CAPILLARY SUCTION IN CONCRETE: EFFECTS OF DRYING PROCEDURE

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ABSTRACT

The effects of drying procedure and initial moisture content on the subsequent capillary suction behaviour of concrete with w/c-ratio of 0.40 has been investigated systematically. The drying temperature was found to be the dominant factor.

Drying at 105 °C produces capillary suction curves with well defined "nick points" and relatively small variations in resistance numbers (m), regardless of the initial moisture levels. Drying at 50 °C, on the other hand, produces very different curve shapes and m-values depending on the predrying time.

In general, more gentle drying procedures produce higher m-values, implying less damage to the original pore structure. It is suggested that more work be undertaken to develope a gentle standard drying procedure for research applications, while for routine control purposes the present 105 °C drying procedure is most practical.

Key words: capillary suction, permeability, predrying

1 INTRODUCTION AND BACKGROUND

The combined capillary suction and pore filling (PF) test has achieved widespread use in Norway as a simple and inexpensive method to make a "first level" characterization of pore structure in hardened concrete. The method gives information about pore size and continuity in the form of the rate of water suction, and allows quantitative determination of two classes of pores: suction porosity (which fill by water suction) and air porosity (which fill by applying external water pressure), corresponding to the porosity determined by optical microscopy.

The suction porosity may be used to estimate the effective w/c-ratio of the hardened concrete, the air porosity as running control of entrained air and/or degree of compaction and, finally, the rate of water suction as a permeability index indicating durability properties.

The water suction stage fills all the empty gel and capillary pores; the name capillary suction is therefore somewhat of a misnomer, since no distinction can be made between gel and capillary pores.

To obtain the water suction rate it is of course neccessary to predry the specimen. As is known, the type of drying procedure greatly affects the pore structure of concrete, thereby greatly influencing the subsequent water suction rate. This makes the choice of drying procedure very important. In recent years drying at 105 °C has become normal in Norway, mainly for practical reasons. A uniform dry state can be achieved in a few days, and the water suction stage generally (except for very low w/c-ratios) has a linear shape vs $\sqrt{\text{time}}$ up to a well-defined "nick-point". Such behaviour can be characterized by a single number which is used as a materials parameter in comparisons. Considerable amounts of data has been collected in routine tests to provide a basis for statements on concrete "quality".

However, there are cases when the method is unable to detect differences in pore structure/continuity known to exist based on other methods. For example, several capillary suction investigations on the effects of curing concrete at elevated temperatures have shown hardly significant effects, while low temperature calorimetry and chloride ion migration tests have demonstrated clear effects. This is the motivation for the present work. It was thought that the harsh treatment of drying at 105 °C alters the pore structure dramatically as well as introduces cracks, thereby eliminating differences in pore structure present before drying.

More gently drying procedures were therefore tried. The problem is that they are more time consuming and generally lead to non-uniform moisture distributions. It was consequently attempted to distinguish between the separate effects of drying temperature, initial moisture content and moisture distribution. In addition, a liquid replacement technique using ethanol was tested.

The work reported here should be taken as a step on the way to establish a variety of capillary suction methods for different purposes: From a quick inexpensive one using drying at 105 °C for routine quality control, to more complicated drying procedures allowing differences in pore structure on a finer scale to be detected.

2 CAPILLARY SUCTION AND PF-TEST

In a capillary suction test the disc shaped specimens are normally exposed to water on one side, e.g. Smeplass /1/. The weight gain is monitored at scheduled times for 4 days. In an ideal case a water absorption curve such as presented in Fig. 1 is obtained. The nick point on the curve represents the end of the gel pore and capillary pore filling. The second slower stage presumable represents water penetration into interlayer positions, and/or filling of the larger air voids.

The first linear part normally follows the general equation of the capillary rise, i.e. linear on a $\sqrt{t}$ scale. Also the second stage is normally linear on the same scale. However, that cannot be theoretically justified. In fact, in the second linear stage the absorbed water content should be related to the volume of the specimen, not to the exposed area as is the first linear stage.

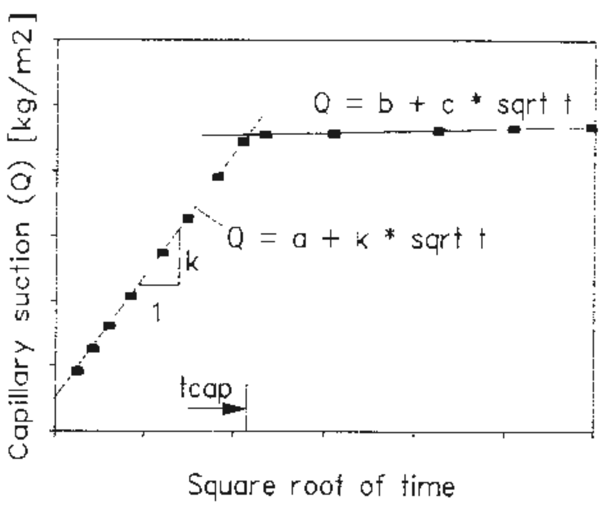


Fig. 1. An ideal capillary suction diagram.

With the help of regression analysis equations for the two linear stages are determined. The intersection point on the X-axis is denoted t_{cap} and it corresponds the time which is needed to fill all the gel and capillary pores. The capillary number, k (kg/m$^2\sqrt{s}$), and the resistance number, m (s/m^2), are determined as follows:

$$k = \frac{dQ}{d(\sqrt{t})}, \quad t < t_{cap} \tag{1}$$

$$m = \frac{t_{cap}}{h^2} \tag{2}$$

where h = thickness of the test specimen, normally 20 mm.

In principle, both the capillary number and the resistance number give the same information. However, the capillary number depends on the cement paste content. Therefore, the resistance number is considered to be a better indicator for the concrete quality.

As mentioned earlier, this is the ideal case. In practice, the relationship is not perfectly linear up to the nick point but deviates downward earlier. Fagerlund /2/ explained the "rounded" transition from the first linear part to the second linear part by a model consisting of parallel capillary tubes with different radii. Later, it has been noticed that the rounded period is longer the lower the water-cement ratio is and, therefore, for some high-strength concretes the two linear parts model does not fit. Also, the ideal model may not fit the case when the initial moisture content is higher than that corresponding to drying at 105 °C. In some cases, a differential curve of the capillary suction gives additional information. In the differential curve the rate of suction is presented as function of time and is calculated by:

$$k'_{(i,i+1)} = \frac{Q_{i+1} - Q_i}{\sqrt{t_{i+1}} - \sqrt{t_i}} \tag{3}$$

where k' = rate of suction [kg/(m$^2\sqrt{s}$)].

A point is plotted on the X-axis at the average value of $\sqrt{t_i}$ and $\sqrt{t_{i+1}}$. In practice, the differential curve gives the momentary capillary number value as function of time. It is used below.

The capillary suction test normally takes four days. Afterwards the specimens are submerged in water for three additional days. Finally, the test specimens are fully saturated by applying water pressure (5 to 10 MPa) over night. If the initial drying was not done at 105 °C, this is done after the pressure saturation to obtain a dry weight. The following weights are recorded:

W_1 weight of oven-dry specimen (105 °C),
W_2 weight after capillary suction and water immersion and
W_3 weight after pressure saturation.

The volume of the test specimen (V) is determined according to the buoyancy principle by weighing the specimen in air and in water. The following porosities can then be calculated /3/:

$$\varepsilon_{total} = \frac{(W_3 - W_1)}{V} \tag{4}$$

$$\varepsilon_{suc} = \frac{(W_2 - W_1)}{V} \tag{5}$$

$$\varepsilon_{air} = \frac{(W_3 - W_2)}{V} \tag{6}$$

where ε_{total} = total porosity,
ε_{suc} = suction porosity and
ε_{air} = air porosity.

The air porosity represents the pore volume that does not fill by capillary suction, and therefore includes air entrained pores as well as larger air voids due to incomplete compaction. Fig. 2 /3/ shows very good correlation between air porosity determined by the present method and the "true" value determined microscopically by point count on thin sections. The capillary suction method gives somewhat higher values in Fig. 2, however, several years experience with both methods does not generally confirm this. Hence, the air porosity is used directly without any correction factor.

The capillary suction - pressure saturation method is in Norway referred to as the PF-method and may be applied also to concrete pieces of irregural shape by determining W_2 after immersion only. Of course, k and m cannot be determined in this simplified version.

PF referrs to the "protective pore ratio" developed by J. Vuorinen in Finland, and is used to assess frost resistance in Finnish Standard SFS 4475 /4/:

$$PF = \frac{\varepsilon_{air}}{\varepsilon_{total}} \tag{7}$$

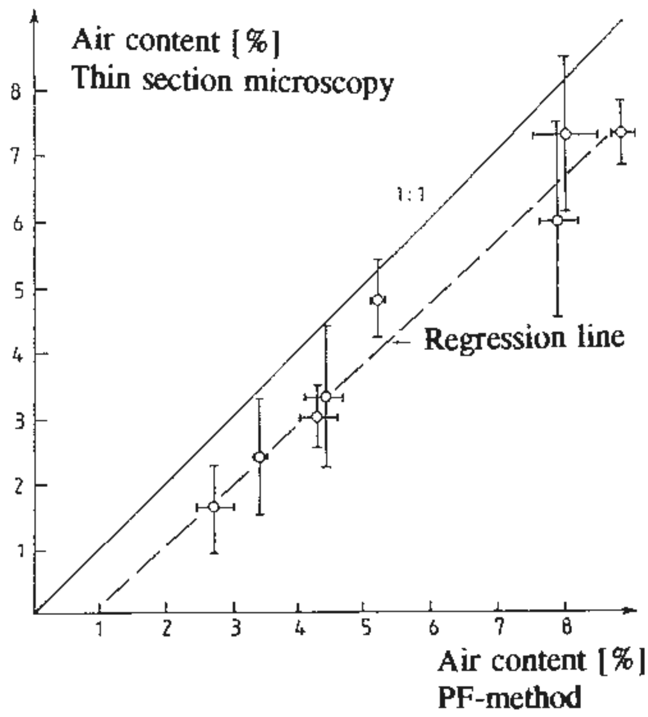


Fig. 2. Air porosity determined by thin section microscopy and by the PF-method /3/. Each data point represents an average of six parallel specimens. Standard deviations are also presented. The coefficient of correlation = 0.98.

3 DRYING OF CONCRETE

The structure of hydrated cement paste is very sensitive to changes in temperature and moisture content. Drying of concrete coarsens its pore structure. The coarsening is presumably due to stresses imposed on the solid matrix due to formation of capillary menisci and reduced thickness of the absorbed water layers. The coarsening effect is largely irreversible. In practice, all drying procedures affect the microstructure. Even mild drying at +20 °C and 58 % RH coarsens the pore structure significantly /5/.

In the test method, the following drying procedures are possibilities:

- drying at 50 °C to a constant weight
- drying at 105 °C to a constant weight
- liquid replacement + drying to a constant weight

Drying at 50 °C to a constant weight has roughly the same effect as the drying in 11 % RH at +20 °C, i.e. the interlayer spaces stay mainly water-filled, and the pores are coated by about one layer of water molecules. This corresponds to a water content of approximately 5 weight % of the competely hydrated cement paste and about 1/3 of the total gel porosity. The drying conditions at 50 °C in an oven are not necessarily constant. The relative humidity can easily vary between 4 and 13 % depending on the humidity of the surrounding air. Drying at 50 °C is also very slow. It may take several weeks to achieve the equilibrium state, depending on the concrete quality and dimensions.

Drying at 105 °C is quite fast. An equilibrium state can normally achieved in few days. On the other hand, the drying procedure is more severe for the concrete microstructure than the drying at 50 °C, and is generally known to lead to microcrack formation.

The stresses induced by the drying procedure, and consequently also the microstructural changes, can be reduced by using a liquid replacement technique (solvent exhange). In the method, the pore water is first replaced with organic fluid, e.g. alcohol. After that concrete is dried using the normal procedure. Alcohol has a surface tension of about 30 % of the value of water. Lower surface tension causes smaller stress during drying. It has been shown /6/ that subsequent evacuation times and temperature of drying are relatively unimportant to the resultant pore size distribution when the liquid replacement was relatively complete.

The rate of liquid replacement is itself a good indicator of the pore structure. The density difference between water and alcohol enables the degree of replacement to be determined by simple weighing. The technique was initially developed for this purpose by Parrot /7/. Feldman /8/ calculated the diffusion coefficient from the replacement data using Fick's Law:

$$W_t / W_\infty = 1.127 \cdot \sqrt{\frac{D \cdot t}{L^2}} \tag{8}$$

where D = diffusivity [m²/s],
t = time [s]
L = half thickness of the specimen [m] and
W_t / W_∞ = degree of the liquid replacement.

Typical diffusivity values for concrete in the liquid replacement test are about 10^{-12} - 10^{-11} m²/s, i.e. in the same range as values obtained for ions such as Na^+, Cl^-, I^-, Cs^+ diffusing through water-saturated ordinary portland cement /8/. This method has the advantage that pore structure properties can be measured with less changes caused by the drying procedure.

4 MATERIALS AND TEST PROCEDURES

4.1 Test concrete

In the present experiments, pre-cast concrete blocks produced by Norwegian Contractors were utililized. Cylinders (d = 104.7 mm) were drilled from the blocks, and then stored in water in the laboratory for more than one year before testing. The mix design, the fresh density and the compressive strength of the concrete are presented in Table 1.

Eight cylinders were used in the tests. The cylinders are denoted by the letters A to H. From each cylinder the first 20 mm (from the casting surface) was removed. From the rest of the cylinders 20 mm thick slices were sawn. Some density differences were noticed between the cylinders. Cylinders B and C were lighter than the others, which affected the test results to some extent. Two separate test series were carried out. In the first, the cylinders A, B, C, D and E were used and in the second F, G and H.

Table 1. Concrete composition, fresh density and compressive strength of the test concrete.

Component	Content [kg/m³]
Cement (HS 65)	417
Silica fume	8
Sand 0-5 mm	917
Coarse aggregate 5-16 mm	938
Water	168
Superplasticizer; Scancem PA(B)	7
Retarder; Scancem R_{20}	1.5
w/(c+s)	0.39-0.40
Density [kg/m³]	2435
Compressive cube strength (28 d) [MPa]	89.0

4.2 Test procedures

The SINTEF/NTH-procedure /9/ was used in the capillary suction and PF-tests, as well as some additional measurements. The capillary suction was measured after 10 min, 30 min, 1 h, 2 h, 4 h, 6 h, 9 h, 12 h, 15 h, 1 d, 2 d, 3 d, and 4 d. The regression analysis was carried out using the data points from 10 min to 6 h for the first linear part and the points from 1 d to 4 d for the second linear part. In some cases, the linear regression analysis could not be applied due to the nonlinear nature of the data.

5 EFFECTS OF DRYING TEMPERATURE AND DRYING TIME

5.1 Results

Three different drying times for both drying temperatures, 50 °C and 105 °C, were used. The capillary suction results are presented in Table 2. The PF-results are presented in Table 3. The discs dried at 50 °C for 7 and 18 days were from the first test series and the others from the second test series. The initial degree of saturation is related to the pressure saturated weight and the oven dry weight (dried at 105 °C for 7 days after the capillary suction test).

Table 2. Average dry densities, initial degrees of saturation (DS) and capillary suction results. The coefficients of variation [%] are given in parentheses. Degree of saturation of 100 % corresponds the moisture content of about 4.6 % of the dry weight of concrete and about 11 vol. %. Six parallel disc specimens.

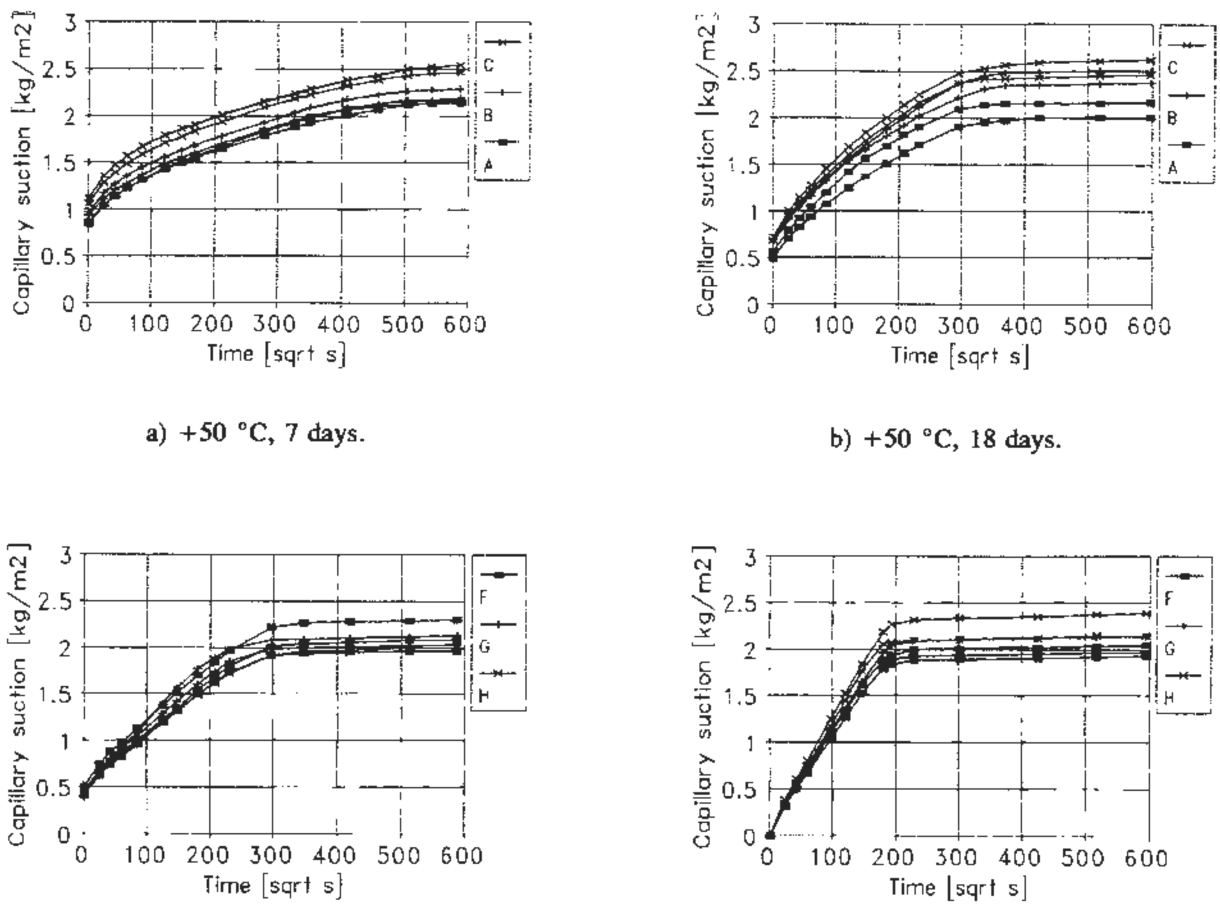
Drying temperature [°C]	Drying time [d]	Dry density [kg/m³]	DS [%]	k [$10^{-2} \cdot$ kg/m² $\sqrt{s}$]	m [$10^{7} \cdot$ s/m²]
50	7	2364	36.8	¤	¤
50	18	2361	24.1	0.62 (7.9)	13.8 (9.3)
50	30	2380	18.6	0.57 (6.1)	16.6 (4.9)
105	1	2375	14.5	0.89 (4.1)	8.1 (3.4)
105	4	2371	1.0	1.09 (6.8)	8.1 (5.3)
105	7	2369	0.1	1.19 (9.1)	7.1 (10.1)

¤ impossible to determine. See Fig. 3.

Table 3. PF-test results. The coefficients of variation [%] are given in parentheses.

Drying temperature [°C]	Drying time [d]	Total porosity [%]	Suction porosity [%]	Air porosity [%]	PF-value [%]
50	7	12.9 (8.0)	11.0 (7.8)	1.1 (19.7)	8.7 (16.0)
50	18	12.3 (10.3)	11.3 (9.7)	1.1 (16.0)	8.5 (9.5)
50	30	11.6 (5.6)	10.5 (5.4)	1.2 (11.0)	9.9 (9.0)
105	1	11.7 (5.6)	10.4 (4.8)	1.3 (12.6)	10.8 (8.3)
105	4	11.9 (7.3)	10.6 (6.2)	1.3 (17.8)	11.0 (11.2)
105	7	12.1 (5.8)	10.8 (5.2)	1.3 (13.2)	10.6 (8.7)

The capillary suction curves for the discs dried at 50 °C and for the discs dried at 105 °C for 4 days are presented in Fig. 3. As can be seen, drying at 50 °C for 7 days did not produce linear relationships and, consequently, regression analysis was not meaningfull. The discs dried at 50 °C for 18 days seems to have a nick point. However, the part before the nick point is far from linear and, therefore, the calculated m and k values are probably meaningless.



a) +50 °C, 7 days.

b) +50 °C, 18 days.

c) +50 °C, 30 days.

d) +105 °C, 4 days.

Fig. 3. Capillary suction curves. The values at time 0 corresponds to the initial moisture content.

5.2 Discussion

It is apparent that drying at 50 °C is very slow. This particular concrete needed at least one month. The discs dried for 18 days at 50 °C seem to have a nick point but the relationship before the nick point was not linear and, therefore, the regression analysis gave unreliable results. The experiments also reveal the effects of the drying temperature on the capillary number and the resistance number values. The resistance number was about 100 % bigger when the specimens were dried at 50 °C compared to those dried at 105 °C. The results of different drying conditions are clearly not directly comparable and, thus, the predrying procedure always has to be specified before results are evaluated.

The discs dried at 50 °C for 30 days achieved close to equilibrium condition. However, drying for only 24 h at 105 °C gave somewhat a lower moisture content, while m was about half as large (Table 2). It is possible that the observed different results are, at least partly, due to the different moisture contents. The effect is further explored in the next section.

The PF-test results were less affected by the drying procedure than the capillary suction results. The air porosity increased slightly with increasing drying temperature. Also the total and suction porosities have the same tendency. The discs dried at 50 °C for 7 and 18 days were from the first test serie and had lower densities. That probably explains the higher suction porosity values.

6 EFFECTS OF INITIAL MOISTURE CONTENT

The preceeding section shows that different drying temperatures give clearly different capillary suction results. However, both the initial moisture contents and distributions were different. The effects of the initial moisture content can be studied by obtaining the same moisture content using both drying temperatures. Three different moisture contents were aimed at 0 %, 25 % and 40 % degree of saturation. The first moisture content is possible to achieve only at 105 °C. The higher initial moisture contents were prepared at 50 °C by controlling the drying time. At 105 °C that is difficult, and so the desired moisture contents were obtained by drying at 105 °C, followed by wetting. After wetting the discs were stored in sealed condition for one week to allow the moisture distribution to even out to some extent.

6.1 Test results

The capillary suction results are presented in Table 4 and the PF-test results in Table 5. The capillary suction curves are shown in Fig. 4. The differential curves gave some additional information and are presented in Fig. 5. All the discs are from the first test series.

Table 4. Average dry densities and capillary suction results. The coefficients of variation [%] are given in parentheses. Six parallel disc specimens.

Drying temperature [°C]	Initial degree of saturation [%]	Dry density [kg/m^3]	k [$10^{-2}\cdot$ kg/m^2 $\sqrt{s}$]	m [$10^7\cdot$ s/m^2]
105	2.6	2355	1.33 (11.7)	5.9 (11.6)
50	24.1	2361	0.62 (7.9)	13.8 (9.3)
105	25.6	2347	1.12 (12.8)	5.1 (9.3)
50	36.8	2364	¤	¤
105	39.7	2362	0.87 (9.2)	5.0 (5.2)

¤ impossible to determine. See Fig. 4.

Table 5. PF-test results. The coefficients of variation [%] are given in parentheses.

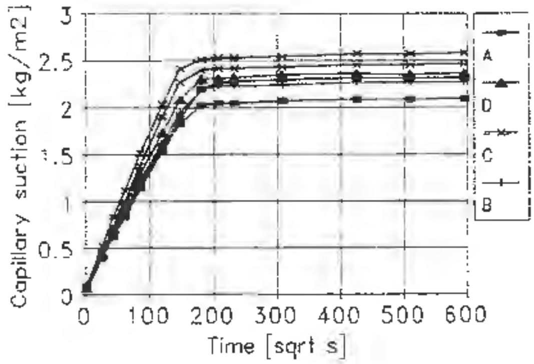
Drying temperature [°C]	Initial degree of saturation [%]	Total porosity [%]	Suction porosity [%]	Air porosity [%]	PF-value [%]
105	2.6	12.6 (7.0)	11.0 (6.7)	1.6 (11.0)	12.9 (7.1)
50	24.1	12.3 (10.3)	11.3 (9.7)	1.1 (16.0)	8.5 (9.5)
105	25.6	12.4 (8.6)	11.2 (8.5)	1.1 (14.4)	9.5 (9.9)
50	36.8	12.9 (8.0)	11.0 (7.8)	1.1 (19.7)	8.7 (16.0)
105	39.7	11.9 (4.9)	10.8 (5.3)	1.1 (10.6)	9.4 (9.8)

6.2 Discussion

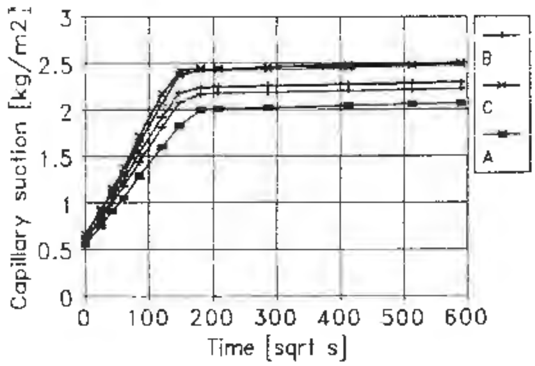
The results show that the drying temperature has a very strong influence on the capillary suction independent of the moisture level. The discs dried at 50 °C had clearly different capillary suction curves than those dried at 105 °C, in spite of having very similar moisture contents. All the discs dried at 105 °C had quite similar suction curves, with marked nick-points, although the moisture contents varied to a great extent. The capillary number obviously depends on the initial moisture content, but the resistance numbers were relatively equal. The shapes of the differential curves suggest that the equalizing for one week after the wetting was not enough. The lower rate of suction in the beginning (Fig. 5.) was probably caused by a higher moisture content near the surface. However, a longer equalizing period probably would have resulted in even more similar suction curves for a given drying temperature.

Why, then does 105 °C drying produce such a highly continuous pore structure (low m-values) compared to 50 °C drying? Clearly, any "blocking" effect of the initial moisture content is not explanation as demonstrated above. Previous work has shown that the pore structure coarsening associated with drying becomes more severe with prolonged drying times at intermediate RH-values /5/ - implying that capillary forces are a main driving force for structural changes. This does not agree with the present results, since at 105 °C the existence of capillary forces is very shortlived. An obvious possibility is that microcracking is greatly increased due to the extreme temperature and moisture gradients during 105 °C drying. A high density of microcracks after 105 °C drying has been demostrated using the FLR (fluorescent liquid replacement) preparation procedure of plane section before microscopic examinations /10/.

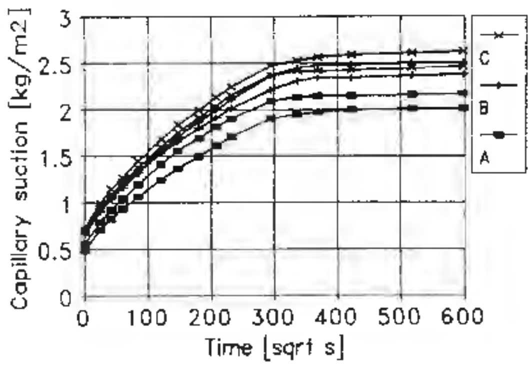
The PF-test results did not significantly depend on the drying temperature or the initial moisture content, as expected. The discs dried at 105 °C to the degree of saturation of 2.6 % had a high air porosity. The reason for that cannot be explained.



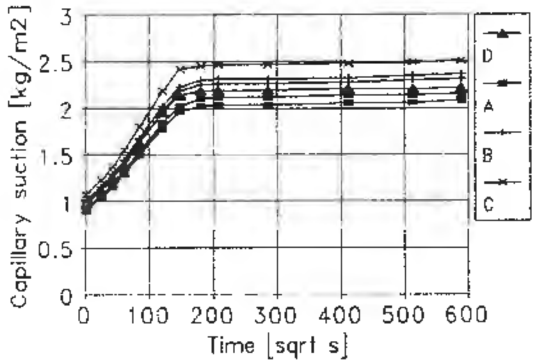
a) +105 °C, 2.6 % DS.



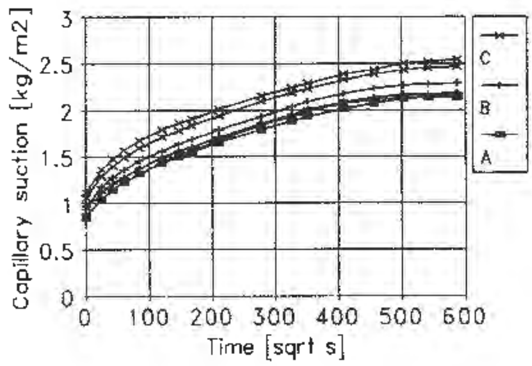
b) +105 °C, 24.1 % DS.



c) +50 °C, 25.6 % DS.

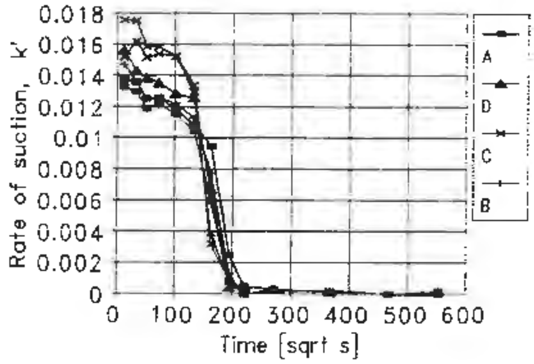


d) +105 °C, 36.8 % DS.

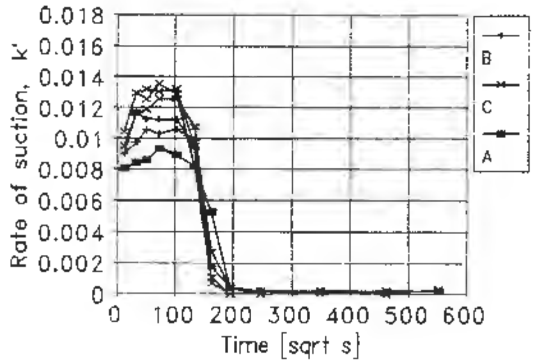


e) +50 °C, 39.7 % DS.

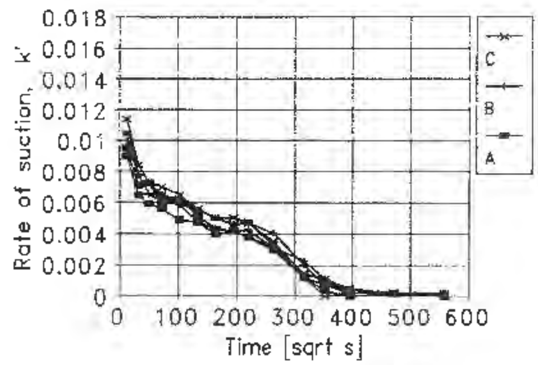
Fig. 4. Capillary suction curves. The values at time 0 corresponds to the initial moisture content. DS = degree of saturation.



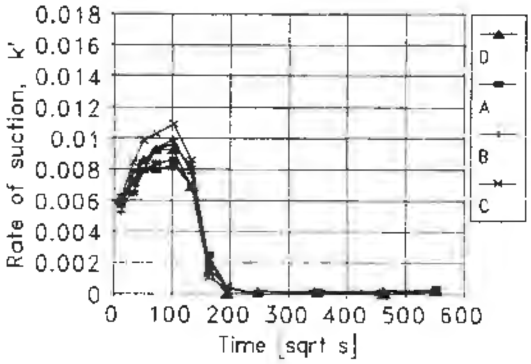
a) +105 °C, 2.6 % DS.



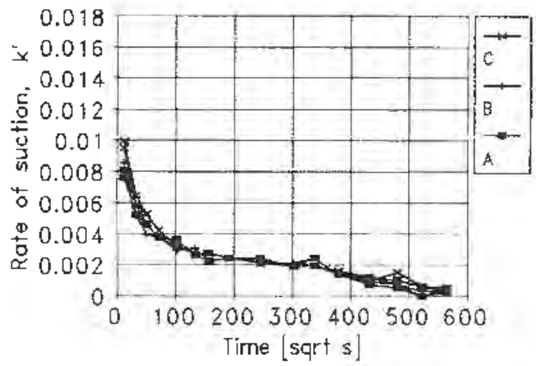
b) +105 °C, 24.1 % DS.



c) +50 °C, 25.6 % DS.



d) +105 °C, 36.8 % DS.



e) +50 °C, 39.7 % DS.

Fig. 5. Differential capillary suction curves. DS = degree of saturation.

7 LIQUID REPLACEMENT

7.1 Rate of replacement

In the liquid replacement test, an ethyl alcohol containing 2 % methylisobutylketone was used. The volume of the alcohol was about six times the volume of the concrete specimens. The degree of the liquid replacement was determined by weighing the specimes periodically and is presented in Fig. 6. The second linear part of the relationship gives the diffusivity D of $2.2 \cdot 10^{-12}$ m^2/s (Equation 8).

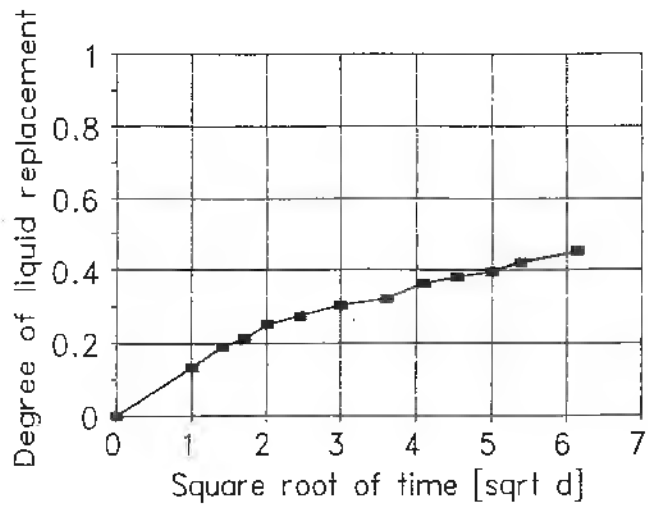


Fig. 6. Degree of liquid replacement as function of square root of time.

7.2 Test results

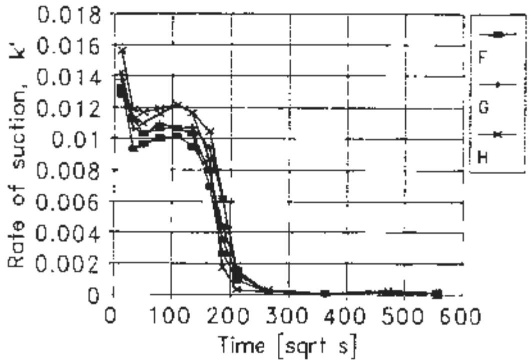
After the liquid replacement the discs were dried at 105 °C for four days. The discs dried directly at 105 °C for four days were used as reference. The liquid replacement caused a slower drying compared to the normal drying procedure. After drying the initial degree of saturation was 2.5 % for the liquid replacement discs and 1.0 % for the reference discs. The capillary suction results are presented in Table 6 and the PF-results in Table 7. The differential curves are shown in Fig. 7.

Table 6. Average dry densities and capillary suction results. The coefficients of variation [%] are given in parentheses. Six parallel disc specimens.

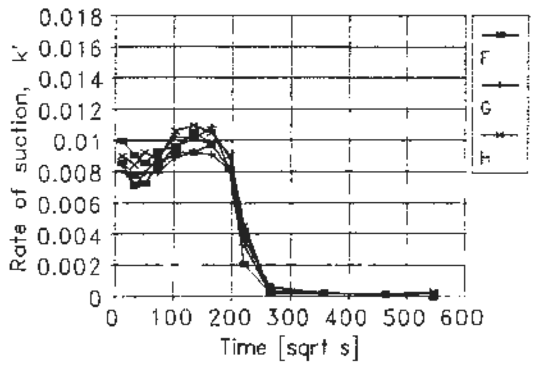
Test	Dry density [kg/m^3]	k [$10^{-2} \cdot kg/m^2 \sqrt{s}$]	m [$10^7 \cdot s/m^2$]
Reference	2371	1.09 (6.8)	8.1 (5.3)
Liquid replacement	2375	0.91 (6.1)	12.0 (7.0)

Table 7. PF-test results. The coefficients of variation [%] are given in parentheses.

Test	Total porosity [%]	Suction porosity [%]	Air porosity [%]	PF-value [%]
Reference	11.9 (7.3)	10.6 (6.2)	1.3 (17.8)	11.0 (11.2)
Liquid replacement	11.5 (4.8)	10.4 (5.0)	1.1 (6.8)	9.7 (6.0)



a) Reference.



b) Liquid replacement.

Fig. 7. Differential capillary suction curves.

7.3 Discussion

The liquid replacement is normally used for very small cement paste or mortar specimens. The specimens used in the capillary suction test are relatively big and, therefore, only a relatively low degree of replacement was achieved. A complete replacement for the particular specimen would have taken more than one year. Although the degree of replacement was only 45 % (Fig. 6), the result was an increase in m by 50 % compared to direct drying at 105 °C (Table 6). More complete replacement would clearly result in even larger m-values. In another experiment a liquid replacement to the same level (45 %) was followed by one week drying at 50 °C and finally 4 days at 105 °C. The result was an m-value of 25, twice as large as that given in Table 6. Thus, with such incomplete liquid replacement, even the final drying procedure plays an important role. Practical use of the liquid replacement technique clearly requires thinner discs. This is acceptable when the goal is to characterize the replacement rate, since more thin discs may be used in parallel to obtain a representative concrete volume. However, it would not be practical to use e.g. 10 mm discs for capillary suction.

The next step in the development work is clearly to acertain that a more gentle drying procedure will increase the ability of the capillary suction method to detect differences in pore structure/continuity caused by e.g. different concrete curing temperatures.

8 CONCLUSIONS

The experiments clearly demonstrate the strong influence of the drying procedure on the capillary suction behaviour of concrete.

The drying temperature is the dominant factor, with 105 °C producing the lowest resistance number, which furthermore is almost independent both on initial moisture content and moisture distribution.

More gentle drying procedures (50 °C or liquid replacement) lead to increased m-values, showing that they produce less pore structure coarsening and less cracking.

It is suggested that alternative, more gentle drying procedures be explored to develope more sensitive capillary suction techniques for use in research work. Such procedures could be combinations, i.e. liquid replacement or drying at 50 °C first, followed by 105 °C to obtain an initial dry state. The present standard method of direct drying at 105 °C definitely has a value for more routine applications.

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