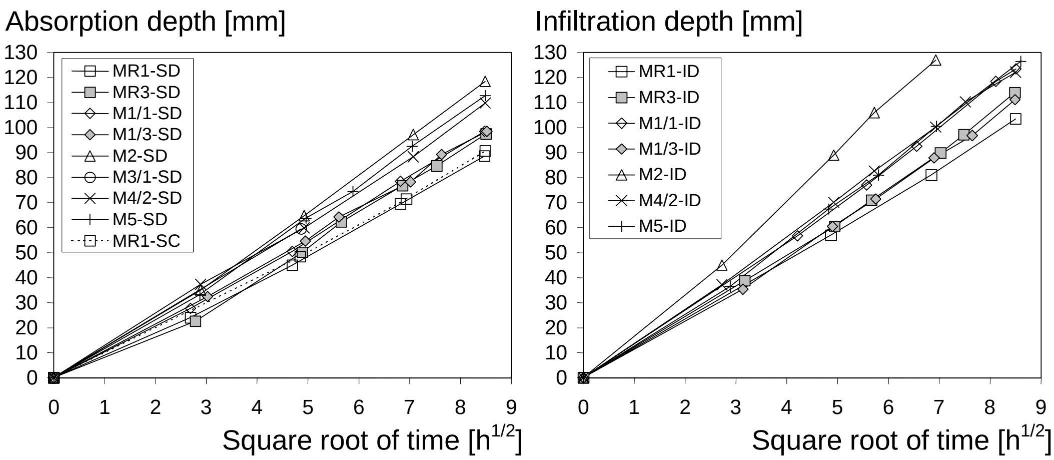

Fig. 5. Absorption depth (left) and infiltration depth (right): Portland cement concretes with normal strength

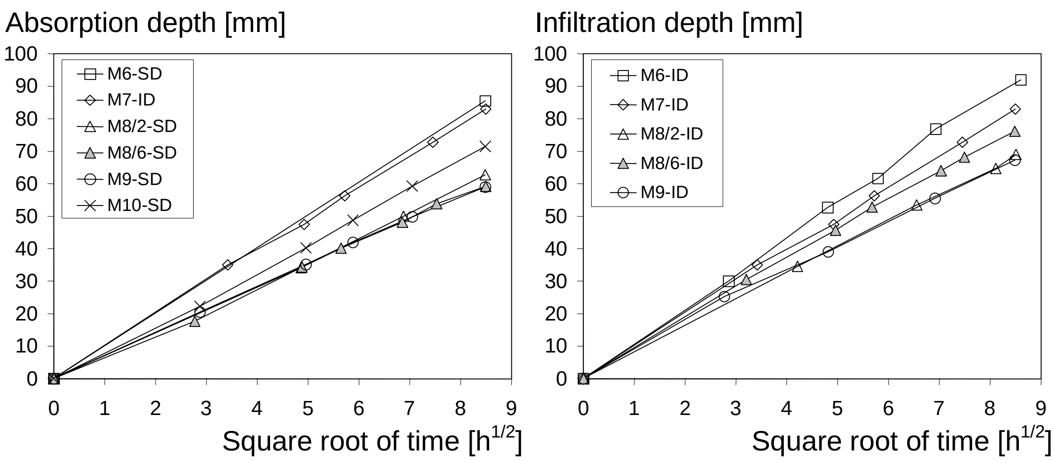

Fig. 6. Absorption depth (left) and infiltration depth (right): Portland cement concretes with high strength

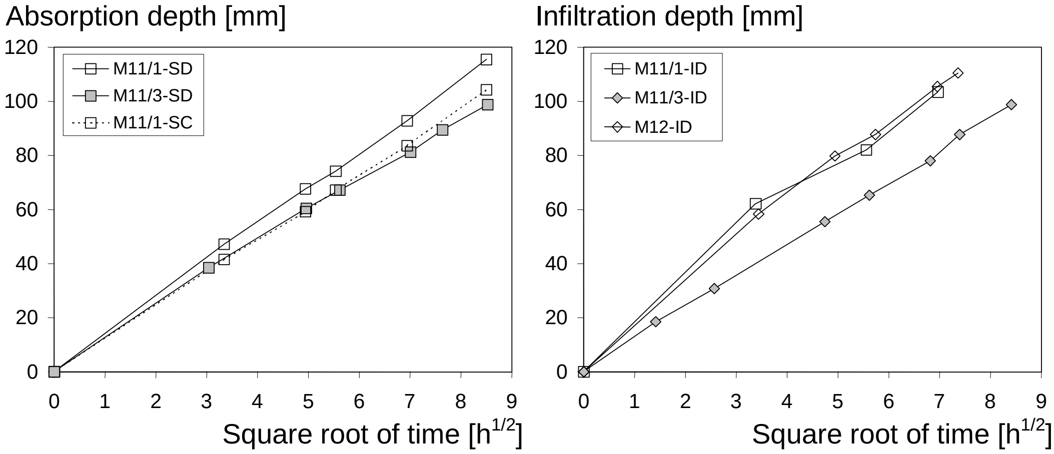

Fig. 7. *Absorption depth (left) and infiltration depth (right): Blast furnace slag cement concretes*

DISCUSSION

The sorption and infiltration tests show in Fig. 2 and 7 an almost perfect straight line in the square root of time plot. A second general feature is that the infiltration results are mostly close. In the sorption results, i.e. the pressure of 20 kPa is not important.

Concrete mixes M1 to M5 are made with a water-cement ratio of 0.55 but with variations in the grading curve. Fig. 2 shows that suction proceeds the fastest with a maximum grain size of two millimetre and a high cement content of 467 kg/m^3 (M2). The same is also true for the infiltration test. Also the mix with fine grading C16 and a cement content of 465 kg/m^3 is fast in suction but not so fast in infiltration. The mixes M1, M3 and M4 vary less because the cement content is rather similar and also grading curves are similar. The concrete mix MR shows the lowest suction and infiltration rates because the water-cement ratio is only 0.50.

Fig. 3 contains the results of the high performance concrete with water-cement ratios of 0.32 and typically a high cement content. Except M10 which has a maximum grain size of 2 mm the others have all 16 mm maximum grain size. There is however a variation in silica fume content. M6 and M10 have the fastest absorption and infiltration. the reason for that is that there is either no silica fume used (M6) or the cement content is very high with 615 kg/m^3 (M10). One should notice that the vertical scale of Fig. 3 is less than half of Fig. 2. All other high performance concrete mixes show smaller absorption and infiltration qualities.

A blast furnace slag cement has been used in the mixes of Fig. 4. The sorptive tests led to results which were similar to those with Portland cement and a water-cement ratio of 0.55 (Fig. 2). The infiltration tests on M12 which has a maximum grain size of 2 mm is different from the others since the infiltration rate is rather high. A similar result has been obtained in Fig. 2 with Portland cement.

The absorption depth and the infiltration depth are rather similar as can be seen from Figs. 5 to 7. The absolute results of MR, M1 to M5 and M11 and M12 are almost the same i. e. the influence of the grading curve is not so strong as in the case of the absorbed fluid volume. However, a closer look to the small variations reveals that the trends of grain size and cement content are the same as in the case of absorbed volume.

Fig. 6 shows the smallest absorption and infiltration depth as has been expected since these concretes are high performance ones.

Table 4 contains the values of the sorptivity and the penetration coefficient. The sorptivity is the quotient of absorbed volume per area divided by the square root of time. The penetration coefficient gives the penetration depth divided by the square root of time. Both quantities characterise physical properties of a material. Both material constants have been derived from sorption and infiltration tests, S_o and S_p and B_o and B_p respectively.

The sorptivity S_o is in the range of 1.0 to 1.49 $l\ m^{-2}\ h^{-1/2}$ for concrete with a water-cement ratio of 0.55. The corresponding value S_p lies in the range of 1.11 to 1.97 $l\ m^{-2}\ h^{-1/2}$. The difference between sorption test and infiltration test is consistent. High performance concretes M6 to M10 show considerably lower values S_o between 0.38 and 0.66 $l\ m^{-2}\ h^{-1/2}$ and S_p between 0.47 and 0.76 $l\ m^{-2}\ h^{-1/2}$. The mixes with blast furnace slag cement fit into the ranges of mixes with Portland cement except M12 in the infiltration test with a high value of 2.24 $l\ m^{-2}\ h^{-1/2}$.

The penetration coefficient B_o ranges between 12.2 and 14.1 $mm\ h^{-1/2}$ for mixes with a water-cement ratio of 0.55. B_p lies between 13.7 and 15.2 $mm\ h^{-1/2}$, i. e. a slight increase due to the pressure of 20 kPa. With a lower water-cement ratio of 0.32 the B_o drops to 7.2 and 10.3 $mm\ h^{-1/2}$ and B_p drops to 8.0 and 11.0 $mm\ h^{-1/2}$. All results are consistent as the influence of grain size, water-cement ratio and pressure are concerned. The B-values for blast furnace slag cement concrete are similar to those of Portland cement concrete.

The effective pore radius r can be calculated from B_o and B_p as shown in Eq. (4) or equivalently also from the sorptivities since penetration coefficient and sorptivity are linked together via the porosity ε (see Eq. (5)). Since the porosity levels out a similar equation occurs for the sorptivity as for the penetration coefficient.

Table 4 contains the results. It can be seen that the pore sizes range between about 0.2 to more than 1.0 μm when the content angle is taken to zero. The values decrease when the cosine of the contact angle is taken as 2/π [3]. The absorbed values increase with the water-cement ratio. A deviation is obvious for M12 with blast furnace slag cement and a high cement content.

The values of r calculated from the sorptivity are always larger than calculated from the penetration coefficient. This feature is certainly due to the fact that the single size tube model is only a rough approximation of reality. It reality the smallest pores have the greatest capillary suction form while the complete filling of the pores are lacking behind. This means that the penetration coefficient should take into account smaller pores than the sorptivity does.

As the absolute values of r are concerned these are rather large compared to pore sizes which are calculated from many intrusion experiments [8]. Obviously, the pores which are reached by the organic fluid are the larger ones and the very small pores are either filled by water of are inaccessible due to other reasons, for instance due to the viscosity of the fluid or of the size of the molecule. This could also mean that the model of the sharp wetting front is questionable. On the other hand, it means that the selection of various fluids could give an impression of the pore sizes which can be detected.

CONCLUSIONS

* The experiments with n-decane have proven the capillary suction law which states that the absorbed volume and the penetration depth are a function of the square root of time.
* The water-cement ratio is the main parameter governing the absorption properties.
* The infiltration test with 20 kPa leads only to a minor increase of the penetration and absorption.

* The pore sizes determined from the absorption test are larger than from the penetration test indicating a different access to pores by different mechanisms.
* Maximum aggregate size and various cement contents lead to different physical properties.

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