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GAS PERMEABILITY MEASUREMENT OF POROUS MATERIALS (CONCRETE) BY TIME-VARIABLE PRESSURE DIFFERENCE METHOD

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ABSTRACT

The theoretical approach for the determination of specific gas permeability coefficient in the time-variable pressure difference conditions of gas flow through porous material is presented. The obtained theoretical results are experimentally verified by the use of suitable modified and improved standard laboratory equipment. Method, involving variable gas pressure, is compared with standardized constant pressure method and a very good agreement was obtained.

Introduction

Fluid permeability of building materials, especially of concrete, although it represents a measure of the fluid flow (gas or liquid), is perhaps, the only direct measurable parameter that is responsible for the structure durability. Therefore, the knowledge of the specific fluid permeability coefficient of certain porous material is as important as it is for instance, its density or strength.

If the measurement of flow is performed by gas, for example, air, oxygen or nitrogen, instead of liquid, it is known as gas permeability. These methods, which use gases for determining permeable porosity are usually very simple and are very often used. According to fluid dynamics, flow theory is the same for gases and liquids, so from this point of view, there is no difference if liquid or gas is used. However, usual building materials interact more often chemically and physically in contact with liquids than with gases. These "flow difficulties," become very serious in the special but numerous cases when liquid is water and porous material concrete. Namely, water-concrete interaction includes appearances such as hydration, solubility (gases, liquids and solids), surface tension and so on. Additional problem in the case of concrete is its tiny permeable pore dimensions, which assumes application of relatively high liquid pressure to obtain measurable flow, because of high viscosity as compared to gas. All circumstances show

that in most permeability measurements, especially in concrete, is better (sometimes the only possible) to do them with gases than liquids.

Gas permeability measurement methods, according to the pressure difference across the porous media, can be divided into two groups. One group of experimental techniques involves constant pressure difference while the other uses in time variable gas pressure difference. The examples of constant pressure laboratory devices are firstly the permeability apparatus by Lea and Nurse(15), then Žagar's(4) and finally standardized devices(5,7,11). On the other hand, are experimental techniques involving variable gas pressure, that were independently designed by Blaine(16) and Rigden(17) and that are intended for determination of specific surface and particle size of granular samples (cement powder, stone powder, sand, etc.). Although these measuring devices were made for granular media, there is no reason, that method could be also applied to solid porous materials, for example concrete and cement stone. Namely, the principle of variable pressure measuring method is to produce pressure, different from surrounding, in a chamber and then to measure the time drop of pressure in this chamber, due to fluid flow through porous material. The advantage of this method is that there is no need to measure the gas flow, as the rate of pressure drop is known. This advantage is significant, because direct flow measurements could be very complicated, even in laboratory surroundings. Secondly, instead of measuring two parameters (pressure and flow) there is to be measured only one (pressure). Further experimental simplification of variable pressure method is that different pressure must be applied only shortly, just to establish different pressure before the measurement starts, while in constant pressure condition permanent source of compressed gas is needed.

Theoretical Approach

Theoretical model for calculating the specific gas permeability coefficient, derived among the first Žagar(4,9) in his works. According to that, specific gas permeability coefficient K_g (in references(3,4,5,7,9) marked as D_s) is given by the equation (considering assumption that the pressure difference is small enough so that we can neglect the influence of gas compressibility):

$$K_g = \frac{\eta \cdot L \cdot Q}{A \cdot \Delta p} \quad (a)$$

where,

K_g	- specific permeability coefficient	(cm^2)
Q	- flow	(cm^3/s)
Δp	- pressure difference $p_1 - p_2$	(Pa)
p_1	- input pressure	(Pa)
p_2	- output pressure (atmospheric)	(Pa)
L	- thickness of the sample in the flow direction	(cm)
η	- dynamic gas viscosity	(Pa·s)
A	- area of the sample perpendicular to flow	(cm^2)

The time variable pressure difference measuring method starts in moment when some pressure, different from surrounding, is established in a closed chamber, in which one wall is a specimen of porous material. Gas flow lasts as long as there exists pressure difference. On the assumption that inside the chamber are isothermal conditions fulfilled, Boyle-Mariotte's law is valid in every moment:

$$P \cdot V = \text{konst.} \quad (b)$$

By differentiation of product (b) with respect to time, satisfying initial (boundary) conditions that the chamber volume is V_k and the pressure P_k , connection between fluid flow (dV/dt) through porous media and pressure change (dP/dt) in the chamber can be obtained:

$$\frac{dV}{dt} = -\frac{V_k}{P_k} \cdot \frac{dP}{dt} \quad (c)$$

(The constant V_k/P_k is proportional to gas compressibility)

On the other hand, watching the permeation of gas through porous specimen thickness L and cross-section A , during some very small time interval dt when the pressure change of Δp can be neglected, it is possible to write according to (a):

$$Q = \frac{dV}{dt} = K_g \cdot \frac{A \cdot \Delta p}{\eta \cdot L} \quad (d)$$

If $\Delta p = P$, (to be consistent with (c)) and substituting (d) into (c) we get

$$\frac{dP}{P} = -K_g \cdot \frac{A \cdot P_k}{\eta \cdot L \cdot V_k} \cdot dt \quad (e)$$

The proportionality constant on the right side of equation, as a simplification, we may denote Ω (1/s):

$$\Omega = K_g \cdot \frac{A \cdot P_k}{\eta \cdot L \cdot V_k}, \quad (f)$$

Integrating both sides of (e) we obtain:

$$\ln P = -\Omega \cdot t + \text{const.} \quad (g)$$

Since we are given $P=P_i$ (initial pressure difference) at $t=0$, so $\text{const.}=\ln P_i$. Solving for P (pressure difference at moment t), we have

$$P = P_i \cdot e^{-\Omega \cdot t} \quad (h)$$

There are two possibilities for absolute pressure's relation in solution (h). The first is that the beginning pressure p_i is greater than surrounding atmospheric pressure p_o ($p_i > p_o$). In that case $P=p - p_o$, where p is absolute pressure at moment t , and $P_i=p_i - p_o$, for p_i absolute initial pressure. So equation (h) becomes

$$p = (p_i - p_o) \cdot e^{-\Omega \cdot t} + p_o, \quad p_i > p_o \quad (i)$$

The second case is for $p_i < p_o$, p_i is a sub-pressure. Expressing as absolute pressures we get, $P= p_o - p$ and $P_i= p_o - p_i$. Then the solution (h) is

$$p = p_o - (p_o - p_i)e^{-\Omega \cdot t}, \quad p_i < p_o \quad (j)$$

The obtained solutions (i) and (j) indicate that the pressure in a closed vessel that has a permeable wall, exponentially changes in time at rate Ω , from p_i to p_o . It is obvious that

the only experimental job is to measure just one parameter, the pressure change in time. The constant Ω must be determined, because specific gas permeability coefficient K_g is inside it as a factor. The simplest way to do that is to use the logarithmic expression (h) inside the time interval, from t_1 to t_2 and corresponding pressures p_1 and p_2

$$K_g = \frac{\eta \cdot L \cdot V_k}{A \cdot P_k} \cdot \frac{1}{t_1 - t_2} \cdot \ln \frac{p_1}{p_2}, \quad \text{where } p_1 > p_2 \quad (k)$$

The other, more reliable possibility is to obtain the experimental data curve, pressure-time, by measurements and then using non-linear regression analysis (fitting method) of that curve to the function of the shape (i) or (j), find "the rate" Ω and finally K_g via equation (f). These procedures can be performed by computer with a proper program for such analysis.

Permeability Measurements

The main aim of experimental work was to verify the presented theoretical model. The first step was to design the measuring device so that former by stated conditions were satisfied. This means to have porous specimen in some closed chamber with constant volume, which can withstand some pressure different from surrounding. It was found that such conditions are achieved if standard measuring cell was slightly modified (Figure 1.). The pressure (vacuum) chamber, as Figure 1. shows, is the bottom part of the cell. The electronic manometer was mounted in it and electrically connected to computer, so the pressure can be permanently measured.

The whole equipment is shown in Figure 2. The outlet of the cell is connected to vacuum pump via stopcock. The other connection on the cap of the cell was left open to surrounding atmospheric pressure. The compressed air bottle is used for radial sealing of the concrete sample. The analog electric potential of pressure gauge was digitized by AD converter and connected to PC communication port. An application program was made,

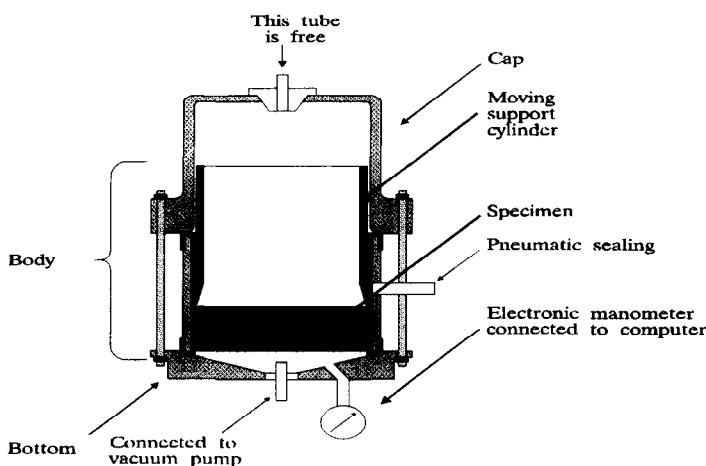


FIG. 1.

Modified permeability measuring cell*

*standard measuring cell is constructively improved, because it is equipped with the moving support cylinder which enables the measurements of samples with different thickness.

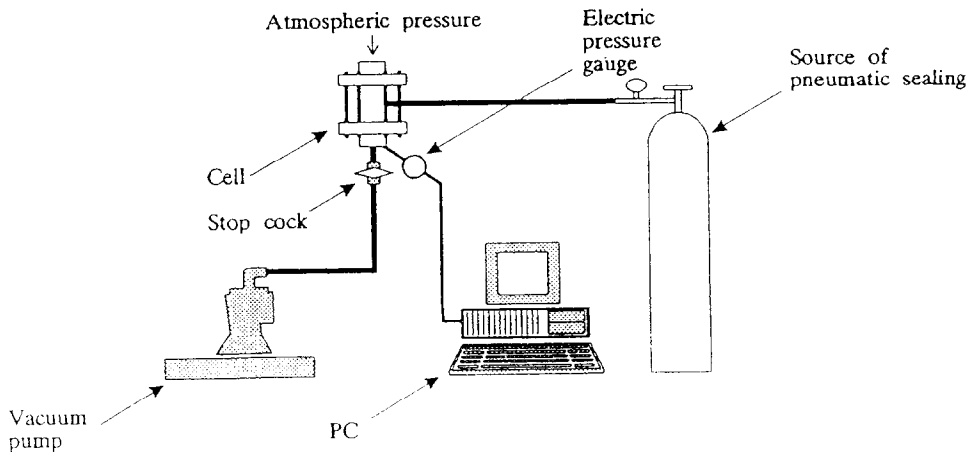


FIG. 2.
Variable pressure permeability equipment.

which task was drawing pressure-time curve and then according to input measuring parameters (height and diameter of sample, volume of vacuum chamber, pressure and dynamic viscosity of air) calculating, in some time intervals, permeability coefficient K_g (via equation (k)) and finally creating data file for further analysis of results. Figure 3. shows PC monitor during the measurement.

Directly calculated K_g (via equation (k)), which is appearing on the screen during the test (Figure 3.), is obtained on the basis of measuring datas inside the specific time interval and serves just for control during the measurement. Therefore more reliable way to calculate K_g is the postpone analysis of the whole pressure-time curve, using data file, what was mentioned at the end of former section.

Concrete Specimens

Parallel measurements by constant and variable pressure method were performed in the same measuring cell on series of thirty plain laboratory-made concrete specimens of different but usual compositions (cement content varies from 200 to 300 kg/m³, water-

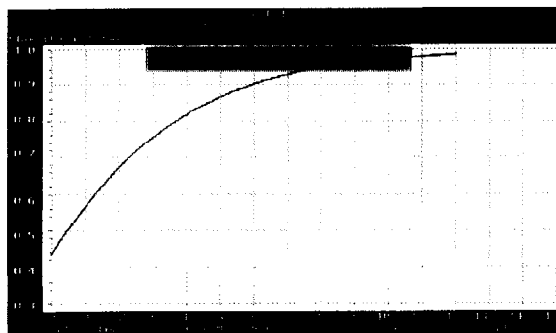


FIG. 3
PC monitor during the measurement

cement ratio (w/c) from 0.45 to 0.65). Specimens were cylinders with nearly the same diameter ($d \approx 100\text{mm}$), and various heights (L) in range from about 25 to 35mm. The concrete specimens were standard conditioned in the laboratory and dried before the testing, at 105°C till constant mass was achieved.

Results

Typical pressure-time curves of some various samples are shown in Figure 4. The corresponding specific air permeability coefficients (K_g) for presented curves, were in approximate range from $5 \cdot 10^{-13} \text{ cm}^2$ to $2 \cdot 10^{-11} \text{ cm}^2$ (Table 1.).

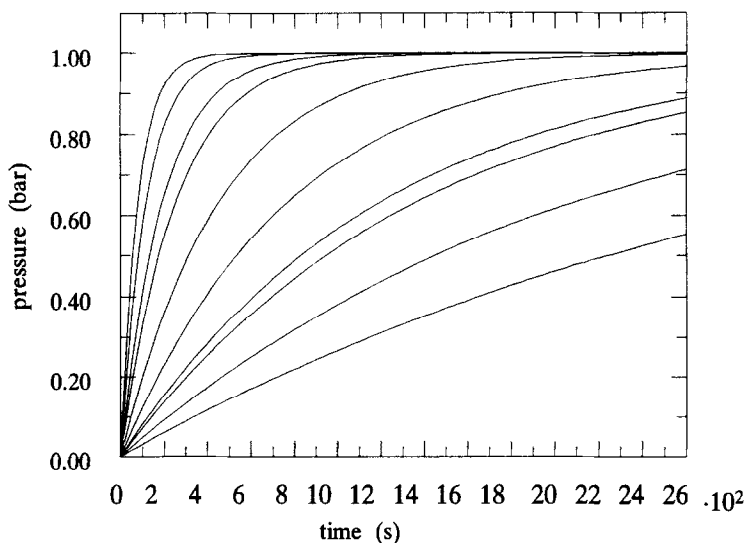


FIG. 4.
Pressure-time curves of different permeable concrete samples(28).

As the pressure-time curve was taken and the experimental data file created, obtained points were analyzed by non-linear regression method. Fitting was made to the equation shaped like (j), for $p_i \approx 0$ and $p_o = 1$ bar (atmospheric pressure), which is called "1st order rate" and which has the general form, $y = 1 - \exp(-\Omega t)$. So argument of exponent, Ω was calculated and then by equation (f) specific air permeability coefficient K_g . The obtained results are given in Table 1. It was found that experimentally pressure-time data curve fits the theoretical function shaped as equation (j), within standard error of about 0.5%. This may be a good reason to believe that theoretical model is correct. In the last column of Table 1. the correspondent results obtained by constant pressure method are presented. The correlation of specific air permeability coefficients measured by constant and variable pressure method is shown in Figure 5. It can be seen in Figure 5. that the methods are in a good agreement. The standard error, calculated by linear regression analysis, for the slope coefficient was less than $\pm 5\%$.

Conclusion

Applying the method of variable pressure for determination of the specific gas permeability coefficient of porous solid concrete, it was shown that this method could be

TABLE 1.

Specific Gas Permeability Coefficients Measured by Variable* and Constant Pressure Method.

**constants: dynamic viscosity of air at 20°C, $\eta=1.89 \cdot 10^{-5}$ Pa·s, vacuum, $P_k=98100$ Pa, chamber volume, $V_k=231$ cm³*

SPECIFIC GAS PERMEABILITY COEFFICIENT					
Variable pressure method				Constant pressure	
sample	height L(cm)	diamet. d(cm)	$\Omega \cdot 10^{-3}$ (1/s)	Kg·10 ⁻¹² (cm ²)	Kg·10 ⁻¹² (cm ²)
T231	2.97	9.82	1.23	2.143	1.419
T231A	3.22	9.82	0.613	1.161	0.781
T232	2.90	9.85	1.22	2.063	2.224
T232A	3.15	9.85	0.480	0.882	0.985
T233	3.21	9.85	0.893	1.675	1.385
T233A	3.15	9.89	0.737	1.345	1.193
T331	2.78	9.84	5.25	8.545	5.138
T332A	3.29	9.85	2.23	4.277	3.180
T337	3.00	9.86	8.61	15.062	12.568
T337A	2.40	9.86	1.37	1.921	3.510
T338	3.26	9.84	2.48	4.731	7.210
T338A	3.06	9.86	1.27	2.256	6.365
T339	2.82	9.86	12.7	20.792	22.015
T339A	3.34	9.86	2.44	4.746	3.644
T346	2.83	9.84	3.32	5.492	6.365
T346A	2.91	9.86	1.83	3.097	2.412
T347	2.77	9.85	3.98	6.437	5.934
T347A	3.21	9.86	1.31	2.447	2.268
T348	3.31	9.84	2.18	4.219	4.092
T348A	3.27	9.85	1.43	2.733	2.667
T431	3.46	9.84	0.938	1.898	1.582
T432	3.47	9.84	0.840	1.706	1.300
T432A	3.53	9.84	0.692	1.429	1.028
T433	3.53	9.86	0.996	2.048	1.732
T433A	3.27	9.86	0.596	1.136	0.903
T448A	3.12	9.85	0.845	1.539	2.167

used in the same manner as standardized method of constant pressure. The advantage of that method was its convenience (measurement of only one parameter that was easily converted in electric signal) that the measurement could be aided by computer (example of Computer Aided Testing (CAT)). This means fully automatic determination of specific gas permeability coefficient. The variable pressure equipment was tested on concrete specimens whose specific permeability coefficients were in approximate range from 10^{-13} to 10^{-11} cm². It was found that the empirical pressure-time curve fit theoretical function within the standard error of 0.5% and the error of gas flow measurement by standard

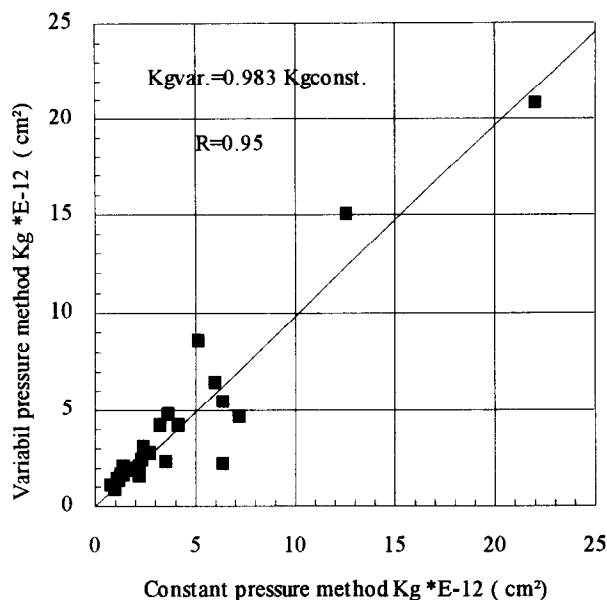


FIG. 5.

Correlation of K_g measured by constant and variable pressure difference method (28)

method rarely drops below 1%. There exists a good reason to believe that this method by the use of appropriate measuring equipment, can be applied "in situ" (on the structure) also. In that case the additional experimental problems are humidity, temperature and the inside volume ("specimen size and geometry") of concrete which is involved in measurement. If this problems could be solved, this method would be among the very few of which results "in situ" are directly comparable with laboratory method.

References

1. D'Arcy H.P.G., Les fontaines publiques de la ville de Dijon, Dalmont, Paris, 1856.
2. Carman P.C., Flow of gases through porous media, Butterworth Scientific Publications, London, 1956.
3. Manegold E., Kapillarsysteme Bd.1, Straßenbau, Chemie und Technik, Heidelberg, 1955.
4. Žagar L., Arch. Eisenhüttenwesen, 26, 777 (1955).
5. DIN 51058. 1. Ausgabe, Juni 1963.
6. Schwiete H.E., Ludwig U., Tonind. Ztg., 90, 562 (1966).
7. TGL 21094/12, Gruppe 152 00, September 1975.
8. Barrer R.M., Grove D.M., Trans. Faraday Soc., 47, 826 (1951).
9. Žagar L., Habilitations-Schrift, RWTH, Aachen (1957) 51.
10. Därr G.M., Ludwig U., Materials and Structures, 6, 185 (1973).
11. BS 1902, Section 3.9, 1981.
12. Hug S., Dissertation, RWTH, Aachen (1971) 97.
13. Carman P.C., Trans. Inst. Chem. Eng., 15, 150 (1937).
14. Carman P.C., I. J. Soc. Chem. Ind. Trans., 57, 225 (1938).
15. Lea F.M., Nurse R.W., J. Soc. Chem. Ind., Trans., 58, 271 (1939).

16. Blaine R.L., ASTM Bull., No. 123 51 (1943).
17. Ridgen P.J., J. Soc. Chem. Ind., Trans., 62, 1 (1943).
18. Walther H., Silikattech., 4, 25 (1953).
19. BS 4359, Part 2, 1971.
20. TGL 22201, December, 1968.
21. Dubrow B., Nieradka M., Anal. Chem., 27, 302 (1955).
22. ASTM C 204 - 78a, 1978.
23. Gräf H., Grube H., Beton 36, 184 (1986).
24. Kollek J.J., Materials and Structures, 22, 225 (1989).
25. Martin G.R., Magazine of Concrete Research, 38, 90 (1986).
26. Garboczi E.J., Cement and Concrete Research, 20, 591 (1990).
27. Schönlin K., Hilsdorf H., ACI-SP 108-6, 99 (1988).
28. Bjegović D., Ukrainczyk V., Čalogović V., Žagar G., Expert verlag, Zürich, 3, 1570 (1993).