MEASUREMENT OF THE RELATIVE GAS PERMEABILITY OF ORDINARY CONCRETE: INFLUENCE OF SATURATION DEGREE AND THE SAMPLE SIZE

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Abstract: The gas permeability is an important indicator to assess the material's ability to prevent the ingress of aggressive chemical species. The presence of aggressive fluids and their transport is by far the most important factor controlling the durability of cement based composites. This paper presents an experimental study of gas permeability of ordinary concrete according to its saturation, because the hydric state of concrete conditions the transfer parameters such as permeability. Thus the world of aggressive penetration varies significantly depending on moisture content and its evolution over time. The experimental results concerning the measurement of gas permeability of concrete samples with different sizes and that of the degree of saturation by weighing and therefore mass loss measurements were performed. The analysis highlights the effect of saturation on the variation of the relative gas permeability of concrete stressing the specific attention to sample size (scale effect). Validation of the law of Van Genuchten based on Mualem's model for porous media has been verified for our ordinary concrete (OC).

1 INTRODUCTION

In concrete structures, durability of concrete and the corrosion of reinforcing steel are intimately linked to the permeability of exposed concrete surfaces [1]. Therefore, permeability, defined as the movement of fluid through a porous medium under an applied pressure head, is the most important property of concrete governing its long-term durability [2]. Gas permeability of concrete is a major indicator for evaluating the ability of this material to prevent the penetration of aggressive agents like carbon dioxide (CO_2) and the transfer of water vapor from drying of the material. For more than a century, Darcy's coefficient of permeability has been used as a measure of its leaktightedness and its durability is often derived from this leaktightedness [3].

The hydric state of the concrete is so important because, according to the saturation rate of the material, different pore sizes are available [4]. Therefore, measuring the permeability is influenced by moisture content of the material [5]. Coussy *& al.* [6] have shown that a change in physical-mechanical properties of cementitious materials strongly depends on their degree of saturation. Within the meaning of Darcy permeability was measured on materials saturated with water, yet the reality is that the concrete is partially saturated

This paper is devoted to the assessment of the durability indicator: gas permeability. We present here the results of an experimental study concerning the measurement of the relative gas permeability of an ordinary concrete (OC) having any value of moisture content between the saturated state and perfectly dry. To achieve this objective, concrete is characterized by its intrinsic permeability (see section 2.2) and the variation of this parameter is studied against the variation of the degree of saturation of the material. In the other hand, this study is carried out with the aim to underlines the effect of the size of the test body (scale effect) on the measurement of gas permeability of concrete versus saturation degree.

The results are likely to be introduced into models to predict mass transfer in concrete and predicting the durability of concrete structures.

2 EXPERIMENTAL PROGRAM

2.1 Concrete composition and specimen curing

The study was conducted on a non-adjuvanted ordinary concrete, it was made with an ordinary Portland cement (CEM II/A 42.5) from the factory of Beni Saf (west Algeria). The chemical and mineralogical compositions of this cement are presented on Table 1. Aggregates (sand and gravel) are from the career of the National Company in Sidi Abdelli Aggregates (wilaya of Tlemcen). The mix proportion of concrete tested in this study is given in Table 2.

We chose for this test campaign, three sizes of cylindrical specimens: (5x10) cm, (11x22) cm and (15x30) cm. This concrete mixture is cast in cylinders molds ((11x22) cm and (15x30) cm) and compacted using a mechanical vibrator. To obtain the concrete specimens with 5 cm diameter, we applied coring, using a core drill, on concrete cylinders with 15 cm diameter, after 28 days curing.

After casting, the concrete, which was covered with a plastic film to avoid water evaporation, was kept in the moulds for 24 hours. Then, each cylindrical specimens used ((15x30) cm and (11x22) cm) was removed from its mould and cured in tap water at temperature of 20 ± 2 °C for 4 weeks to stabilize the hydration mechanism**.**

Table 1: Chemical and mineralogical (Bogüe) compositions of Portland cement

Chemical composition (%)		Bogue composition (%)	
SiO_2	24.82	C_3S	55.65
Al_2O_3	5.62	C_2S	20.18
Fe_2O_3	3.07	C_3A	9.77
CaO	60.98	C_4AF	9.5
MgO	0.74		
SO_3	2.45		
K_2O	0,43		
Na_2O	0.30		
CaO libre	0.94		
Fire loss	1.71		

Table 2: Mix proportion of Concrete

Mix ingredients (kg/m3)	Amount
Cement CPA-CEM II / A 42.5	350
Aggregate 15-25 mm	533
Aggregate 8-16 mm	432
Aggregate 3-8 mm	144
Sand	660
Water/Cement ratio	0.5
Dry apparent density	2471
Total open porosity (%)	13.7
Compressive strength at 28 days (MPa)	35

At the age of 28 days, the specimens were removed from the water. First, we applied coring of 5 cm diameter on two cylindrical specimens. Then, samples of 5 cm thickness were sawn from all the original specimens of concrete by using a diamond blade saw, to obtain. One disc, with 5 cm-thick, is extracted from the middle (central portion) of each concrete cylinder to use for permeability tests. The discs are then ground true. Their thickness is measured with an accuracy of 0.1 mm. Their curved surface is sealed with two epoxy resin coats to ensure a one dimensional gas or water flow inside the discs.

2.2 Gas permeability test procedure

The permeability was measured in the GeM Laboratory, using a constant head permeameter, known as the CEMBUREAU parmeameter, and Nitrogen is used as a neutral percolation gas [7]. A general view and the general layout of the apparatus are given in figure 1 and 2 respectively.

Figure 1: General view of gas permeameter (The CEMBUREAU), (GeM Laboratory - UIT of Saint-Nazaire (France))

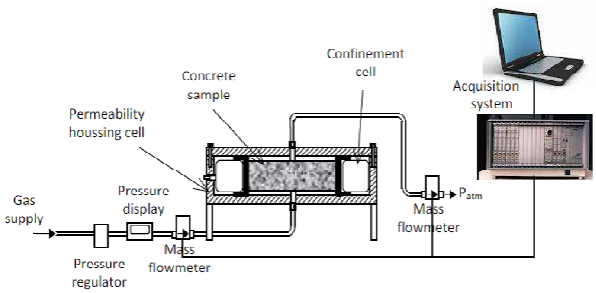

Figure 2: General layout of the apparatus of gas permeameter (CEMBUREAU)

The test consists in subjecting a concrete test body, to a constant pressure upstream P_{inj} (Injection pressure) to the stabilization of the flow of gas through the material (steady state); the downstream pressure is the atmospheric pressure P_{atm}. The gas flow is measured upstream and downstream of the test body by mass flow meters which convert the mass flow rate in a volume flow rate equivalent. The principle of conservation of mass to Darcy's law for isothermal flow of a gas, considered perfect, to determine the apparent permeability K_a (in m²) [8]. The apparent permeability (K_a) is not only dependent of the pore structure of the material, but also varies with the applied mean pressure; $P_{moy} = (P_{inj}/P_{atm})/2$.

Permeability measurements are carried out in an air-conditioned room (20 ± 1 °C). Each disc is tested at five differential pressures: 0.1, 0.15, 0.2, 0.25 and 0.3 MPa (3 x 10^5 N/m²). A pressure difference up to 0.5 MPa can be applied to the specimens in the pressure cells which are sealed by a tightly fitting polyurethane rubber pressing under high pressure against the curve surface. After initializing the percolation of nitrogen through a specimen at a given applied pressure, sufficient time (about one hour) is provided for the establishment of the steady state flow before an actual measurement is taken. This condition is verified by taking two measurements separated by a 10-min time interval. If two values differ by less than 2%, the steady state flow condition is assumed to be achieved.

By assuming laminar of a compressible viscous fluid through a porous body, the apparent gas permeability, denoted K_a , can be calculated from direct measurement, for each differential pressure (one side of the test specimen is submitted to a constant inlet pressure P), by using equation (1) derived from Hagen-Poiseuille's law when applied to compressive fluids (gases) [7]:

$$K_a = \frac{2.\mu.Q.L.P_{atm}}{A(P_i^2 - P_{atm}^2)} \qquad (1)$$

Where: Q is the volume gas flow (m³/s); A : cross-sectional area (m²); L: Thickness (in the flow direction) of the sample (m); μ : coefficient of viscosity of the gas (1.76 x 10^{-5} N.s/m²) for nitrogen gas at 20 °C); P_{inj} : Inlet pressure, i.e., applied absolute pressure (N/m²); P_{atm}: atmospheric pressure (N/m²).

To determine the intrinsic permeability of the material, that is to say independent of the permeability gas pressure, it is necessary to make several measurements at different pressures. Thus, the apparent permeability was measured under pressure gradients between

0.1 and 0.3 MPa in steps of 0.05 MPa. To obtain the value of the intrinsic permeability K_{int} which is solely a characteristic of the void network (independent of the applied mean pressure and theoretically of the fluid, liquid or gas), and which is associated to pure viscous transport, we used Klinkenberg's method [9] based on equation (2)

$$K_a = K_{int}\left(1 + \frac{\beta}{P_{moy}}\right) \quad (2)$$

$$P_{moy} = \frac{P_{atm} + P_{inj}}{2} \quad (3)$$

Where the so-called Klinkenberg's constant β is a characteristic of both the porous medium and the percolating fluid. $\beta.K_{int}$ is the slope of the straight line (linear regressions). This method has already been used by various authors (Bamforth, 1987; Perraton *& al*., 1999; Abbas *& al*., 1999; Villain *& al*., 2001) [10-11-3-5]. Klinkenberg's concept is based on the observation that the measure gas flow is higher than the value theoretically expected, as a result of slip effects in addition to pure viscous flow [12].

The determination of intrinsic permeability (K_{int}) consists in measuring apparent permeability (K_a) at different pressures (P_i) and in plotting it against the inverse of the mean pressures ($1/P_m$). From the five differential pressures applied, the correlations of the linear regressions are satisfactory with coefficient higher than 0.99 [13].

The intrinsic permeability (K_{int}) is the limit value of the apparent permeability when the $1/P_m$ tends towards 0 (i.e. P_m tends towards infinity), that is to say when the gas tends towards a condensed phase (liquid) [14]. The permeability is then regarded as the intrinsic gas permeability K_{int} determined in equation (2) and depending on the degree of water saturation of the tested concrete [3].

2.3 Drying procedure

Preconditioning procedure (a procedure of drying out moisture), is an unavoidable necessity to measure the relative gas permeability of concrete. The procedure of preconditioning must be rapid and easy to perform and minimize the modification of the porous structure of the material [15]. In order to verify these requirements, some preconditioning procedures consist of heating the specimen at 80 °C for 28 days and after at 105 °C before permeability measurements [16-17-5]. These procedures are very rapid and easy, but they modify the pore structure [18-19] and may induce microcracking due to drying shrinkage and then artificially increase permeability of concrete. For that purpose, numerous studies aim to develop experimental preconditioning procedure for determining gas permeability [3-13-15-17].

In this study, after 28 days of curing and after preparing concrete samples with different sizes (diameters, 15 cm, 11 cm and 5 cm; thickness, 5 cm), concrete specimens were divided in three groups.

About the first group concerned only the concrete samples with 15 cm diameter; three samples for gas permeability test were used. Before testing, the samples were dried at 60 °C increments and the duration of each level is equivalent to 24 h drying in the ventilated oven. In the second group, nine specimens with different sizes (three test bodies for each diameter: 5, 11 and 15 cm), were placed in an oven and dried at 70 °C increments and then retained to achieve different levels of saturation. The duration of each level is equivalent to 48 hours drying in a ventilated oven. Then, before testing, all of the test bodies in this study are cooled for 24 h in desiccators at 20 °C to achieve thermal stabilization before starting the permeability test. Their mass is then measured with an accuracy of 0.02 g to determine their moisture content. Thus by weighing, it was possible to obtain various degrees of saturation inside the sample to get information on the gas permeability saturation dependence. In order to have uniform saturation state, the lateral surface of the samples were covered with epoxy resin and aluminum adhesive, so that the gas transfer, during drying and the measurement of the gas occur only in so-way

(perpendicular to the two planar surfaces of the samples).

Accounting for the slow drying process, due to drying step by step, the relatively small specimen thickness and the lack of mass loss during the water redistribution period, we expected good saturation state uniformity

The characteristics of this preconditioning were chosen, first, to limit the risk of cracking due to temperature gradients, second, to obtain the saturation level as homogeneous as possible and to determine the influence of different drying methods. Given the large size of the test bodies, like slices with 15 cm diameter and 5 cm thickness, to be closer to the perfectly dry state, it took us several months of drying. The complete saturation of the samples tested was carried out under vacuum in a desiccator as recommended by the AFREM [16].

3 RESULTS AND DISCUSION

3.1 Nature of the gas flow

The permeability tests are performed at constant pressure, in continuous operation. According to several authors (Villain *et al.* and Perraton *et al.*)[5-11], it is necessary to verify the nature of the gas flow during the test gas permeability, because, according to Klinkenberg [9], the determination of the intrinsic permeability is located in the context of a viscous flow.

According to Perraton [20], the measurement points corresponding to a viscous flow are aligned and the slope of the line must be greater than or equal to 1. In the case of a non-constant mean pressure during the test and high gas flow rates, the measuring points are no longer aligned and the curve has a concavity turned towards the axis (slope at any point less than 1) [5]. We applied this method to verify the nature of the gas flow for each sample tested in this study.

Figure 4 shows a portion of these results for test bodies of similar size (15 cm diameter) and different degrees of saturation. According to the curves obtained (figure 3), it is clear that

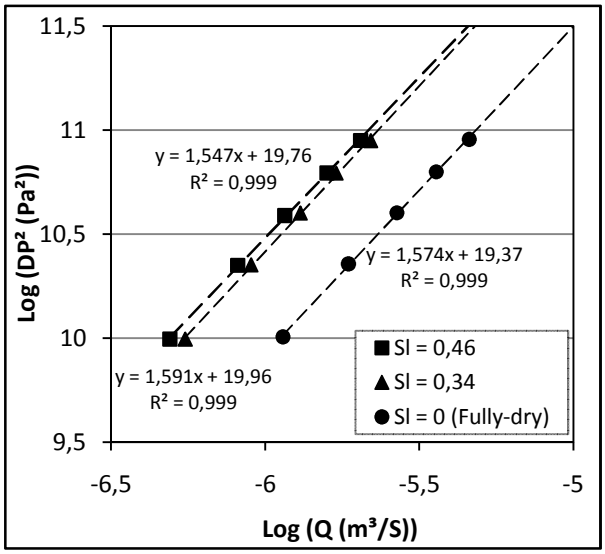

Figure 3: Relationship between the square of the pressure gradient DP ² and the gas flow rate Q in the permeability test

the linearity property is respected and the slope of the three lines is greater than 1, which is consistent with the assumptions of the method. It is therefore considered that the flow is viscous and, in this study, the intrinsic permeability can be approximated the theory of Klinkenberg.

3.2 Evolution of gas permeability with the degree of saturation

The evolution of intrinsic permeability with the degree of saturation is given in figure 4. These results correspond to three samples of concrete from the same batch of concrete and the same size (15 cm diameter) for which the degree of saturation is determined by weighing after drying progressing (see section 2.3).

The results show that gas permeability is influenced by the saturation degree of the concrete samples. This confirms the results obtained by several researchers.

The gas permeability of a concrete specimen increases when the saturation rate decreases [3-21]. This can be explained by the fact that, more there is continuity between the water molecules in the porous network of the material; more the passage of gas is prevented.

The intrinsic permeability curves as a function of saturation may be made by means of logarithmic curves.

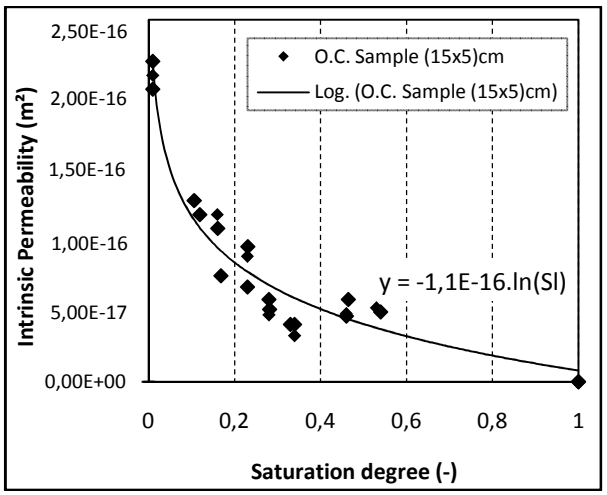

Figure 4: Intrinsic permeability of the concrete according to the degree of saturation

The slope $\beta.K_{int}$ of the Klinkenberg's straight, as function as the rate of saturation, complete characterization of concrete tested [5]. It calculates the apparent permeability of the concrete at a mean pressure different from that used for the measurement from the equation (1). From these measurements of the permeabilities at different degrees of saturation of concrete specimens, it is possible to deduce a curve $K_{rg} = K_{rg}(S_l)$, where K_{rg} is the relative gas permeability (Figure 5).

Relative gas permeability K_{rg} (defined as the ratio between the permeability measured at a given saturation $(0 < S_l < 1)$ and permeability measured in fully dry $(S_l = 0)$) can be correlated with the degree of saturation. So, different relationships have been proposed by different researchers attempt to quantify the influence of water saturation S_l on concrete permeability [3-5].

In the literature, there are analytical formulas for the relative gas permeability K_{rg} which are often accepted and which are corresponding with the famous equation given by Van-Genuchten [22] for modeling the permeability of a porous material;

$$K_{rg}(S_l) = (1 - S_l)^q . \left(1 - S_l^{1/m}\right)^{2m} \qquad (7)$$

And using the model of Mualem [23] (with $K_{rg}(S_l) = 1$ when $S_l = 0$ (fully-dry)) [24]. In this formula, m is a coefficient to be determined from the capillary pressure curve of the material $(P_C = P_C(S_l))$ [25]. From a series of measures from the literature, values of q between 3.5 and 5.5 appear to be satisfactory [26]. The value of $q = 5.5$ can be generalized for calculations of the relative permeability to gases of all cementitious materials [27].

With $m = 0{,}5$, the evolution of the relative gas permeability, according to the degree of saturation, will be defined by the following formula (equation (8)) [26]:

$$K_{rg} = (1 - S_l)^q . (1 - S_l^2) \qquad (8)$$

The validity of this law of Van Genuchten modified with q = 2.5 to 5.5 for modeling the permeability of a porous material, has been verified with the experimental results with our concrete (figure 5).

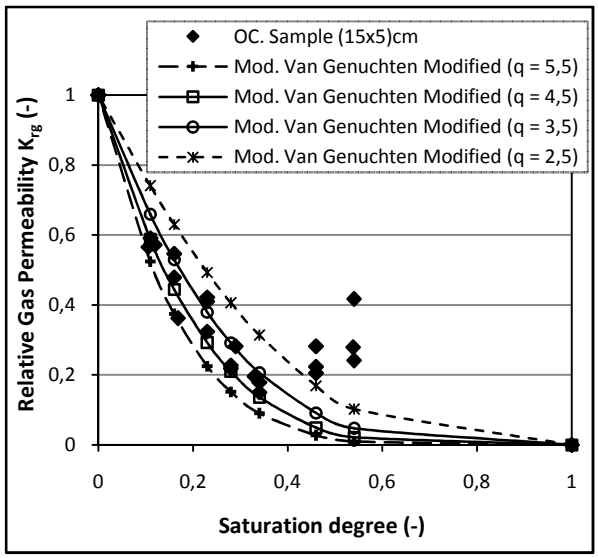

Figure 5: Relative gas permeability of concrete according to the degree of saturation

The relative gas permeability for all the samples, with the same size (15x5) cm, was calculated and the values are shown in figure 5. The relative gas permeability deduced from the modified Van Genuchten's relation is also represented in this figure.

For a small variation of water saturation in the 0 and 0.2 range, the experimental gas relative permeability decreases strongly to about 40 % of permeability rate to its intrinsic permeability measured at fully-dry condition (S_l = 0).

In the range of variation in degree of saturation (S_l between 0 and 0.4), relative permeability calculated from modified Van

Genuchten's relation fits almost perfectly with the experimental results, with values of q between 3.5 and 4.5 for the three samples tested. The relative agreement between calculated and experimental values is due to the fact that equation (8). Beyond 60% saturation, relative gas permeability, calculated by the analytical formula (equation (8)), given by Van Genuchten, becomes almost zero. This is in agreement with previous studies, quantitative or qualitative, to highlight the influence of this variable (degree of saturation) on the transfer properties. Thus, Picandet *& al.* [21] show that whatever the mechanical condition and the concrete mix (ordinary OC, high performance HPC or high performance fiber HPFC), more the material is dry, more the gas permeability of the concrete increases. Therefore, water saturation is an important parameter influenced on gas permeability.

3.3 Influence of sample size on the permeability test

In this section, we focus on scale effects on the measurement of relative gas permeability, especially to the influence of specimen size on the permeability depending on the degree of saturation.

3.3.1 Test results of gas permeability of samples with different sizes

Figure 6 shows the evolution of the intrinsic permeability of the different test body (sample with different sizes) based on their degrees of saturation. In this part of the study, an arithmetic mean of three measurements on samples of the same size is given.

The results show that the intrinsic permeability decreases with increasing the diameter of the concrete samples. The intrinsic permeability of concrete is then not only influenced by the degree of saturation, but also by the size of the test body. The evolution of the intrinsic permeability of concrete between the dry and 20 % of liquid water saturation degree is very important. In this range, the permeability decreases significantly from 10^{-16} to 10^{-17} m². Preconditioning (dry procedure) and size of the test body of the concrete tested, have a great influence on the measure of the permeability.

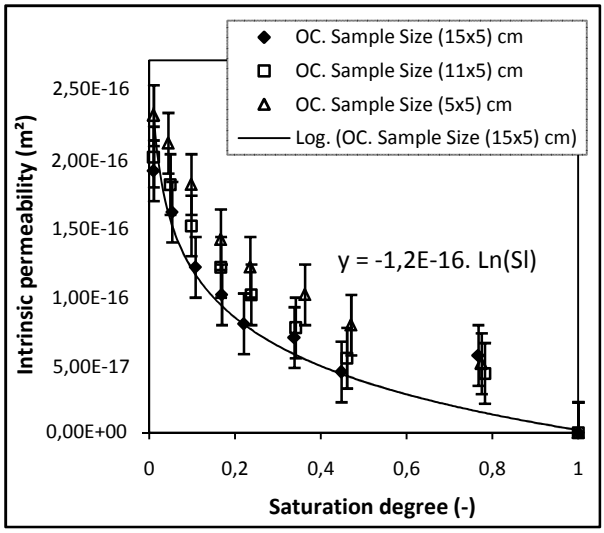

Figure 6: Intrinsic permeability depending on the degree of saturation

The intrinsic permeability curves as a function of saturation degree may be made by means of logarithmic curves. In figure 6 the logarithmic curve corresponding experimental results obtained with (15x5) cm samples, is presented.

3.3.2 Evolution of the Klinkenberg constant β of samples with different sizes

The coefficient β , known, Klinkenberg constant of all the test bodies (with different diameters; (15, 11 and 5) cm) are calculated by using equation (2) (see section 2.2). Figure 10 shows the evolution of the permeability constant β, for the different samples as function as saturation rate of concrete.

According to figure 7, the values of Klinkenberg constant β shows a continuous increase with the decrease on the degree of saturation. A straight line fit shows a good regression coefficient of 0,968.

Since all specimens tested in this study are of the same batch of concrete and thus a similar material, we could not distinguish between the values of Klinkenberg constant β of the three sets of specimen sizes, because β

is a constant which depends mainly on the porous structure of the material.

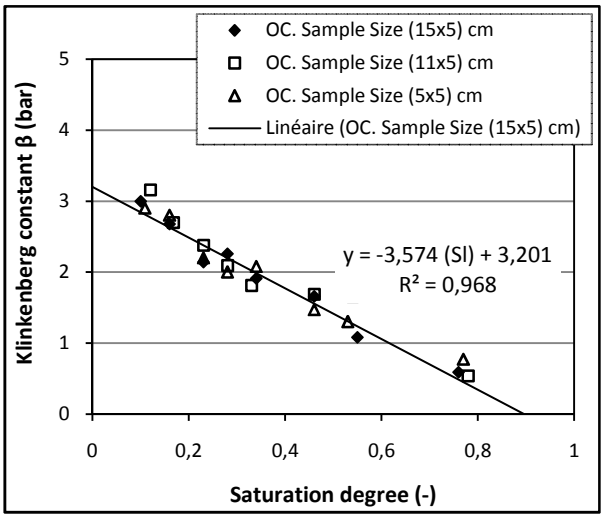

Figure 7: Evolution of Klinkenberg constant β as fonction as the saturation degree of the concrete

The linear fit for the concrete sample with size (15x5) cm is given in figure 7. Using the straight line fit, Klinkenberg constant β can be calculated by the following equation:

$$\beta(bar) = -3{,}574(S_l) + 3{,}201 \qquad (9)$$

3.3.3 Relative gas permeability of samples with different sizes:

The evolution of the relative gas permeability of test bodies with different sizes is shown in figure 8. The validity of the law according to Van Genuchten's relation (equation (8)) was checked with the relative permeabilities obtained with three different diameters and with different coefficients q.

According to the curves obtained (see figure 11), we observe that the relative gas permeability is influencing by size of the test body. K_{rg} increases when the size (diameter) of the test body decreases. For all the samples (with different sizes) and for a small variation of water saturation degree in the 0 – 0.2 range, there was a drastic influence on the relative gas permeability of the material (OC) by its moisture content. Rapid decrease of K_{rg} was observed when S_l increases, in this range ($0 < S_l < 0.2$). Also, figure 11 shows that more the coefficient q is less, more the model of Van Genuchten fits with the experimental results corresponding to the little test bodies.

Beyond a degree of saturation of 0.2 and with an intermediate range of S_l , there is a larger gap between curves and the modified Van Genuchten model does not fit with the experimental results of K_{rg}. This can be explained by the fact that, drying procedure in this part of study (dry at 70°C) modify the pore structure and may induce microcracking due to the dry shrinkage and then artificially increase gas permeability.

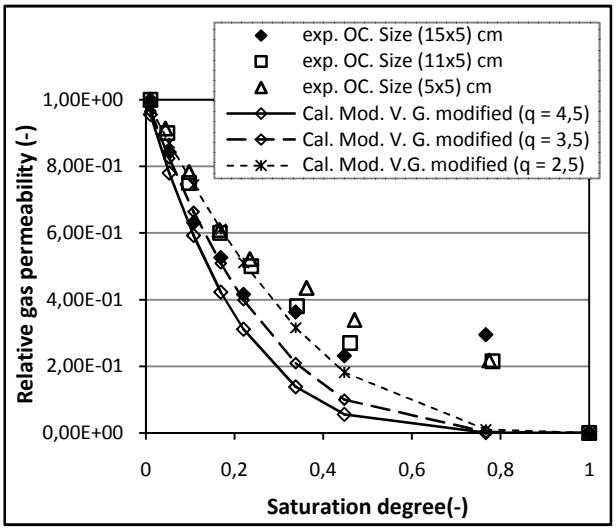

Figure 8: Relative gas permeability of different test bodies of concrete (with different sizes)

As shown in figure 8, K_{rg} is influenced by the size of the sample tested. We believe that the parameter q should depend not only on the nature of the material but also on the sample size.

3.3.4 Loss of relative density

The evolution of the loss in relative density of different samples with different sizes is given in figure 9. The drying protocol followed has covered the whole field of degrees of saturation (S_l) for the type of concrete studied (OC).

According to figure 9, the relative permeability of the large samples ((15x5) cm) based on the loss density is somewhat lower than those obtained with other samples (medium and small), confirming that the

drying is also influenced by the size of the test body.

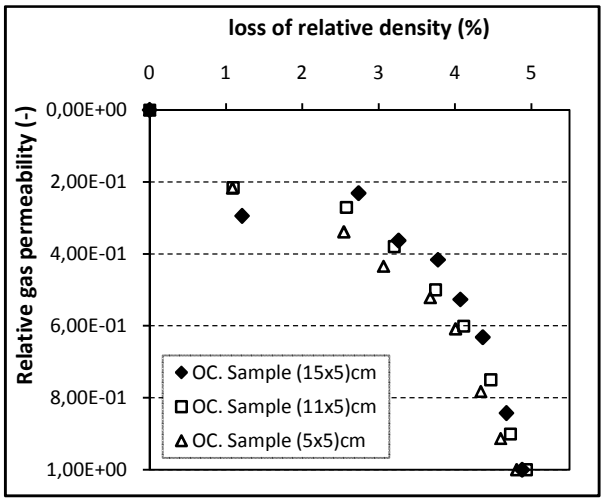

Figure 9: Relative gas permeability as a function of the loss of relative density

The relative permeability of the material increases gradually with the loss in relative density (masse/volume) of different test bodies. Their dry states correspond to about 5% relative weight losses (see figure 9).

4 CONCLUSIONS

In this study, with the apparent gas permeability obtained at different inlet pressures on our material (OC), it is possible to assess the "Intrinsic" gas permeability, which is independent of the applied mean pressure and which is associated to pure viscous transport, by using Klinkenberg's method.

The intrinsic coefficient of permeability can be a better parameter for characterization of a concrete for durability compared to the traditional Darcy's coefficient of permeability, as it is independent of the fluid properties and the applied pressure gradient. It is hence, a characteristic of the porous medium alone.

The different drying protocols followed has covered the whole field of degrees of saturation (S_l) for the type of concrete studied (OC). Therefore, preconditioning applied has a great influence on the measurement.

The experimental results show that the relative permeability of concrete is strongly dependent on the degree of saturation.

The originality of this study was to test concrete with different sizes to get an estimate of the scale effect. The results show that more the surface of the sample tested is large, more its permeability decreases. The sample size is an important parameter influencing the relative gas permeability of concrete.

One of the aims of this study was to determine if the relations given by Van Genuchten and based on the work of Mualem, largely validated for soils, can be used in predicting durability of our ordinary concrete which is partially saturated. Comparison between experimental values and calculated values using Van Genuchten's parameters obviously shows that the values of some parameters have to be revised. From a series of steps we have made, the analytical results obtained for the relative gas permeability with the Van Genuchten relations appear to be satisfactory for low saturation level (0 to 0.2), with values of q between 3.5 and 4.5 (often taken equal to 0.5 in the case of soils). However, the analytical formula of Van Genuchten amended does not seem appropriate for different specimen sizes and for different saturation levels. We conclude that the coefficient q depends not only on the nature of the material but also on the size of the sample.

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