Towards a harmonization of permeability measurements under pressure and in vacuum

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ABSTRACT

To measure the tightness of nuclear containment structures and their durability, air permeability measurement is of particular interest. It allows to estimate leakage rates quickly and has the advantage of being non-destructive. However, the devices used in the laboratory and those used in situ differ. The CEMBUREAU permeameter is the subject of a standard (XP-P18-463) whereas this technique is limited to the laboratory and requires a particular sampling and confinement, inadequate to the real structures. This implies that a core sampling of the structure is necessary but that is not always possible, hence the interest to develop new techniques of non-destructive measurement on site. In this perspective, many works have been carried out and several approaches have been apprehended. One of these techniques is the measurement of permeability by the Torrent apparatus which is widely studied today. These two techniques rely on different equipment and different physical phenomena. The CEMBUREAU permeameter operates under pressure, i.e., the gas passing through the sample is injected at a pressure higher than atmospheric pressure, whereas the Torrent permeability measurement is performed in vacuum. A standardization or at least a harmonization of the results from different tests is necessary for a better analysis and estimation of the durability of the structures.

Keywords: Permeability, CEMBUREAU, Torrent, Durability

1. INTRODUCTION

Controlling and guaranteeing the tightness of the containment of nuclear power plants is a worldwide challenge, and even more so in France, with more than fifty reactors currently in operation. Nuclear power plants are regularly inspected to verify their tightness in case of an accident. These conditions correspond to an increase in internal pressure and temperature of up to +0.5 MPa and 180°C respectively for two weeks.

To test the tightness, the reactor building (inner vessel) is pressurized to the design pressure and the leakage is evaluated by following the mass loss over time. At the same time, single leakage measurements are performed in the space between the reactor vessels using collection boxes. The difference between the global leakage rate leaving the enclosure and the rate associated with the singularities constitutes the diffuse leakage in the healthy zone. There are still large uncertainties in the evaluation of the singular leakage, which results in a poor estimation of the diffuse leakage. In addition, these tests are restrictive and require a high level of logistics and are only carried out about once a decade in France, which could prove problematic in the context of rapid leak detection.

The tests classically carried out in laboratory under pressure (CEMBUREAU), are destructive techniques, which require the taking of samples on site representative of the structure. However, the small size of the samples and the impossibility to come and core a power plant makes these tests obsolete. A technique developed in the 1990's, allows to make in situ measurements (Torrent, 1992). The Torrent permeameter, developed by the engineer of the same name, makes it possible to measure surfaces permeability in vacuum in a non-steady state, which is difficult to interpret. Moreover, the investigated zones are small in diameter (about 5 cm) and in depth. This raises

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questions about the representativeness of tests on walls that can reach several tens of centimeters of thickness, as is the case for nuclear power plants.
To propose a new interpretation of these under vacuum tests, a laboratory methodology has been developed. It consists in determining the apparent permeability of reference under pressure and steady state (CEMBUREAU) for several concretes in variable states of saturation, then to compare this value with those obtained in vacuum and steady state, then in vacuum and transient state, configuration which is the closest to the in-situ measurement Finally, correlations are proposed to harmonize the different techniques. Five measurement techniques will be presented here:

- Two techniques carried out in laboratory working in steady state (CEMBUREAU under pressure and a technique in vacuum). They consist in measuring an air flow through a cylindrical specimen confined in a cell and subjected to a constant pressure gradient.
- Two new laboratory approaches developed at LMDC operating in transient regime (simple cell with a vacuum time of sixty seconds and another with a longer vacuum time). They consist in measuring a pressure rise in a cell stuck to the surface of a confined specimen in which a vacuum has been initially made.
- An on-site measurement technique (Torrent, 1992) which works like the two previous ones in vacuum and in transient regime.

The reference value of the literature being that of the CEMBUREAU, the objective is to manage, from the four other tests, to go back to this permeability. The measurement from the Torrent shows still important differences, which originate from the technique or the interpretation of the results, whereas the four other tests lead, after calculation of the systematic error, to convincing results.

2. MATERIALS AND METHODS

In this part, the materials used, and the concrete mix-designs are presented. The protocols used as well as all the pre-conditioning of the samples are also explained, since these have a strong impact on the results.

2.1. Materials and conditioning prior to testing

The mix-designs were chosen so that the concretes studied cover the range of porosity representative of site concretes from the point of view of transport. The variation of the saturation state also allows to act on the porosity accessible to the gas and to widen the study range. The work is part of the global framework of the project Non-Destructive Evaluation of Nuclear Power Plant Containment (NDEN), the concrete formulated is the one used on Vercors (Multon et al., 2022), which is the 1/3 scale nuclear power plant model, representative of the double-walled containment of a 1300 MWe nuclear power plant. The two other mix-designs are: an ordinary concrete (OC) and a high-performance concrete based on CEM I (HPC). The formulations of the different concretes studied are presented in Table 1.

Table 1 Concrete mix-designs (in kg/m^3)

Constituents	NDEN	HPC	OC
Sand 0/4 rec GSM LGP1	830	-	-
Sand 0/4 R Garonne	-	858	941
Aggregate 4/11 R GSM LGP1	445	-	-
Aggregate 8/16 R BALLOY	550	-	-
Aggregate 5/12.5	-	945	-
Aggregate 4/14 R Garonne	-	-	1020
CEM I 52.5 NCE CP2 NF Gaurain	320	400	280
Techno 80 Sika plast	2.4	-	-
Masterglenium Sky 537	-	3	-
Chryso Optima 175	-	-	1.96

Total water	198	172	180

The concrete is poured into cylindrical moulds of 15 cm diameter and 20 cm height which are vibrated on a vibrating table in two layers. The covered moulds are stored in a humid chamber at 20 ± 2°C for twenty-four hours, the demoulding then takes place, and the samples are kept in lime water as soon as they are demoulded. To obtain a relatively stable material with respect to hydration, the duration of this water cure is fixed at 60 days minimum (Saul, 1951; Waller et al., 2004). The different permeability measurements were made on specimens in given states of saturation, given the strong dependence of this measurement on the internal humidity of the samples. The preconditioning adopted to limit the thermal damage and to reach the saturation states is the following (Carcasses and Abbas, 2001) start drying in a ventilated oven at 40°C until a degree of saturation of 80% is obtained, continue drying at 50°C until a degree of saturation of 45-40% is obtained, then drying at 60°C until a degree of saturation of 20% is obtained, then drying at 80°C until constant mass (less than 0.05% mass variation over 24 hours), Finally drying at constant mass at 105°C, reference for updating the real saturation states of the samples.

The objective of such a drying is to limit the part of cracking induced by too important hydric and thermal gradients. The capillary depressions generated by a complete drying lead to stresses exceeding the tensile strength of the concrete, resulting in uncontrolled cracking.

2.2. Characterization of the studied materials

The fresh and hardened properties determined on 3 samples are reported in the following Table 2.

Table 2 Characterization of materials in fresh and hardened states (standard deviation in brackets)

Concretes	NDEN	HPC	OC
Density ρ (kg/m3)	2417	2433 (17.54)	2390
Slump (mm)	215	190	-
Air content (%)	2.4	2.1	-
Compressive strength 60d (MPa)	46.8 (1.66)	57.8 (1.31)	40.2 (0.68)
Young Modulus (MPa)	39200 (1450)	38800 (444)	31700 (415)
Water accessible porosity Φ (%)	15.2	13.5	18.0

2.3. Steady-state measuring devices for in vacuum and under pressure

The CEMBUREAU permeameter is used to perform in vacuum and under pressure tests. In steady state, the pressure gradient is maintained until a constant flow rate is obtained. An air flow point is recorded every 5 seconds by a computer linked to a digital thermal mass flowmeter system to ensure that the steady state is established (Sogbossi, 2017). The apparent permeability at a given pressure is obtained using the Hagen-Poiseuille equation:

$$k_a = \frac{2\mu L P_2 Q_{v2}}{A(P_1^2 - P_2^2)} \quad \text{(Eq. 1)}$$

With: P1: the absolute injection pressure (Pa) P2: Atmospheric pressure (Pa), Q_{v2}: Flow rate measured at the sample outlet (m^3/s), k_a: Apparent permeability (m^2), A: Surface crossed by the flow (m^2), L: Length of the sample (m), μ: Dynamic viscosity of the fluid (Pa.s).

The apparent reference permeability k_{aref} is obtained from the flow rate Qv measured with the CEMBUREAU for an inlet pressure of 2 bar of absolute pressure, i.e., a pressure differential of 1 bar.

Figure 1. Cembureau apparatus for concrete permeability measurement in laboratory

2.4. Transient and steady state measurement devices under controlled flow conditions

To study the influence of the flow regime, the CEMBUREAU cell is also used in vacuum and in transient regime by making the time of vacuum in the upper volume, then by measuring the speed of pressure rise in the system. The pressure in the upstream volume (bell) is fixed at 15 mbar to represent the same vacuum as in the TPT[†] test. The following figure shows the schematic diagram of the CEMBUREAU apparatus in vacuum.

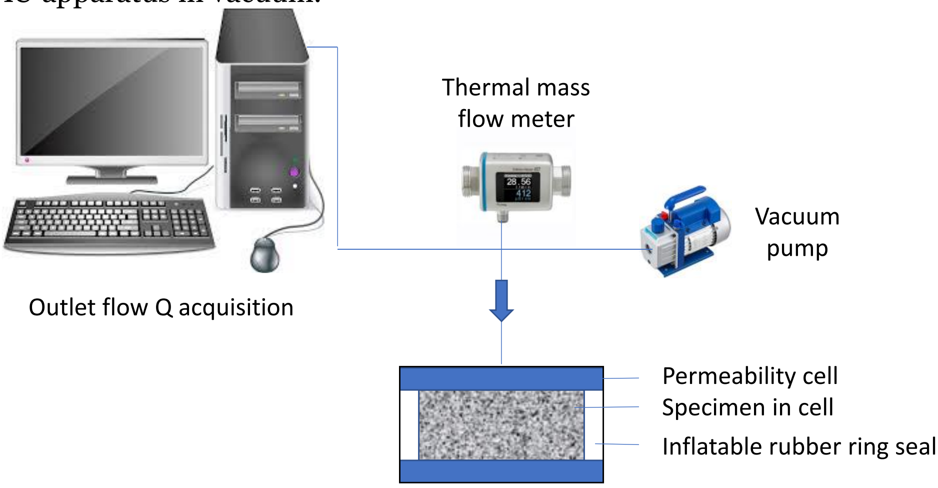

Figure 2. Cembureau apparatus for concrete permeability measurement in laboratory and in vacuum

Two vacuum times are studied: a time at least equal to the time of establishment of the steady state (TESS[‡]), which allows the use of the relation (Eq.2) and a time of 60 s, the same as in the TPT[§] test. In this case there is no analytical exact solution to the mass transport problem because the test takes place in transient regime. The solution can be approximated by a simplified method similar to that of Torrent or by a numerical approach using finite elements. The exact depth mobilized during the test with a vacuum time of 60 seconds is not known. The permeability calculation equation (Eq.3) must consider the thickness investigated during the measurement (Sogbossi, 2017). Sogbossi proposed to evaluated the apparent permeability in transient conditions by (Sogbossi, 2017.):

$$k_{ac} = \frac{2\mu L}{A(P_a^2 - P_c^2)} V_c \dot{P}_c \qquad \text{(Eq. 2)}$$

With k_{ac}: permeability coefficient (m^2) according to the bell method, P_c: Pressure in the bell (Pa), $\dot{P}_c$: Initial velocity of pressure rise in the bell, V_c: Volume of the bell (m^3), A: Cross-section of the specimen (m^2).

$$k_{aT0} = \frac{8\mu}{\emptyset}\left(\frac{V_c}{A}\right)^2 \frac{P_a}{(P_a^2 - P_c^2)^2} \dot{P}_c^2 (t_v + t) \qquad \text{(Eq. 3)}$$

[†] Time Permeameter Torrent
[‡] Time of Establishment of the Steady State
[§] Time Permeameter Torrent

t_v: vacuum time; t = time at which the ascent rate is taken; ∅ air accessible porosity of the concrete (%).

Due to the great thickness of the walls of real structures, and in particular for the containments of nuclear power plants, the vacuum time required is too long to perform a permeability measurement under controlled conditions. The main difficulty is then to evaluate the concrete depth impacted by the air flow. In this approach, Torrent proposed to evaluate this depth, L_o, from the mass balance of the moles of air passing through the concrete to reach the central cell during the Eq. 4:

$$L_0 = \sqrt{\frac{2 \cdot k_{a_t} \cdot P_{atm} \cdot (t_v + t)}{\varphi \cdot \mu}} \quad \text{(Eq. 4)}$$

with: k_{at} the unknown permeability of concrete crossed by the air flow, t_v, the vacuum time, t, the time after the stop of pumping, φ, the porosity of concrete and μ, the air viscosity.

Equation (Eq.3) is used for short vacuum times or low permeability concretes, if the total thickness of the sample is not crossed by the air flow. It allows to estimate the permeability, because of the pressure profile present in the thickness at the time of the measurement. This effect fades as the measurement progresses, which is why the rate of ascent is taken at the end of the pressure acquisition curves. Finally, this equation can also be used on the upwellings obtained with the Torrent permeameter, which allows to compare permeability obtained by the various approaches.

2.5. In-situ Torrent permeameter

The Torrent permeameter creates a vacuum for 60 seconds in two concentric bells, one which will be called the inner bell and the other the outer bell. The inner bell is used to make the measurement and the outer bell to confine the flow in a single direction. The depression is then maintained in the external bell and cut in the internal. With the help of a membrane the pressure difference between the bells is kept constant. The recording of the pressure rise in the inner bell is carried out continuously at intervals of 20 mbar and for a maximum duration of 12 minutes. All the details are given in (Torrent, 1992).

Figure 3. Torrent apparatus for concrete permeability measurement in field

3. RESULTS

All tested configurations are listed in the following table. To facilitate the understanding of the reader, especially concerning the graphs and their interpretations, we recommend that the reader refer to this table.

Table 3. Names and explanations of the acronyms used for the results obtained

Type of permeability	Type of pressure	Type of state	Vacuum time	Designation
Apparent permeability measured	Under Pressure	Steady State	Time of Establishment of the Steady State	$k_{aref\ UP\ SS}$
Apparent permeability measured	In Vacuum	Steady State	Time of Establishment of the Steady State	$k_{a\ IV\ SS}$
Apparent permeability measured	In Vacuum	Transient State	Time of Establishment of the Steady State	$k_{a\ IV\ TS}$
Torrent permeability measured	In Vacuum	Transient State	60 seconds	$K_{T\ IV\ TS\ corrected}$

3.1. Permeability in steady states: CEMBUREAU under pressure and in vacuum

Figure 4 shows the relationship between the reference measurements, k_{aref}, and permeability measured under vacuum with the same equipment: the CEMBUREAU. Based on the relation of Klinkenberg (Klinkenberg 1941), a coefficient of pressure is used (Sogbossi et al., 2019) to relate k_{aref} to the apparent permeability obtained in vacuum for the various states of saturation. This coefficient depends theoretically on the characteristic size of the pore network. In this study, it can be evaluated at 0.56, as shown for the experimental measurements (Fig. 1). In order to evaluate the air leakage of a structure under pressure, it is therefore necessary to determine both the intrinsic permeability and the slope of Klinkenberg's law which are given by the following equation:

$$k_a = k_i\left(1 + \frac{b_k}{P_m}\right) \qquad \text{(Eq. 5)}$$

With k_a, the apparent permeability, k_i, the intrinsic permeability, P_m, the mean pressure between the atmospheric pressure and the pressure of the test, and b_k, the Klinkenberg gas slippage factor.

For low permeability, i.e., below 2.10^{-17} m², the results are scattered. This is associated with measurements in vacuum and with measuring devices which are then used at their limit in terms of flow measurement. This is also because the pressure gradient in vacuum is lower than the gradient obtained for measurement under pressure.

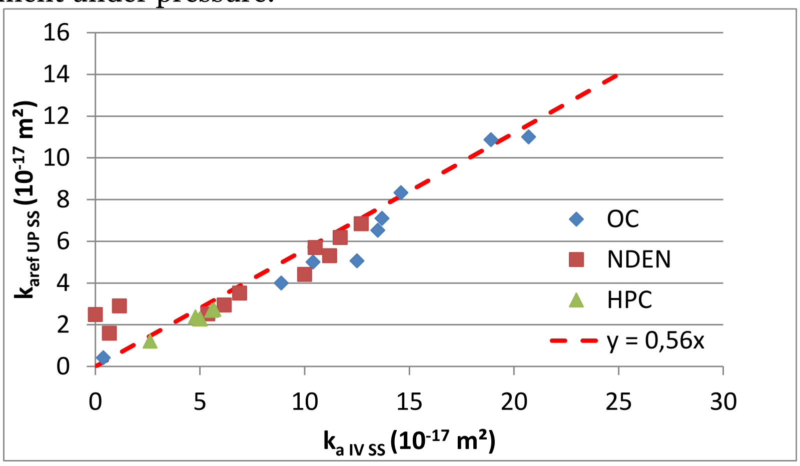

Figure 4. Apparent permeability at CEMBUREAU, k_{aref} Under Pression Steady State ($k_{aref\ UP\ SS}$) vs. apparent permeability k_a In Vacuum Steady State ($k_{a\ IV\ SS}$) P=15mbar

These results show the possibility of switching from apparent permeability under pressure to apparent permeability in vacuum, and vice versa, in the context of unidirectional steady state testing.

It is therefore possible to make accurate predictions of permeability results from either under pressure or vacuum tests. In the case of nuclear power plants, it is then possible to find the normative value under pressure from tests under vacuum.

3.2. Vacuum measurement: steady state and transient state after a vacuum time = TESS

Figure 5 shows the apparent permeability under vacuum and steady state conditions as a function of the apparent permeability under vacuum and transient conditions calculated by the previous equation Eq. 2, since the vacuum time is long, and the sample is crossed by the air flow. The graph seems to show that the type of regime does not influence the permeability results since the relationship between the two results is the line of equality. Obviously, this must be moderated for low permeability which are highly affected by experimental artefacts in this range of measurements.

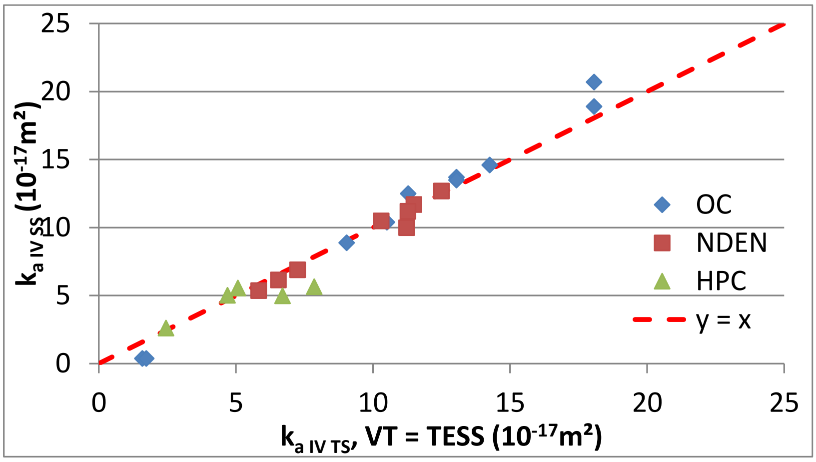

Figure 5. Apparent permeability in vacuum and steady state ($k_{a\ IV\ SS}$) and in vacuum and transient state ($k_{a\ IV\ TS}$) with a vacuum time = TESS

These results show the possibility to go from the apparent permeability obtained in transient to the apparent permeability obtained in steady state for a measurement in depression. In view of the conclusions given in part 3.1, it is therefore possible to use the apparent permeability in transient regime with a long vacuum time measured in vacuum to find the measurement at CEMBUREAU.

3.3. Vacuum measurement: transient state after a vacuum time = TESS and a vacuum time = 60 s.

Figure 6 shows the permeability obtained in vacuum and transient conditions after a vacuum time equal to the steady state time (TESS) and a vacuum time of 60 seconds (as used for in field apparatus). In this case, the flow can cross the thickness of the sample during the vacuum. If it happens, equation 2 is used to calculate the permeability if it does pass through or equation 3 if it does not. Therefore, if the L_0 calculated from equation 4 is greater than the thickness of the sample, equation 2 is used, otherwise equation 3 is used. The curve shows some deviations, especially in the central area of the graph, i.e., for permeability values between 5 and 15.10^{-17} m². However, the general trend is never more than 30% away from the equality line. In view of the possible deviations for the same permeability measurement on one sample, this is within the measurement uncertainty. Moreover, when drying creates additional percolation, the difference in analysis area as a function of the duration of the vacuum leads to the development of local heterogeneities that can create dispersion.

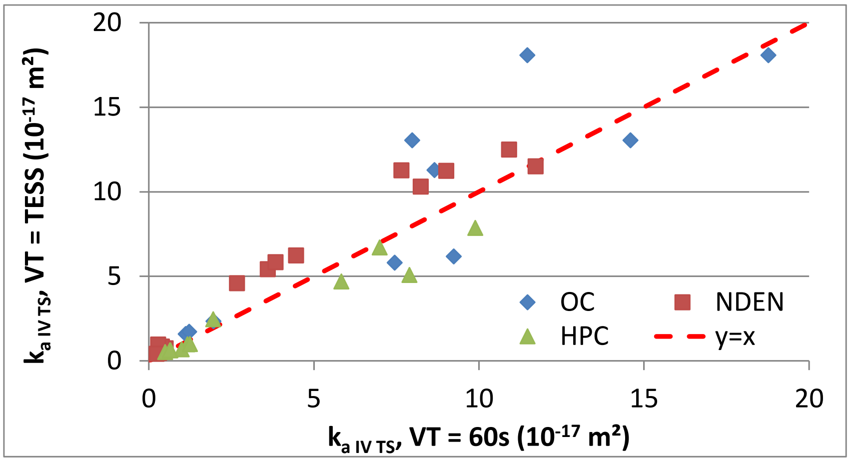

Figure 6. Apparent permeability in vacuum and steady state and in vacuum and transient state with a vacuum time = 60 s

These results show the possibility to switch from the apparent permeability obtained in transient regime and in vacuum for short or long vacuum time. Because of the previous conclusions, exposed in 3.1 and 3.2, within the framework of a 1D measurement in laboratory, i.e., with a good control of the direction of the flow, it is possible to determine the traditional CEMBUREAU measurement and thus a normative value.

3.4. Measurement with the Torrent permeameter and in vacuum and in transient regime after a vacuum time = 60 s in laboratory cell.

The previous steps have shown the possible correlation of the different techniques for the estimation of a reference permeability, from the CEMBUREAU and the previous equations. The final objective is to make the link between the *in-situ* measurement technique, the Torrent permeameter, and the tests carried out in the laboratory, those which are the closest to this technique, i.e., in transient regime after a vacuum time of 60 s. The permeability calculated from the Torrent permeameter ($k_{T\ IV\ TS\ corrected}$) considers the pressure rise rates provided by the Torrent permeameter combined with the equations proposed previously (Eq. 2 and Eq. 3 depending on the depth investigated) and corrected for the real porosity. These values are compared to the k_{ATo} or k_{aC} determined in (Sogbossi et al., 2019).

What emerges from this graph is that the results obtained by the Torrent permeameter are almost systematically inferior to those obtained by the CEMBUREAU. This translates into experimental results below the equality line (Figure 6)

In addition to the fact that the analysis area is not the same between the Torrent (5 cm diameter cylinder) and the CEMBUREAU (15 cm diameter cylinder), which can lead to errors in representativeness due to possible heterogeneities, it is difficult to consider the evolution of pressure in the thickness of the samples in a transient regime. It is possible that very porous paste/aggregate interfaces are over-represented in the case of small diameter samples, which is the case of the Torrent permeameter. Moreover, parasitic fluxes are involved in the Torrent permeameter measurement, whereas they are controlled in by the CEMBUREAU cell by the rubber ring (Figure 2) for the measurement in laboratory. Their estimation is being investigated with numerical simulations of the Torrent permeameter test. The modelling is based on the equations for the transport by permeability of air developed by Verdier et al. (Verdier et al., 2002). After calibration, the pressure isobars in the concrete samples were plotted to assess the direction of airflow during the test. The air flow is perpendicular to the lines shown. The isobars were nearly perpendicular to the axis of symmetry of the central cell throughout the pressure rise, as assumed by analytical methods (Sogbossi, 2017; Torrent, 1992) with some inflection below the joint between the cells. All the results are presented in Figure 7.

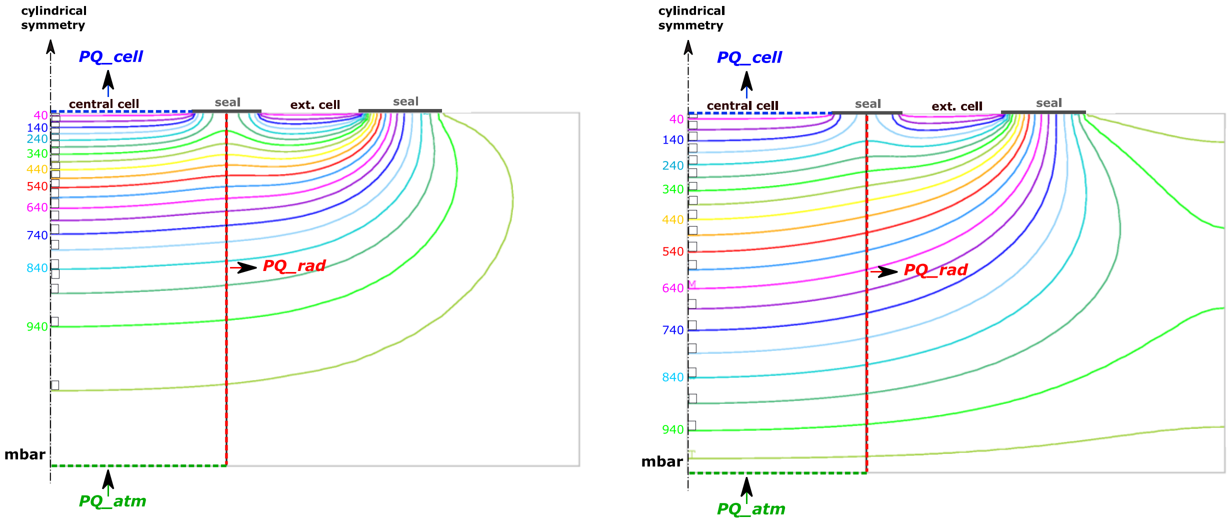

Figure 7. Isobars in the concrete sample (50 mm thick). On the left at the end of the period of vacuum (60 seconds), on the right at the end of the period of measurement (330 seconds after the end of the vacuum in the central cell) for a sample of concrete at 30% of saturation

The model can then be used to evaluate the part of the radial flow entering the investigated area and assumed to be zero by the analytical methods. For this purpose, three mass fluxes were determined by the modelling during the pressure rise: PQ_cell, the flux entering the central cell, PQ_atm, the flux passing through the investigated surface of the sample on the face at atmospheric pressure, and PQ_rad, the air entering (negative flux) or leaving (positive flux) the investigated volume along the radial direction (Figure 7). Figure 8 represents the evolution of the three fluxes during the pressure increase throughout the test. At the end of the 60 seconds of vacuum, once the pump is stopped, the flow entering the cell is maximal: the air present in the concrete porosity before the test enters the cell, it is at this moment that the pressure gradient is maximal. Gradually, the air must come from further into the concrete, the air flow entering the cell decreases and the air flows through the other surfaces increase. For a thin sample (50 mm) with a low degree of saturation (30%), the three flows change little after 200 seconds of testing.

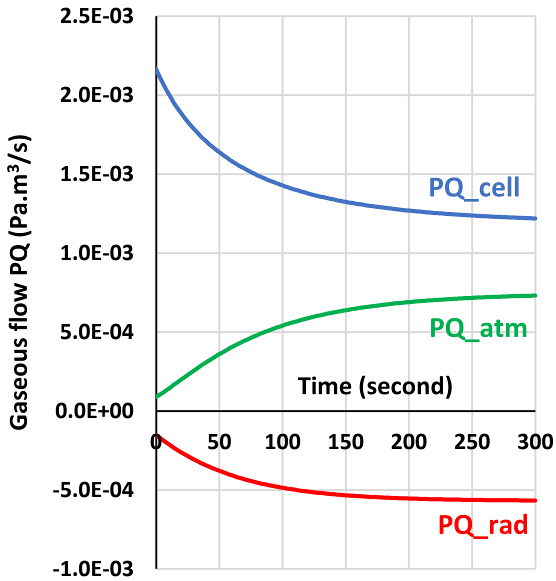

Figure 8. Gaseous flows during the pressure increase: in the cell, PQ_cell, at the external surface, PQ_atm, and at the mean radius between the two cells, PQ_rad, for one sample of concrete at 30% of saturation

To take all this into account, and in particular the through-flow, Torrent corrects its measurement by a double flow when the flow crossed the samples (which corresponds to the case when the investigated thickness indicated by the apparatus is larger than the thickness of the concrete element).

4. RESULTS AND CONCLUSIONS

The permeability under pressure or in vacuum in steady state with the CEMBUREAU cell give the same results whatever the type of concrete (Figure 4). This means that the type of measurement does not influence the permeability results. Nevertheless, the possible measurement range is smaller in terms of saturation for vacuum test due to the lower pressure gradient than under pressure.

The permeability in vacuum in permanent or transient regime with the CEMBUREAU cell also give similar results (Figure 5) whatever the type of concrete. The regime does not influence the permeability results if the air flow direction is controlled by an external cell in laboratory.

The permeability in vacuum after a long vacuum time (vacuum time to reach the steady state) and a vacuum time of 60 seconds give once again the same results (Figure 6) in laboratory cell. This means that a vacuum of 60 seconds is sufficient to estimate the permeability of the material.

The problem is the comparison between the results of permeability in vacuum in transient regime between the Torrent permeameter and the laboratory cell. There is a slight tendency to overestimate the permeability with the Torrent permeameter. The size of the samples and the parasitic flows present on these tests could partly explain this difference, as developed in section 3.4. A corrective factor of 0.6 is proposed for the moment to consider the parasitic flow and thus recover the good correlation between the two measurement techniques which seems to provide satisfactory results.

Figure 9 groups all the measured or calculated permeability values in relation to the reference value taken as being the value under pressure and in steady state. Some values have been corrected for the effects of the pressures and depths investigated, those measured with the Torrent permeameter after a vacuum of 60 seconds. From Figure 9, it seems possible to relate the permeability in vacuum and under pressure. The proportionality around the straight line of equality between the reference points and all the other measurements, whether under pressure, in vacuum, in steady state or transient, is well observed. Most of the experimental points are within a range of 50% of the mean, which is still within the uncertainty due to the heterogeneity of the concrete and the degree of saturation.

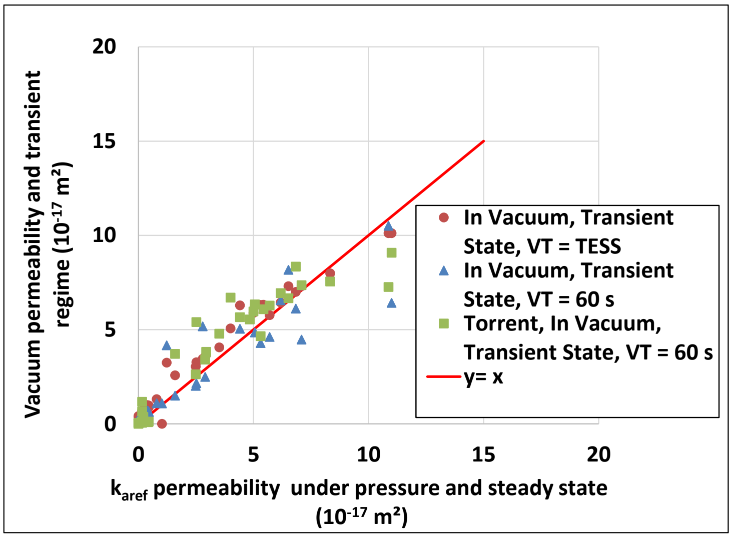

Figure 9. Corrected permeabilities obtained by the different measurement techniques in vacuum and in transient state as a function of the reference permeability obtained under pressure and in steady state.

Based on these results, the objective now is to be able to carry out in situ tests on larger surfaces to be representative of the structure. The extension of the void time and the increase of the size of the apparatus to investigate larger depths as well as the consideration of the saturation profile in the thickness of the studied structure are avenues of improvement of these tests.

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