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# Permeability measurement of fresh cement paste

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ABSTRACT

Fresh cement paste permeability is a key parameter to understand the hydro-mechanical behavior of cement-based materials, i.e., rheological properties and static stability. However, its permeability measurement is not easy to assess. The porous medium is not rigid and tends to change due to hydration kinetics. Two measurement methods, with 70 mm and 20 mm initial height specimens respectively, are presented and compared in this paper. The first uses a basic cell of soil permeability measurement and consists of simultaneous consolidation and percolation tests. The second uses a displacement-controlled oedometer cell equipped with pore water pressure transducers, and consists in inducing consolidation to a given void ratio first and, consecutively, in accurately measuring the permeability. A good correlation of results is observed. A comparison with theoretical models confirms that, from one fitted parameter relative to particle characteristics, a relationship between permeability and void ratio can be established.



## 1. Introduction

Hydraulic conductivity is commonly called permeability coefficient ($m\,s^{-1}$) or "permeability" for short, when 20 °C water is considered as the percolating fluid. It is a key parameter to understand the hydro-mechanical behavior of fresh cement-based materials [1]. For instance, bleeding and segregation strongly depend on the paste permeability [2]. Moreover, dynamic or quasi-static stability of cement based materials controls casting, pumping [3] or forming processes like extrusion [4]. The homogeneity of the material is thus directly linked to water conductivity properties, i.e. the ability of water to move through the porous matrix made of solid particles.

Consequently, concrete permeability characterizes the ability of the concrete material to remain homogeneous during the forming process. The lower the permeability, the better the stability. This statement is valid when the material flows under its own weight or when the flow is ensured by external solicitation (pumping and extrusion). The permeability is therefore a key parameter, on which the homogeneity of the hardened material also depends. Criteria based on water permeability about extrusion ability [4] and bleeding in concrete [5] exist in the literature. Moreover, the evolution of pressure exerted by concrete on casting formwork is largely influenced by the concrete permeability [6]. During setting, permeability also should act on the rate of capillary pressure build-up and consequently on the plastic shrinkage [7].

Concrete permeability depends on the particles' nature (size distribution and specific surface area) and also on the particles' packing [8]. Specific surface influences the water adsorption forces that govern the attraction and friction forces. The particles' assembly which defines the porous solid network determines the flow path and velocity. As a consequence, water permeability theoretically depends on the particle size distribution and on packing inside the mix (i.e. Water to Cement mass ratio, W/C, in case of homogeneous cement paste).

Water conductivity measurements have been mostly developed in soil mechanics. They can be classified in two types:– Direct methods based on Darcy's law. The percolation velocity is measured under a given hydraulic head [6]. This method consists in measuring, with a constant hydraulic head, the water flow through a sample for which the mixing and shearing history is known.– Indirect methods based on the soils consolidation theory. One-dimensional compression is applied to a drained sample inside an oedometer cell (commonly called the consolidation test).

Fresh cement-based materials do not exhibit a stable porous network when they are submitted to pressure gradients or water flows. Moreover, cement hydration has a significant effect on the permeability when setting occurs, usually between the second and third hydration hours [1]. As a result, the permeability measurement of fresh cement needs specific experimental protocols.

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In this paper, two experimental methods are presented to measure the permeability of cement paste. This is a first step for the determination of concrete permeability.

The first method consists in both percolation and consolidation tests under a constant hydraulic head. A common soil permeability cell has been used in order to perform measurements on a 100-mm diameter cylindrical sample drained on only one face. The consolidation kinetic is analyzed using the cumulative volume rate of percolated water, flowing through the sample. According to the consolidation theory, a finite differences method can be used to determine precisely the average coefficient of consolidation and permeability over the sample height and elapsed testing time.

The second method consists in inducing water percolation through a cement paste layer while the instantaneous mixing proportion is monitored. The developed permeameter is set up on a compression machine which controls and monitors the sample volume. After compression, interstitial overpressure dissipates and water flows out of the sample. When water pressure dissipation ends, the tested sample has a steady and homogeneous W/C ratio and permeability measurement stage is performed. From one sample, several W/C ratios can be studied successively before hydration kinetic influences the permeability measurement.

The measurements according to these two methods should allow the validation of results and test procedures (hydraulic gradient, initial consolidation, test duration, sample geometry, etc.). Some experimental protocols can then be defined according the expected accuracy of measurements. Also, the commonly used computation assumptions (homogeneous material in the sample and constant permeability during the whole testing time) are analyzed and discussed.

If the results corroborate, the permeability should be analyzed according to the void ratio (i.e., void to solid volume ratio) also relative to the compacity or the water-to-cement mass ratio of the cement paste.

## 2. Materials and procedure

### 2.1. Material

The water-to-cement mass ratio (W/C) ranges from 0.3 to 0.4 to cover the range of currently used concrete. A “CEM I/52.5 N CE CP2 NF” cement of specific density 3.15 was used. The characteristics of mix components are summed up in Table 1.

The largest grain size is lower than 100 μm. The average grain diameter is approximately 15 μm. This cement comes from the cement factory of St Pierre La Cour (Lafarge, France).

### 2.2. Porosity or void ratio

The tested cement pastes are considered as fully saturated. The weight-to-volume ratios of the pastes are checked before the filtration tests. In soil mechanics, the soil porosity is commonly expressed as the void ratio, e (i.e., void-to-solid volume ratio). In saturated condition, the void volume is assumed to be equal to the water or liquid volume. It can be written as a function of the W/C ratio, and the specific unit weights of cement particles $\gamma_c$ (kN m$^{-3}$) and of water at 20 °C $\gamma_w$ (kN m$^{-3}$).

$$e = \frac{W}{C}\frac{\gamma_c}{\gamma_w}. \tag{1}$$

**Table 1**
Physical characteristics of tested material.

| $\mu_w$ | $\rho_w$ | $\rho_s$ | Specific surface |
|---|---|---|---|
| Pa s | kg m$^{-3}$ | kg m$^{-3}$ | m$^2$ kg$^{-1}$ |
| 0.001 | 1000 | 3150 | 339 |

In this study, the specimen height, h, is always lower than its initial $h_0$. This is due to the consolidation phenomenon which squeezes out a part of the water initially contained in the material, for a given initial W/C ratio. Assuming that the dry mass of the specimen, C (kg), is constant (i.e. only the fluid phase can flow through the filter grid system), a variation Δh of the initial height of the specimen, $h_0$ (m), induces a W/C mass ratio variation Δ(W/C) and a void ratio variation, Δe, as follows:

$$\Delta\left(\frac{W}{C}\right) = \Delta h \ \frac{S\,\rho_w}{C} \tag{2}$$

where S is the section of specimen (m$^2$), and $\rho_w$ the water unit mass (kg m$^{-3}$).

$$\Delta e = \frac{\Delta h}{h_0}(1 + e_0). \tag{3}$$

### 2.3. Procedures

A planetary Hobart mixer was used for the mix. It provides sufficiently high shear rates for small batches (5 l). Water was added while the cement was mixed. Once the cement was moistened, the hydration time started, and a high rotation speed was applied during 5 min. Temperature was kept constant at 20 ± 2 °C during the experimental tests.

$H_w$ (m), the pore water head or hydraulic head is also called total head or piezometric height in soil literature. The permeability, K (m s$^{-1}$) is basically deduced from the measured percolating flow rate Q (m$^3$ s$^{-1}$) through a specimen section S (m$^2$) from Darcy's relationship:

$$\frac{Q}{S} = -K\frac{dH_w}{dz} = Ki \tag{4}$$

The vertical hydraulic gradient, i ($i = -dH_w/dz$), applied during permeability measurements could have a significant influence on the measured parameter: when i increases, a better measurement accuracy is obtained since the percolation flow increases, however the sample can be changed noticeably due to the leaching effect or due to the increase in the induced effective stresses.

Normative recommendations in soil mechanics suggest mean hydraulic gradients according to the estimated permeability of the material: 20 between $10^{-6}$ m s$^{-1}$ and $10^{-8}$ m s$^{-1}$ and 50 between $10^{-8}$ m s$^{-1}$ and $10^{-10}$ m s$^{-1}$. In this study, i ranges from 15 to 130 but most of the presented results have been measured with hydraulic gradients between 20 and 50.

## 3. Filtration test

This first method consists in both percolation and consolidation tests under constant hydraulic head using a simple device. It could easily be performed on site if needed, and many tests could simultaneously be conducted and compared. The volume of tested samples can be up to 600 cm$^3$ and could include aggregates. The presented results are limited to cement paste, but the cell volume would be large enough to be representative if further studies are conducted on mortar or concrete.

### 3.1. Experimental setup and procedure

A 102 mm diameter steel cell of a common soil permeameter is used. A constant hydraulic head is applied. The setup is described in Fig. 1. A perforated plate and two paper filters are placed at the bottom and the top of the sample. The perforated plates homogenize the pressure at the boundary (top and bottom) surface. The paper filters

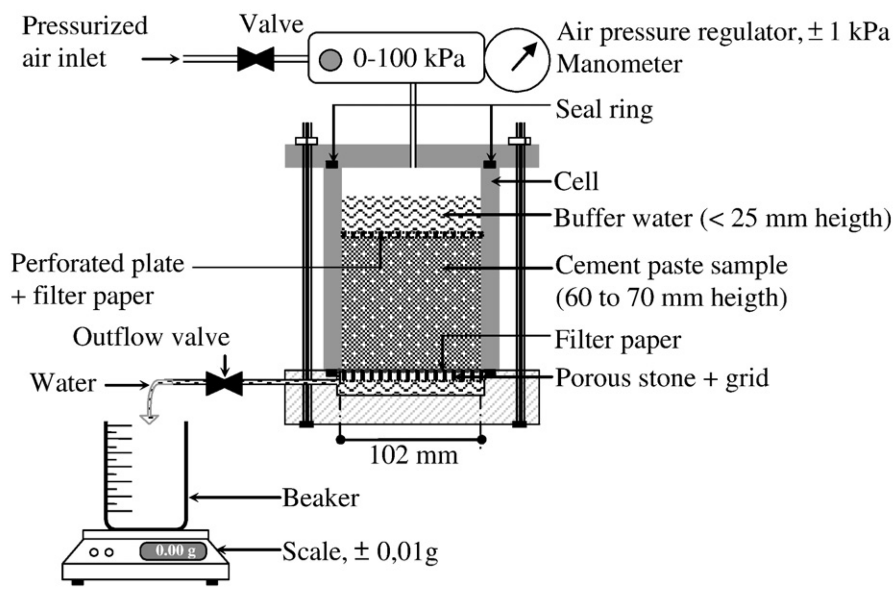


**Fig. 1.** Filtration experimental setup.

ensure that only water flows outside the sample and no cement particles mix in the buffer water above. The sample weight is adjusted when it is poured into the cell in order to control its volume. The height of the upper plate is measured according to the free height remaining at the top of the cell, within 1 mm accuracy, before and after the test, in order to check the sample density and deduce the settlement which occurred during testing.

The sub plate of the cell and outlet pipe is filled with water in order to measure the water flow at the beginning of the test (i.e. as soon as the valve is opened). The weight of the percolated water is recorded over the entire test period.

The cell is covered by a top plate including an inlet pressure connector. If the applied pressure is lower than 3 m, the constant hydraulic head is applied by means of a Mariotte bottle with a siphon height measured with 2 cm accuracy. Else, it is connected to an air pressure regulator of 1 kPa accuracy. In that case, 180 g of water is added above the top perforated plate in order to ensure that the cement paste remains water-saturated during the entire test (see Fig. 1).

The inlet valve pressure on the top of the cell is first opened while the outflow valve remains closed. No flow occurs, and the hydraulic head in the whole sample is supposed to be equal to the controlled inlet head pressure. After 15 min hydration time, the test begins: the outlet valve is opened, and the percolating time, set at $t_0$, starts.

The measured parameter is the evolution over time of the percolated water volume. Its mass is measured within ±0.1 g accuracy and is recorded using a data acquisition system at 2 s time interval during the testing period.

### 3.2. Considered theory

Theoretically, the pressure head at the bottom of the sample drops instantaneously toward zero when the outlet valve is opened, as shown in Fig. 2. A strong hydraulic gradient occurs in the bottom part of the sample and a downward water flow occurs. The hydraulic gradient tends to be constant throughout the sample over the percolating time. If the sample is regarded as a rigid porous matrix and the compressibility of water is not considered, the hydraulic gradient could be assumed to be constant as soon as the test begins. However, in the case of fresh cement paste, the solid matrix is soft.

It could then be assumed that fine particles constitute a porous material with elastic properties, and the void of the elastic skeleton is filled with water as presumed in the soil consolidation theory [9]. Stress applied to this system will produce a gradual settlement, depending on the rate at which water is being squeezed out of the void. In the case under scrutiny, significant time is needed to reach a constant hydraulic gradient along a sample a few centimeters in height and consequently, a constant outflow rate (see Fig. 3). A poro-elastic theory as proposed by Biot [10,11], should then be considered in the case of fresh cement paste.

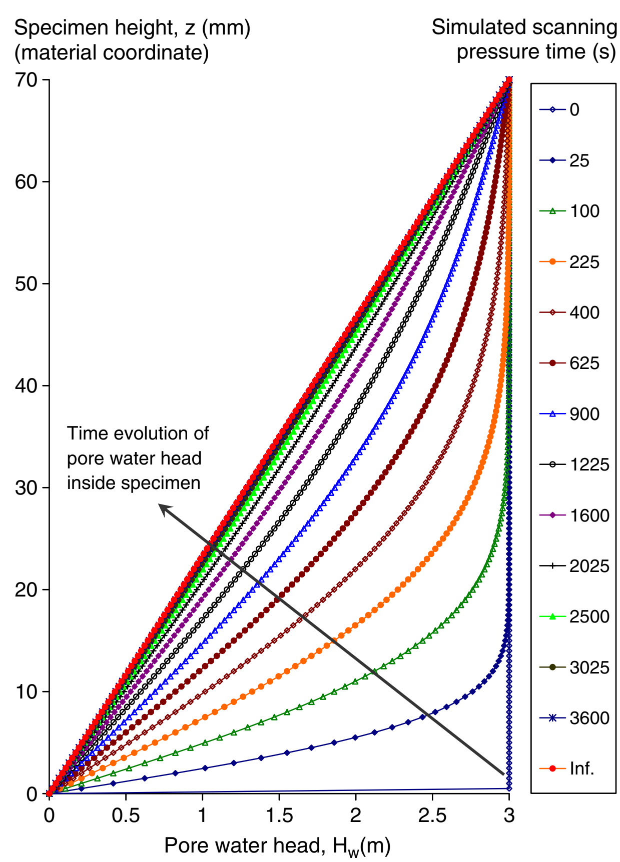


**Fig. 2.** Simulated evolution of pore water head versus vertical material coordinate during percolation inside a 70 mm initial height specimen with initial W/C = 0.3.

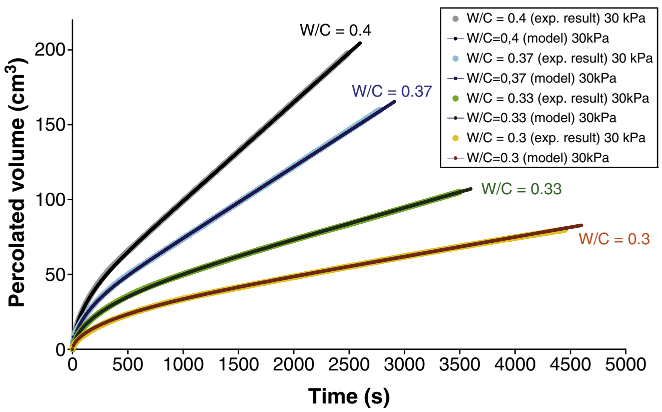


**Fig. 3.** Percolated water volume versus time in case of 70 mm initial height specimen with 30 kPa differential pressure: experimental and fitted data.

In this study, the treatment is relative to a constant applied load and to the one-dimensional problem. The classical soil consolidation theory according to Terzaghi [9] is then applied to simulate the sample behavior and the cumulated outlet volume of water. The following basic properties have been assumed: –homogeneity of the material, –verticality of the induced strains and water flow, –linearity of stress–strain relation, –small strains, –the water contained in the pores is incompressible, and –the water flows through the porous skeleton according to Darcy's law.

According to this theory [9,10], the small-strain coefficient of consolidation $C_v$ ($m^2 s^{-1}$) and the permeability are assumed to be constant since they are considered as the average coefficients throughout the sample and over the testing period. It should be noted that the instantaneous decrease in permeability due to the slight decrease in void ratio induced by consolidation itself is only partially taken into account in this theory. The relative decrease in permeability during the test is assumed proportional to the settlement-induced strain while it should be, according to the Poiseuille theory, relative to the square of void ratio variation as defined by Kozeny Carman [12] (see Section 5.1).

Due to the mixing process, the cement paste cannot be fully saturated. It is carefully poured in the permeameter cell and vibrated in order to avoid entrapped air. It could be assumed that micro air bubbles dispatched within the specimen lead to higher compressibility of the fluid filling the pores, and may induce a slight overestimation of K during the consolidation phase.

The evolution of the excess pore water pressure, u* (kPa), inside the specimen, according to time, t, and the vertical material coordinate (i.e. Lagrangian coordinate), z, can be then written as follows:

$$\frac{\partial u*}{\partial t} = C_V \frac{\partial^2 u*}{\partial z^2}. \tag{5}$$

The total pore water head, $H_w$, is linearly dependant from u: $H_w = H_0 + u^* \gamma_w$ where $H_0$ (m) is a constant relative to the referential hydrostatic head (usually the outlet water head level) and $\gamma_w$ ($kN\,m^{-3}$) is the unit weight of water. Therefore, Eq. (5) can also be written using the variable $H_w$ instead of $u^*$.

The evolution of $H_w$ along the sample, as shown in Fig. 2, can then be computed according to Eq. (5) and the initial boundary conditions: at $t_0$, $H_w = 0$ at the bottom of the cell, since the permeability of the filter and perforated plate is assumed to be much greater than that of the specimen, while u, in the rest of the sample, is equal to the constant applied pressure on the top of the cell, i.e., $H_w = 3$ m (water head pressure) in case of results shown. For $t > t_0$, the values of u at the bottom and the top of the specimen are kept constant.

### 3.3. Simulation and parameters identification

According to the considered theory, a constant factor makes it possible to determine K ($m\,s^{-1}$) if $C_V$ is known:

$$K = \frac{C_V \gamma_w}{E_o} \tag{6}$$

where $E_o$ (kPa) is the oedometric modulus of the material and $\gamma_w$ ($kN\,m^{-3}$) the unit weight of water. $E_o$, is actually a mean value, which is assumed to be constant during the consolidation step. It is also defined as the reverse of the coefficient of compressibility, $m_v^{-1}$, in soil literature [13,14].

Implicit finite differences method, consisting a 0.5 mm thickness discretization, Δz, and 1 s fraction time, Δt, has been used to solve the Eq. (5) in the vertical direction (according to z) and compute the profile evolution of water head pressure along specimens as shown in Fig. 2. From the simulated pressure profiles, Darcy's relationship allows us to deduce, during the percolating time step $n$, the water volume $\varsigma_n^1$ percolating over the section S of the specimen and through the bottom layer (numbered 1) due to the local hydraulic gradient, $i_n^1$. At percolating time $t = n$, the cumulated outflow volume, $V_n^{out}$, is deduced from the sum of $\varsigma_n^1$ as follow:

$$V_n^{out} = \sum_{t=1}^{n} \varsigma_n^1 = KS\Delta t \sum_{t=1}^{n} i_n^1 \tag{7}$$

From this relation, the experimental outflow volumes can be fitted according to 2 parameters among $C_V$, $E_o$ or K.

To improve the model in a further step, the semi implicit (Crank–Nicolson) finite differences method has been selected in order to take into account the variations of $C_V$ and/or K according to the variation of the void ratio, e, due to consolidation during percolation, if needed. However, if the water-to-cement ratio ranges between 0.3 and 0.4, Fig. 3 shows a good fitting of the model with the experimental results, which allows us to determine precisely only one mean value of K and of $C_V$, over the global sample height and elapsed testing time. Table 2 sums up the range of fitted values of K and $C_V$. The permeability clearly increases when W/C increases.

This study focuses on permeability, the same trend could however be observed in case of $C_V$, but in this case, this parameter represents a mean value along a specimen. The effective stress applied is not uniform. Even at the end of the consolidation step, it remains greater in the lower part, decreasing linearly with height z. A void ratio gradient is induced inside the specimen. $C_V$ and $E_o$ should be locally or specifically studied to establish a relationship between W/C and $C_V$ or $E_o$. Moreover, in this test configuration, no significant trend has been observed about $E_o$ versus W/C.

Results show that fresh cement paste, before 1 h of hydration, behaves as consolidating material when external effective stress is applied. The consolidating theory could therefore predict the paste behavior correctly, when W/C ranges from 0.3 to 0.4, since the material is similar to fine saturated soil.

**Table 2**
Range of fitted parameters and measured specimen settlement in case of 30 kPa inlet pressure.

| Initial water to cement ratio, W/C | Hydraulic conductivity, K ($m\,s^{-1}$) | Consolidation coefficient, $C_V$ ($m^2 s^{-1}$) | Relative settlement $\Delta h/h_0$ |
|---|---|---|---|
| 0.3 | $0.32 \cdot 10^{-7}$ | $0.65 \cdot 10^{-6}$ | $6.7 \pm 0.4\%$ |
| 0.4 | $1.88 \cdot 10^{-7}$ | $3 \cdot 10^{-6}$ | $10.3 \pm 1\%$ |

### 3.4. Data analysis

The filtration induces a higher effective stress gradient in the lower part of the sample (see Section 3.2). A part of the water contained in the paste is then squeezed out, due to the consolidation effect, inducing an overflow during the first period of the test as shown in Figs. 3 and 4. When the overpressure dissipates, the hydraulic gradient along the specimen becomes linear (see Fig. 2), and the out flow rate stabilizes.

The consolidation also induces a settlement of the global height of samples, and a decrease in the void ratio, e, of the tested material. The settlement of the specimen was measured at the end of the test, and was also deduced from the fitted model, computing the difference between the cumulated inlet and outlet volume at the top and the bottom of the specimen. Results agree and the mean relative settlement is presented in Table 2. From these measurements and the initial W/C ratio, the mean value of void ratio, e, inside the specimen, during the permeability measurement phase, is deduced using Eqs. (1) and (3).

In order to simplify the analysis and compare all of the experimental data, the constant flow rate, occurring after the consolidation phase (see Fig. 3) has been considered to measure the permeability of the material remaining at constant mean void ratio. The overflow due to consolidation can be computed from the fitted model. When it represents less than 3% of the total outflow, a constant flow rate is assumed and a linear regression can be fitted in order to determine permeability. Thereafter, the permeability values presented take into account the mean specimen settlement due to consolidation: the slight increase in the hydraulic gradient, and the slight decrease in void ratio, e, when a relationship of K versus e is considered in Section 5.2.

The consolidation process tends to be shorter when the initial water-to-cement ratio decreases. In the present study, it has been assumed to be negligible beyond 2200 s in case of initial W/C = 0.3 (see Fig. 4). To evaluate the permeability, the linear regressions were applied, over a period of 300 s at least, beyond: 27; 30; 33 and 36 min in case of initial W/C = : 0.4; 0.37; 0.33 and 0.3 respectively. The correlation coefficients confirm the linearity, i.e., a constant flow rate during the considered periods.

It should be noted that the evolution of permeability due to cement hydration has been studied in Section 4.2 and in previous studies [1]. From these experiments, the permeability of fresh cement paste at rest could be assumed to be constant over the first hydration hour, since no significant variation has been measured during this period.

### 3.5. Hydraulic gradient effect on measured permeability

When water percolates through samples, a leaching effect may take place. The finest particles may be washed out and cake on the filter, or a dissolution and/or precipitation of ions due to circulating fluid may occur. In such cases, the permeability of the material should change significantly, and should depend on the percolated volume and the flow rate. Darcy's assumptions would no longer be verified. Higher hydraulic gradients would induce a higher flow rate and would magnify the leaching effect.

In Fig. 3, the applied pressure is equal to 30 kPa for each sample. For a given W/C ratio, tests were also carried out with 6 levels of hydraulic heads, (i.e. differences between the hydraulic head at the top and bottom of specimen). 1, 2 and 3 m using a Mariotte Bottle, and 3, 5 and 8 m using the air pressure regulator, set at 30, 50 and 80 kPa respectively (see Fig. 1). It should be noted that the mean water level above the outlet of the cell is less than 0.1 m, i.e. less than the 1 kPa regulator accuracy. Hence, the pressure due to water head inside the cell has been neglected in the case of an air pressurized test. To ensure the validity of Darcy's law, the permeability was plotted according to mean hydraulic gradients ranging from 15 to 130 in Fig. 5. Since no significant trend was observed, the permeability is assumed independent on hydraulic conductivity for each W/C ratio studied.

Otherwise, the effect of probable ion dissolution in the saturating water on the viscosity of the percolating water during the first hour of hydration has been measured. No change in water viscosity was detected. The viscosity of the percolating fluid can be assumed as constant over the test period.

In such a context, Darcy's law is considered as verified and the permeability measurements can be assumed to be valid.

## 4. Permeability measurement using a controlled oedometer

This second method consists in inducing water percolation through a 20 mm initial height cement-paste sample (approximately 40 $cm^3$ volume). It requires a compression testing machine controlling displacements and pore water pressure sensors. This makes it possible to measure permeability more accurately, and to proceed with many permeability tests for several compaction states from the same fresh cement paste sample.

### 4.1. Experimental device

The measurement device is an oedometer device placed on a compression press. The oedometer ring is 50 mm in diameter and 20 mm in height. The specimen is drained one way, with pore pressure measurement on the bottom face. Porous stones are placed on the top and bottom surfaces of the specimen. Two filter papers are placed between the porous stone and the specimen in order to ensure

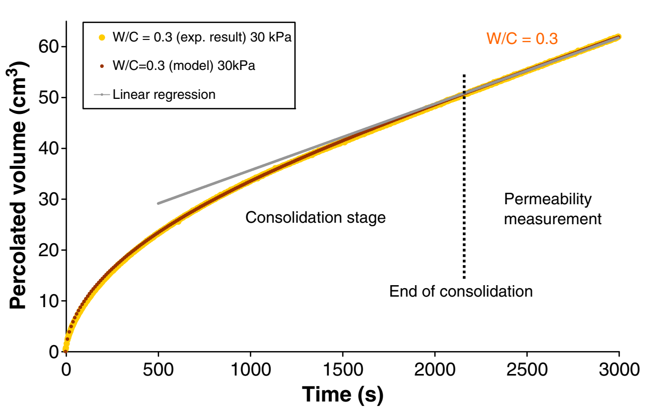


**Fig. 4.** Typical percolated water volume versus time showing consolidation and Darcy's percolation phase.

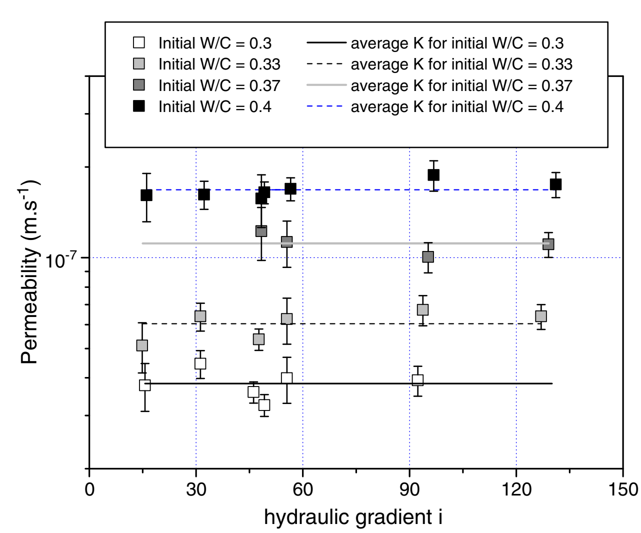


**Fig. 5.** Hydraulic gradient effect on derived permeability value.

that only the fluid phase can flow through the drainage system. A perforated piston is placed on the top face of the specimen and put in contact with a load frame in order to measure the vertical stress applied to the sample. The variation of the specimen height is measured by a displacement transducer with an accuracy of 0.01 mm.

During all the tests, the oedometer cell is totally immersed. In this way, a constant water head is applied to the top face of the specimen. Two kinds of test stages can be performed: loading stages and permeability measurement stages.

The loading stages correspond to classical one-dimensional compression tests. A variation in height of the specimen is applied while the induced stress on the material is measured. The bottom face is kept undrained (valve A is closed in Fig. 6), and the pore pressure transducer allows the monitoring of the generated excess pore pressure.

The permeability measurement stages proceed for a fixed height of the specimen. The test configuration is like the usual rigid-wall permeability cell. The permeability tests were conducted using the constant head method with bottom up water flow. The constant hydraulic head was applied by means of a Mariotte siphon connected to the bottom face of the cell (valve A in Fig. 6 open).

As shown in Eq. (4), the permeability, K, is calculated from Darcy's law from the rate of inlet water volume $Q = \Delta V/\Delta t$:

$$K = \frac{\Delta V\ h_i}{\Delta t\ S\ \Delta H_W} \tag{8}$$

- $h_i$: height of the specimen during the permeability measurement stage
- $\Delta H_W$: Applied hydraulic head (m), difference between hydraulic head at top and bottom of specimen.

With this method, different stages of permeability measurement can be performed successively for decreasing values of void ratio on the same specimen.

### 4.2. Hydration influence on permeability

A cement CEM I 32.5 R was first used in order to evaluate the effect of hydration time and hydraulic gradient on permeability measurements. A relationship between the void ratio and permeability is also presented in that case.

In order to determine the hydration effect, an initial permeability test was performed for a given void ratio of 0.945 (W/C = 0.30) and for a constant hydraulic gradient.

The permeability test was performed over 90 min. The time evolution of the cumulated volume of inlet water is shown in Fig. 7. The inlet flow rate appears almost constant until 3600 s and decreases significantly beyond that time. Therefore, test durations are limited to 3600 s.

### 4.3. Hydraulic gradient effect on permeability value

For a given W/C value of 0.34, i.e. for a fixed height of the specimen, four permeability measurement stages were performed for different values of applied constant water head. Results are shown in Fig. 8.

As observed in Section 3.5 for the filtration test, the permeability variations due to hydraulic gradient are minor, lower than 4% in this case, and no significant trend appears. Therefore, cement paste permeability seems independent of the hydraulic gradient in the tested range as shown in Fig. 8. In consequence, the testing procedure and the computation of permeability using Darcy's law (see Eq. (8)) can be regarded as valid.

### 4.4. Experimental protocol and test procedure

Tests were performed with four permeability measurement stages on each specimen. Each permeability measurement stage proceeded until the inlet volume reached the volume of void inside the specimen.

Five specimens made with different initial W/C ratios were tested in order to determine the permeability evolution on the same range of void ratios from different specimens.

A first load was applied until contact between the load frame and the piston was detected (by measuring the loading force). The displacement was stopped and the height of the specimen kept constant while the pore water pressure on the bottom of the specimen was monitored. The pore pressure, generated during the loading stage, decreased toward zero until the primary consolidation was completed. From the dissipation stage, the consolidation coefficient $C_V$ ($m^2 s^{-1}$) was deduced. For instance, if W/C is equal to 0.37, $C_V = 2.4\,10^{-6}\ m^2 s^{-1}$, which is in accordance with the value deduced in Section 3.3, (see Table 2).

Then, a permeability measurement stage was performed by connecting the Mariotte bottle to the bottom drainage system of the

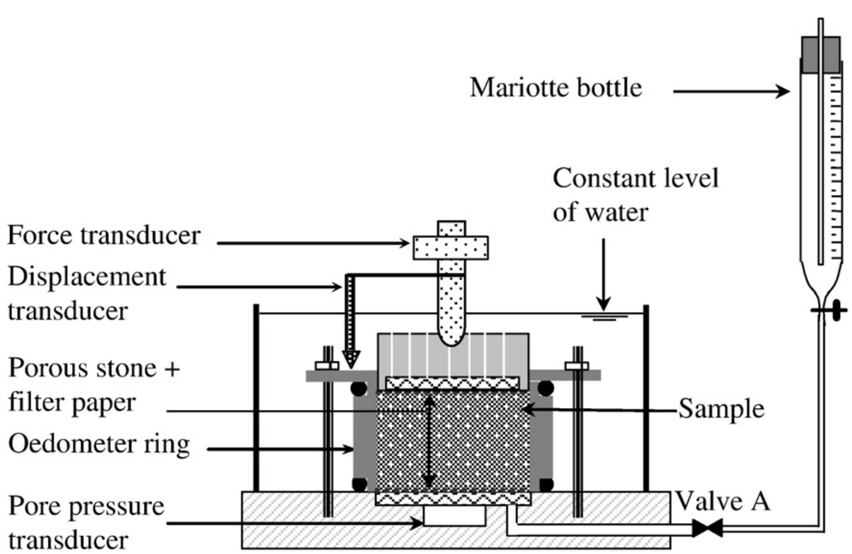


**Fig. 6.** Controlled oedometer device.

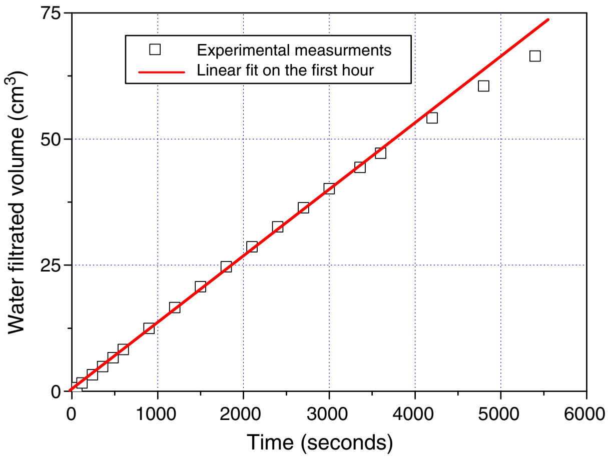


**Fig. 7.** Evolution of inlet water volume over time.

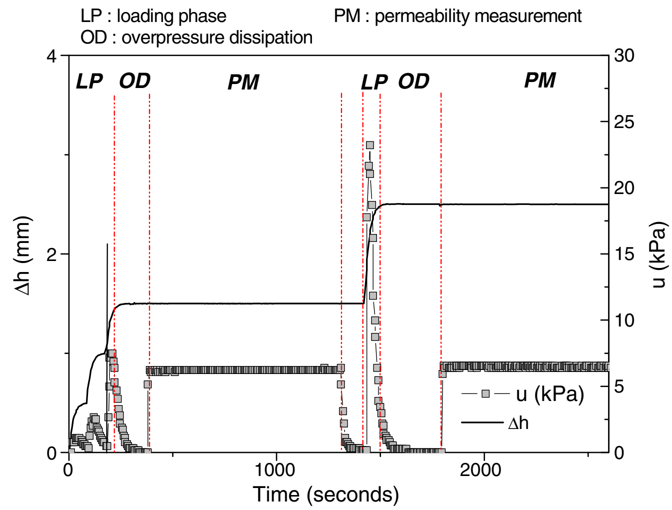


**Fig. 9.** Sample height and pore water pressure at the cell bottom during specimen testing.

cell, i.e., a constant water pressure was applied. When the inlet volume was at least equal to the void ratio, the permeability measurement stage was stopped, and a new loading stage was performed in order to decrease the void ratio. Fig. 9 shows the pore water pressure measurement at the cell bottom during the testing procedure.

The results, obtained with a CEM I 32.5 are summed-up in Fig. 10. A unique relationship between permeability and void ratio of cement paste appears.

If a linear relation between e and log K is assumed, the extrapolated values of K for a void ratio equal to 1.0 varies from 2.71 $10^{-7}$ m $s^{-1}$ to 2.94 $10^{-7}$ m $s^{-1}$ with an average value from the five tests of 2.85 $10^{-7}$ m $s^{-1}$ and a standard deviation of 9.05 $10^{-9}$ m $s^{-1}$.

As shown in Fig. 10, from the five tests, a unique relationship between void ratio and K can be assumed.

## 5. Permeability modeling and methods comparison

### 5.1. State of the art

Predicting a material's permeability according to its microstructrure has been an important source of research for scientists. Many studies and publications are related to this matter.

The most famous and often-used model is probably the Kozeny–Carman equations [12]. Solving the Poiseuille problem, the authors predict the hydraulic conductivity K as a function of the void ratio for a network of capillary tubes. The specific surface, S ($m^2 kg^{-1}$), and a material constant C are the constant parameters relative to the porous material as shown by Eq. (9):

$$K = C \frac{g}{\mu_w \rho_w} \frac{1}{S^2(\rho_s/\rho_w)^2} \frac{e^3}{1+e} \quad (9)$$

where $\rho_s$ and $\rho_w$ are the unit mass weight of solids and water respectively, g the constant gravity and $\mu_w$ the dynamic viscosity of the percolating fluid (i.e., water).

Since its first appearance, this equation has been adapted and improved for various designs of material. For instance, the C constant and the specific surface can be written as a function of the material tortuosity [15], the size distribution and shape of solid particles [16], the pore connectivity [17], and/or the percolation threshold [18]. Samarasinghe et al. [19] have proposed to adjust the void ratio coefficient (equal to 3 in common Kozeny–Carman) to fit the experimental data better.

Nevertheless, none of these improvements can be generalized for every material. As a consequence, the classical Kozeny–Carman formulation has been chosen in the present study.

In the case of spherical particles assemblies, Carman proposes a coefficient C equal to 0.2. The author claims that this value provides

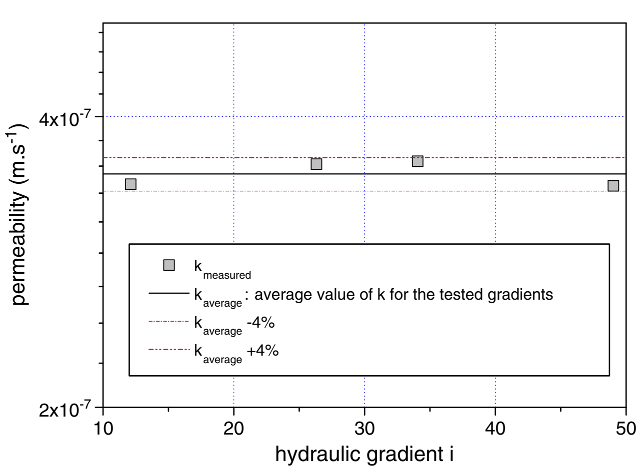


**Fig. 8.** Effect of hydraulic gradient on permeability.

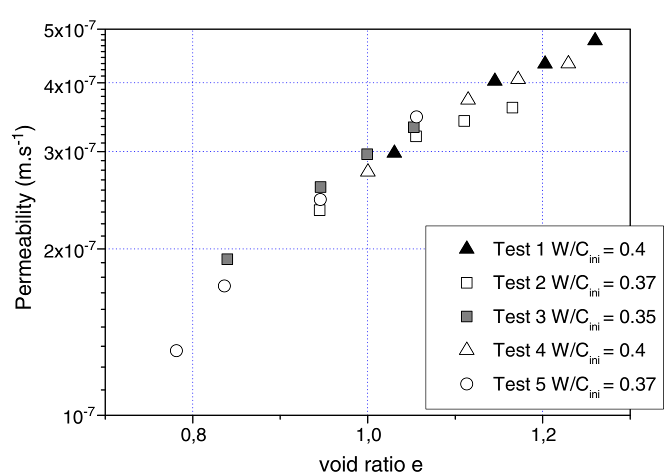


**Fig. 10.** Evolution of log K versus void ratio, e, for 5 tests.

the best experimental results taking into account the possible deviations due to tortuosity.

It is a widespread view that the Kozeny–Carman equation is valid for non-plastic soils such as sand or gravel, but is inadequate for clays. More recently, Chapuis and Aubertin [20] have reminded us that Carman [21] and Taylor [22] did not find a linear relationship between K and $e^3/(1+e)$ in case of clay materials. Also Lambe and Whitman [23] show that the hydraulic conductivity depends on the chemical nature of the fluid, i.e., in case of µm mean diameter particles, fluid polarity and Van Der Waals forces have to be taken into account in the model. In such cases, authors such as Taylor [22] and Michaels and Lin [24] also proposed an empirical linear relationship between log K and e.

$$\Delta(log_{10}K) = \frac{\Delta e}{C_k}. \qquad (10)$$

This relationship is known to provide a good approximation of permeability when the material initial void ratio is less than 2.5 [25,26].

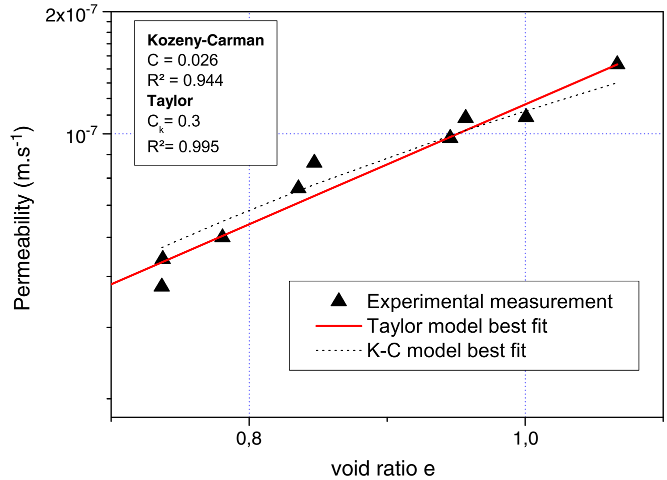


**Fig. 12.** Analysis of method B results.

### 5.2. Comparison methods

Thereafter, the method described in Sections 3 and 4 are named method A and method B respectively. The mean results obtained from these two methods are plotted in Figs. 11 and 12 versus the void ratio e and compared to the Taylor and Kozeny–Carman models.

The Taylor and Kozeny Carman models both present a constant parameter (respectively C and $C_k$) to be adjusted according to the experimental data. The two models are fitted by least squared method from experimental data obtained with methods A and B. The fitted values of C, $C_k$ and $r^2$ coefficients are given and the relationships between K and the void ratio, e, are plotted in Figs. 11 and 12.

The A method provides a better correlation with the Taylor model with a coefficient of 0.945, while the application of the Kozeny–Carman model leads to a C value of 0.033, associated to a lower coefficient of correlation: 0.667, see Fig. 11.

The B method gives experimental data in agreement with the Taylor and Kozeny–Carman models, with a coefficient of correlation higher than 0.944, see Fig. 12.

It appears that the coefficient $C_k$ is significantly different between the two methods, whereas the C coefficient of the Kozeny–Carman model obtained from the two methods is close.

The difference between permeability values is small between the two experimental tests. For a given tested void ratio, the permeability values vary within ±30%, which is accurate according to the literature. The difference between the results obtained by the two methods can be partly explained by consolidation gradient and sample heterogeneities.

–In method A, the sample height is 70 mm, which is quite high for a standard permeability test. Such a height accentuates the influence of sample heterogeneity after consolidation. An average void ratio is calculated assuming the sample remains homogeneous, but a gradient of void ratio should exist along the height of the specimen (see Section 3.3). In layered material, the equivalent permeability coefficient is mainly governed by the less permeable layer. As a consequence, the void ratio associated to the deduced permeability is slightly over-estimated, but gives acceptable results as shown by the correlation with the other method.

–In method B, the specimen is initially subjected to a loading force before the permeability measurements. The effective stress initially applied ranges from 20 kPa (first displacement stage) to 800 kPa (last displacement stage), while the applied water head pressure is about 6 kPa. As a consequence, the influence of the effective stress due to the water driven pressure during the permeability measurement stage is lower than in method A. The material is already over-consolidated and does not consolidate during this stage since the height of the specimen is kept constant.

Fig. 13 sums up the whole of the experimental results (method A + B). For the Kozeny–Carman model, the value of C providing the best fit is 0.028.

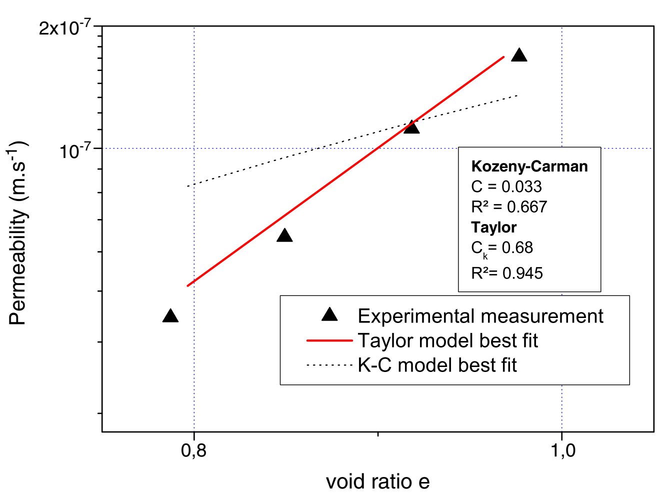


**Fig. 11.** Analysis of method A results.

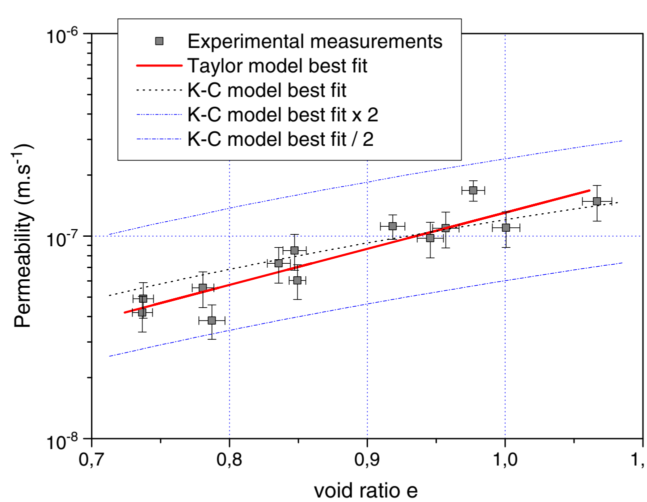


**Fig. 13.** Analysis of tests results (method A + method B).

Thus, the difference between experimental and modeling value is always lower than 50% which is considered to be accurate according to standard soil permeability measurements [20]. With the same size distribution of spherical particles, Carman suggests a C value of 0.2 (i.e., multiplied by a factor of approximately 10 since the deduced values of C range from 0.026 to 0.033). But in the case of cement, grain shapes are far from spherical and percolating paths are more tortuous and could be locally thinner. The particles' roughness has to be taken into account. Moreover, the minimal water amount linked to the grains of high specific surface particles should reduce the mean flow path diameter [27]. Roussel et al. [28] also explain that the local radius of sphericity, and micro structure interactions, such as Van Der Waals forces, have to be integrated to find a representative particles diameter. This would justify the dividing factor of 10 about the C parameter.

The Taylor empirical modeling fits well with all of the data. The $C_k$ parameter is close to those obtained for cohesive plastic fine soils such as silts or clays [29].

In conclusion, these two simple models provide good K(e) relationship after fitting only one parameter. However, as for fine-grained materials, no model provides a direct predicted permeability value.

## 6. Conclusions

This paper presents methods of measuring the permeability of fresh cement paste, in the first hours of the hydration period, with a W/C ratio ranging from 0.3 to 0.4.

The first method, A, uses a simple device, (i.e., a common soil permeameter cell). Pressure applied on the top of the cell induces a water percolation through a 60 to 70 mm initial height layer of material. The same process could easily be used to test mortar or concrete. But this method needs special care in the analysis stage. The consolidation effect must be considered to deduce precisely, within 30 to 60 min, the permeability of the material. A longer testing time would lead to a better accuracy, but the hydration effect of cement would induce a decrease in the permeability of the material.

The second method, B, using a controlled oedometer, allows an accurate permeability determination of a limited-height sample (20 mm) in a rigid wall permeameter. A better control of the sample heterogeneity induced by consolidation and particles migration under fluid movement is achieved.

The two methods appear relevant for the permeability determination, if correct assumptions are made concerning the permeability computation. Method B seems to be more efficient in permeability determination due to a better monitoring of sample height and homogeneity, due to induced over consolidations before the percolation stages. However, method A gives permeability values with an acceptable level of accuracy using a simpler device and test procedure, with a higher representative sample volume.

Results confirm that fresh cement paste, in the range of hydration time under scrutiny, W/C, and effective stress, behaves according to the soil consolidation theory. Its permeability increases when the void ratio decreases, and ranges from $3.8\,10^{-8}\,\mathrm{m\,s^{-1}}$ to $1.7\,10^{-7}\,\mathrm{m\,s^{-1}}$ in this study. Moreover, hydration does not induce a notable decrease in permeability of cement pastes until the first hour of hydration.

The application of two simple models (Kozeny–Carman and Taylor) on whole tests results shows that the permeability versus the void ratio can be well simulated when only one parameter, relative to the grain shape and size distribution, is identified by fitting. Moreover, it appears that the difference between experimental value and modeling is always less than 50%. This accuracy is in accordance with the permeability evaluation.

The fitting parameter $C_k$, of the Taylor empirical model, is close to those obtained for clays or fine-grained soils, while the fitting parameter C, of the Kozeny–Carman phenomenological model, is divided by ten from the usual values obtained for sand (i.e., 0.2). This point shows clearly that grain-scaled interactions such as the Van Der Waals forces have to be taken into account for a complete modeling of the permeability of cement paste.

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