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On the development of an acoustic permeameter to determining the mean Darcy permeability for porous tissue scaffolds

Alessandro Schiavi
Department of Applied Metrology and Engineering, INRIM: Istituto Nazionale di Ricerca Metrologica, Torino, 10135, ITALY; a.schiavi@inrim.it

Andrea Prato, Alessio Facello, Giovanni Durando and Fabio Saba
INRIM: Istituto Nazionale di Ricerca Metrologica, Torino, 10135, ITALY; a.prato@inrim.it; a.facello@inrim.it; g.durando@inrim.it; f.saba@inrim.it

This paper deals with the permeability determination, with particular attention to the intrinsic Darcian permeability of porous tissue scaffolds, by applying the alternating airflow method (as described in ISO 9053-2), in the peculiar conditions of ultra-slow oscillating flows, in order to keep pure laminar flow condition of Darcy's regime, with Reynolds number close to unit, (or less). The measurements are performed by using an accurate sub-infrasonic "acoustic permeameter" (i.e., able to generate e quantify infrasound pressure level in the range between 2 Hz and 0.1 Hz), developed and realized at INRIM[I]. The accuracy of the system is established on the basis of a direct microphone calibration procedure, using the permeameter itself as a calibrator, based on the pistonphone principle. Among several applications related to airflow resistivity and permeability, it is of interest the possibility to exploit this system for the measurement of the intrinsic Darcian permeability of porous tissue scaffolds used in regenerative medicine and applied biology. It has been shown that experimental results obtained with this system (in a way according to ISO Standard 9053-2), are compatible with results obtained with the specific Standard ASTM F2952.

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1. INTRODUCTION

The accurate quantification of permeability or flow resistance of permeable materials is very useful in a wide range of application in the field of materials science and engineering, since it is related to the mass transport properties and to the microstructural parameters of porous, fibrous or granular materials. Several international Standards provide the technical procedures to quantify the flow resistance of materials, such as ASTM C522[1], ISO 9053-1[2], and ISO 9053-2[3], and permeability, such as ASTM 2434-68[4] (for granular soils), ISO 4410[5], (for fibrous reinforcements), ASTM D737[6], and ISO 9237[7] (for textile fabrics), and ASTM F2952[8] (for porous tissue scaffolds). Experimental methods used to quantify the flow resistance or permeability of porous materials are generally based on the measurement of the pressure drop differential (inlet/outlet), and of the flow velocity of a fluid, through the permeable material under investigation. On the other, many different procedures and experimental techniques can be traced back in the technical and scientific literature, over the last decades.

In this paper we deal with the airflow resistivity/permeability determination, with particular attention to the intrinsic Darcian permeability of porous tissue scaffolds, by applying the alternating airflow method (as described in ISO 9053-2), in the peculiar conditions of ultra-slow oscillating flows, in order to keep pure laminar flow condition of Darcy’s regime, with Reynolds number close to unit, (or less). The measurements are performed by using an accurate sub-infrasonic “acoustic permeameter” (i.e., able to generate and quantify infrasound pressure level in the range between 2 Hz and 0.1 Hz), developed and realized at INRIM[9].

2. PERMEABILITY

A. THEORETICAL BACKGROUND

I. DARCY-FORCHHEIMER EQUATION

Permeability is a property of porous materials quantifying the ability for fluids (gases or liquids) to flow through them. Permeability, as defined from the combination of Darcy’s and Forchheimer’s laws, is related to the effective porosity, to the shapes of the pores and their level of interconnectivity[10]. In this context we refer to intrinsic permeability, namely the permeability in a porous material that is 100% saturated with a single-phase fluid. On the basis of the generalized constitutive Darcy–Forchheimer equation, relating the pressure loss of a one-directional fluid flow through a porous material, due to friction (as a combination of viscous losses and inertial losses), with respect to the velocity of the flow, the overall permeability of the porous material is derived:

$$-\frac{\partial P}{\partial x}=\frac{\mu U}{k_D}+\frac{\rho U^2}{k_F} \tag{1}$$

where ∂P is the pressure drop gradient of the fluid flow (upstream and downstream) across the porous material, in Pa, x is the flow-stream direction, in m, μ is the dynamic viscosity of the fluid, in Pa·s, ρ is the fluid density, in kg/m^3, U is the fluid flow velocity through the porous material in the x-direction, in m/s, k_D is the intrinsic Darcian permeability, in m^2, and k_F is the inertial permeability (non-Darcian permeability), in m.

As it is possible to notice from equation (1), the pressure drop occurring in the fluid flow through the material (along x-direction), is conditioned by two terms velocity-dependent: the first term, U-dependent (also known as Blake-Kozeny equation), is related to the viscous losses in laminar flow, the latter, U^2-dependent (Burke-Plummer equation), is related to the inertial losses in turbulent flow.

Over the last decades, several empirical and theoretical models were developed to clarify the physical meaning of the viscous and inertial resistive terms, within the Darcy-Forchheimer equation.

According to the recent Ergun-Wu resistive model[11], both the intrinsic Darcian permeability k_D and the inertial permeability k_F, can be expressed with a well-defined physical meaning, as a function of inner morphology of the porous material, and to the pores shape, as follows:

$$k_D = \frac{\varphi^2 D_p^2 \varepsilon^3}{72\tau(1-\varepsilon)^2} \tag{2}$$

$$k_F = \frac{\varphi D_p \varepsilon^3}{0.75\tau(1-\varepsilon)}\left(\frac{3}{2}+\frac{1}{\beta^4}-\frac{5}{2\beta^2}\right)^{-1} \tag{3}$$

where φ is the pore sphericity, D_p is the average diameter of the pore cross-sectional area, ε is the effective porosity (i.e., the fractional volume of pores allowing the fluid flows through the material, without accounting voids of closed pores and dead-end pores), τ is the pore tortuosity, and β is the pore narrowing ratio. According to Wu, both pore tortuosity τ, and pore narrowing ratio β can be expressed as a function of the effective porosity ε only, as follows:

$$\tau = 0.5\left[1+0.5\sqrt{1-\varepsilon}+\beta\cdot\sqrt{1-\varepsilon}\cdot\sqrt{\left(\left(\sqrt{1-\varepsilon}\right)^{-1}-1\right)^2+0.25}\right] \tag{4}$$

$$\beta = \left(1-\sqrt{1-\varepsilon}\right)^{-1} \tag{5}$$

These parameters are of fundamental importance in mass transport within actual porous materials, since pore tortuosity τ represents the ratio between the actual length L_s of the porous material, along the macroscopic pressure gradient in the *x*-direction, and the average effective length L_p of the tortuous pore, namely $\tau = L_p/L_s$; and the pore narrowing ratio β allows to estimate the average diameter of the throat D_t, occurring in diverging/converging pores within the porous material, namely $\beta = D_p/D_t$.

II. DARCY'S LAW FOR LINERAR FLOWS IN LAMINAR REGIMES

In practice, it was demonstrated that, if the interstitial Reynolds number Re_i is at least lower than 10, the fluid flow through the porous material is laminar, with negligible inertial losses due to the turbulent effects. Under this condition, it is possible to consider that losses only depend on the viscous term, equation (2), thereby on the intrinsic Darcian permeability k_D only:

$$-\frac{\partial P}{\partial x} = \frac{\mu U}{k_D} \tag{6}$$

After Chor and Li[12], on the basis of the proposed Ergun-Wu model, it is possible to calculate the deviation from linearity, due to the inertial losses, according to the following relation:

$$e = \frac{0.75\tau(Re_i)^2}{72\tau(Re_i)}\left(\frac{3}{2}+\frac{1}{\beta^4}-\frac{5}{2\beta^2}\right) \tag{7}$$

In which the interstitial Reynolds number Re_i is given by:

$$Re_i = \frac{\rho\varphi D_p U}{\mu(1-\varepsilon)} \tag{8}$$

According to equations (7) and (8), by assuming an exemplificative (and unfavorable) condition in which the porosity $\varepsilon \sim 0.9$, the sphericity $\varphi \sim 1$, the average pore diameter $D_p \sim 1$ mm, with a fluid flow velocity $U \sim 0.01$ m/s, the interstitial Reynolds number is $\mathrm{Re}_i = 6.7$, that provides a linear error $e \approx 4\%$.

Thus, at macroscopic level for linear flows, the intrinsic Darcian permeability k_D (equation (6)), can be measured on the basis of the following experimental relation, directly related to airflow resistivity r:

$$k_D = \mu \frac{U}{\Delta P} \cdot L_s = \mu \frac{Q_v}{\Delta P} \cdot \frac{L_s}{A_s} = \frac{\mu}{r} \tag{9}$$

where ΔP is the pressure gradient upstream and downstream the scaffold, in Pa, Q_v is volumetric linear airflow, in m^3/s, L_s is the length of the porous medium (along the flow direction), in m, and A_s is the cross-sectional area of the porous medium perpendicular to the flow direction, in m^2.

III. DARCY’S LAW FOR OSCILLATING FLOW

By assuming that, instead a linear flow, a slow oscillating flow is used to evaluate the Darcian intrinsic permeability (according to ISO Standard 9053-2, in which is specified an alternating airflow method for the determination of the airflow resistance), the experimental relation (9), can be rewritten in these terms:

$$k_D = \mu \frac{Q_v}{\Delta P} \cdot \frac{L_s}{A_s} = \mu \frac{q_{v,rms}}{p_{rms}} \cdot \frac{L_s}{A_s} = \mu \frac{\omega \partial V \left(\sqrt{2}\right)^{-1}}{\gamma p_0 \partial V \left(V_0 \sqrt{2}\right)^{-1}} \cdot \frac{L_s}{A_s} = \mu \frac{\omega V_0}{\gamma p_0} \cdot \frac{L_s}{A_s} \tag{10}$$

where $q_{v,\mathrm{rms}}$ is the alternating r.m.s. value of volumetric airflow, p_{rms} is the sinusoidal r.m.s. value of pressure component (depending on the atmospheric static pressure p_0, with heat capacity ratio $\gamma = 1.4$, at a known temperature T and relative humidity RH), ∂V is the volume variation induced by the oscillation the volume of air V_0, and ω is the angular frequency of the airflow oscillation, in rad/s.

3. THE EXPERIMENTAL METHOD

The determination of the Darcian intrinsic permeability k_D, on the basis of relation (10), is realized by measuring the sinusoidal r.m.s. value of pressure component p_{rms} (with a low-pressure field microphone), with respect to the effective microphone sensitivity, at the given angular frequency ω, as described below.

A. THE ACOUSTIC PERMEAMETER

The acoustic permeameter, shown in Figure 1, provides permeability measurements, from the accurate quantification of the acoustic pressure wave drop of a very slow alternating airflow through the samples, generated by an oscillating thick and stiff piston in a closed cavity, by using a single low-frequency pressure field microphone.

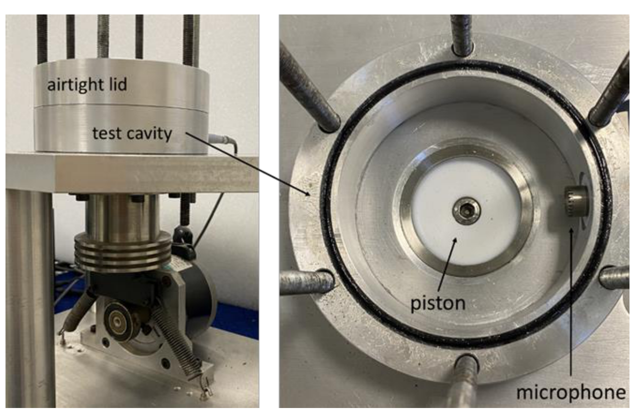

Figure 1. The acoustic permeameter and the test cavity with the piston and the microphone.

As a first step, the sensitivity of the microphone (B&K ½" microphone, type 4193) is determined on the basis of a calibration method, as schematically shown in Figure 2. By means of the piston (driven by a crank-connecting rod system, managed by a stepper motor with accuracy of $1.6 \cdot 10^{-3}$ rad·s^{-1} /step), very slow pulsations ($\omega \sim 1$ rad·s^{-1}) of the air volume V_0 in the test cavity are generated, inducing pressure oscillations. The test cavity is closed by a full airtight lid. The occurring pressure waves amplitude $p_{\text{rms}} = \gamma p_0 \partial V \left(V_0 \sqrt{2}\right)^{-1}$, act on the microphone membrane generating a proportional voltage variation. This signal is amplified (G.R.A.S. Power Module, type 12AK), sent to a DAQ (NI – USB4431, 24 bit, 102.4 kS/s), and analyzed in terms of angular frequency and amplitude. The ratio between the measured output voltage and the pressure waves amplitude provides the microphone sensitivity (in that given conditions), expressed in mV/Pa$_{\text{cal}}$, as a calibration result.

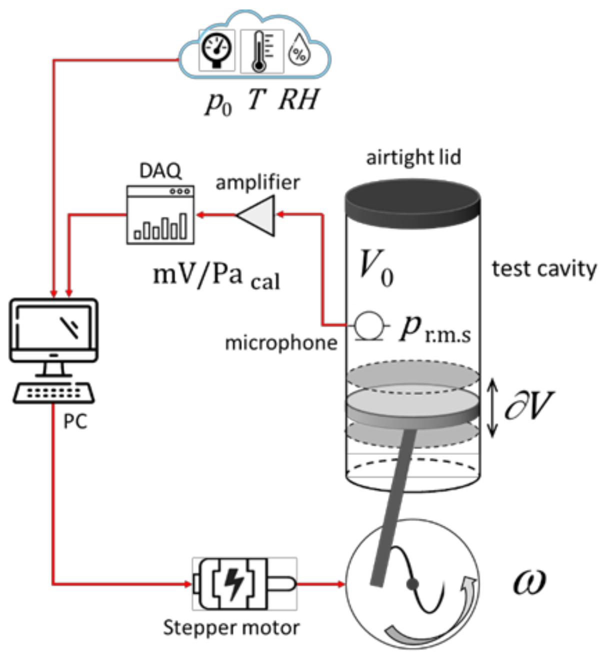

Figure 2. The calibration methods of the microphone within the test cavity.

Once the sensitivity of the microphone is determined, the full airtight lid is removed and replaced by an airtight lid with a passing hole closed by the porous/permeable sample to be investigated, as shown in Figure 3. In order to avoid losses, all junctions are sealed to each other with O-rings held under pressure by passing screws. In addition, a thin layer of petroleum jelly closes any possible further leakage in the seals. The sample is allocated in a specific holder in teflon, sealed with a teflon tape; a further plasticine seal is placed between the sample and the holder, in order to avoid possible leakage at the edge.

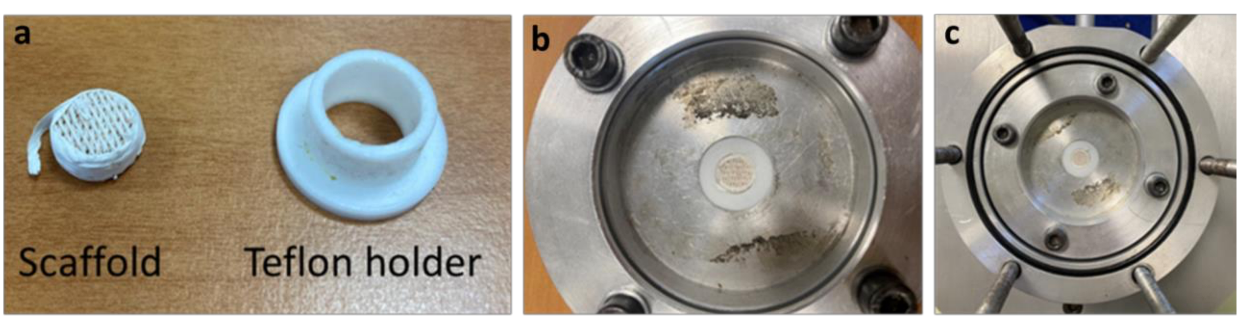

Figure 3. a) the sample, sealed with Teflon tape, and the holder; b) the sample in the holder allocated in the proper support of the acoustic permeameter (an airtight lid with a passing hole closed by the porous sample under investigation); c) the support on the acoustic permeameter.

The slow pulsations ($\omega \sim 1$ rad/s) of the air volume V_0 through the samples, generate an alternate flow rate $q_{v,\mathrm{rms}} = \omega \partial V \left(\sqrt{2}\right)^{-1}$, low enough to keep interstitial Reynolds number close to unit, in order to avoid turbulent airflow within the solid phase of the porous/permeable sample. The amplitude of the pressure wave, measured by the microphone, is reduced with respect to the calibration conditions, since a certain quantity of air is slowly pumped in and out through the porous sample, as shown in Figure 4.

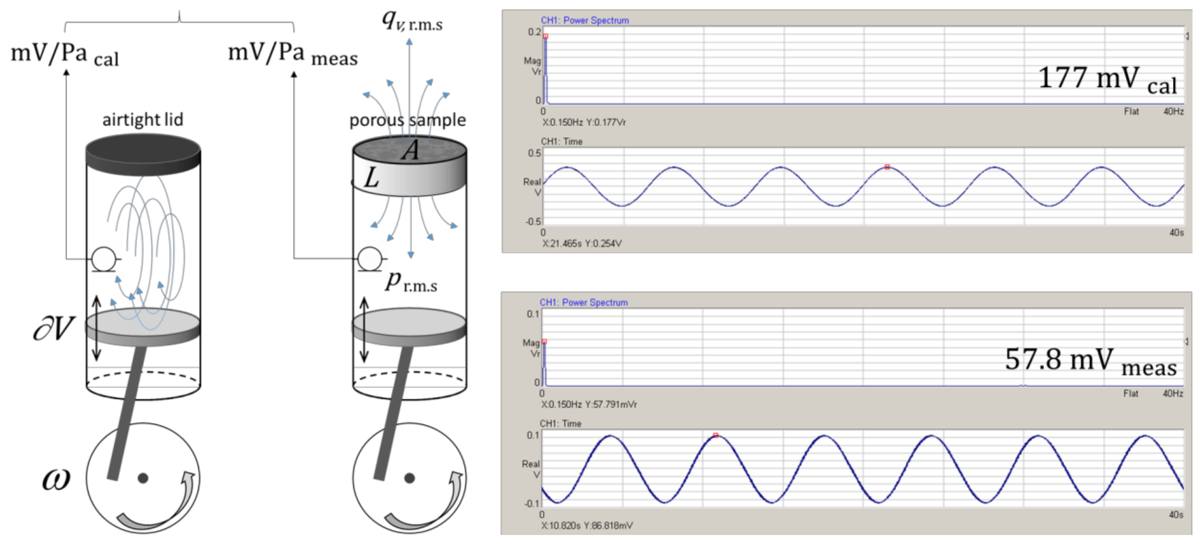

Figure 4. The conceptualization of the intrinsic Darcian permeability measurement method by using the acoustic permeameter, and the actual experimental output data (time/frequency domain)

The pressure wave drop is then determined by the ratio between the amplitude of the pressure wave measured in the hermetically closed air volume (i.e., the microphone sensitivity, in $\mathrm{mV/Pa_{cal}}$), and the amplitude of the pressure wave measured in the same volume of air enclosed by the porous/permeable sample, in $\mathrm{mV/Pa_{meas}}$. Thus, intrinsic Darcian permeability k_D is experimentally determined as follows:

$$k_D = \mu \frac{\omega V_0}{\gamma p_0} \cdot \frac{L_s}{A_s} \cdot \frac{mV_{cal}}{mV_{meas}} \tag{11}$$

The dynamic viscosity μ of the air, for acoustic applications, is calculated according to Rasmussen model[13], using as input data the environmental air temperature *T*, the atmospheric pressure p_0, and relative humidity *RH* during the measurements.

According to the experimental model applied for the calculation of the intrinsic Darcian permeability, equation (11), the only quantities to be accurately determined, are the relative amplitudes of the alternating pressure waves (in mV/Pa) and the angular frequency ω of the pulsations, then the geometrical dimensions of the cavity V_0 (containing the air subject to the pulsations), the geometrical dimensions of the sample under investigation, namely the surface area A_s, perpendicular to the air flow, and the thickness L_s, along the fluid flow, the atmospheric static pressure p_0 (during the measurement), and the relative environmental conditions, in terms of temperature and relative humidity. Moreover, depending on the scaffolds properties, it is possible to vary both the geometrical dimensions of the acoustic permeameter test cavity V_0 (between $2 \cdot 10^{-4}$ m^3 and 10^{-3} m^3), and the airflow velocity, from the angular frequency ω (between 12.6 rad/s and 0.63 rad/s, or 2 Hz and 0.1 Hz), in order to optimize the microphone dynamic response.

Taking into account that heat losses on the wall of the test cavity are not negligible at $\omega \sim 1$ rad/s (i.e., 0.15 Hz), a correction coefficient is applied to the sinusoidal r.m.s. value of pressure component p_{rms}, based on the classical Gerber analysis. For the geometrical dimensions of the test cavity, the complex correction coefficient at 0.15 Hz, corresponds to 1.09+0.09i.

B. EXPERIMENTAL DATA ANALYSIS OF POROUS TISSUE SCAFFOLDS

The intrinsic Darcian permeability k_D of porous tissue scaffolds is determined, along the related uncertainty contributions of the physical and the mechanical quantities of Darcy's law for oscillating flows, equation (11), on the basis of the general rule of random error propagation according to GUM[14], and the experimental results are expressed with a confidence level of 95%. In general, measurements are performed several times on the same sample, and at least on 3 individual sample of the same porous tissue scaffold, in order to take into account both the measurements repeatability and reproducibility.

By considering all variables (geometrical dimensions of measuring system and investigated porous/permeable samples, the physical properties of airflow, and the output voltage data) as independent, the uncertainty assessment applied to calculate the expanded uncertainty of each sample $U(k_D)$ is based on the following relation:

$$U(k_D) = k \cdot \sqrt{\sum_{i=1}^{N} \left(\frac{\partial k_D}{\partial x_i}\right)^2 u^2(x_i)} \tag{12}$$

in which k is the coverage factor (namely, k=2 provides an interval having a confidence level of approximately 95%), k_D is given in equation (11), x_i is the i^{th} independent variable of equation (11), and $u^2(x_i)$ is the standard uncertainty, associated to the independent variable x_i.

The measurements are carried out at a constant airflow pulsation $\omega = 0.926$ rad/s. The volume of air confined in the test cavity is $V_0 = (2.498 \pm 0.009) \cdot 10^{-4}$ m^3, and the volumetric airflow results $q_{v,\text{rms}} = 1.07 \cdot 10^{-6}$ m^3/s, since $\partial V = 1.64 \cdot 10^{-6}$ m^3 (with a peak-to-peak stroke displacement of $1.71\cdot10^{-3}$ m, and a piston surface area of $1.92\cdot10^{-3}$ m^2). The atmospheric static pressure is measured by a barometer (Druck DPI 280), with a fractional accuracy of $1 \cdot 10^{-4}$, in the range 800-1100 mbar.

By way of example, Table 1 reports the detailed uncertainty analysis for a single measurement of intrinsic Darcian permeability k_D of a specific sample of porous tissue scaffold.

Table 1. Uncertainty analysis, according to GUM rules, equation (12), for a single measurement of intrinsic Darcian permeability.

Variable x_j	Value	Note	$u^2(x_j)$	$\partial f/\partial x_i$	$u_c^2(y)$
μ/Pa·s	$1.81\cdot10^{-5}$	tolerance	$3.3\cdot10^{-17}$	$1.5\cdot10^{-6}$	$7.0\cdot10^{-29}$
ω/rad·s^{-1}	$9.26\cdot10^{-1}$	tolerance	$1.3\cdot10^{-5}$	$2.8\cdot10^{-11}$	$1.1\cdot10^{-26}$
V_0/m^3	$2.49\cdot10^{-4}$	tolerance	$2.1\cdot10^{-13}$	$1.1\cdot10^{-7}$	$2.4\cdot10^{-27}$
p_0/Pa	$9.78\cdot10^{4}$	accuracy	33.3	$-2.7\cdot10^{-16}$	$2.4\cdot10^{-30}$
L_s/m	$9.85\cdot10^{-3}$	tolerance	$9.8\cdot10^{-10}$	$1.2\cdot10^{-9}$	$1.3\cdot10^{-27}$
A_s/m^2	$8.12\cdot10^{-5}$	tolerance	$1.3\cdot10^{-11}$	$-1.4\cdot10^{-7}$	$2.6\cdot10^{-25}$
$Volt_{cal}$/V	$1.77\cdot10^{-1}$	accuracy	$1.1\cdot10^{-7}$	$2.8\cdot10^{-10}$	$8.5\cdot10^{-27}$
		resolution	$3.3\cdot10^{-11}$	$2.8\cdot10^{-10}$	$2.6\cdot10^{-30}$
$Volt_{meas}$/V	$5.78\cdot10^{-2}$	accuracy	$6.5\cdot10^{-7}$	$-8.6\cdot10^{-10}$	$4.8\cdot10^{-25}$
		resolution	$3.3\cdot10^{-11}$	$-8.6\cdot10^{-10}$	$2.4\cdot10^{-29}$
k_D /m^2	**$1.14\cdot10^{-11}$**			Variance, ***u²***	$2.9\cdot10^{-25}$
				Standard uncertainty, ***u***	$5.4\cdot10^{-13}$
				Confidence level	95%
				Expanded uncertainty, ***U*(*k_D*)**	**$1.07\cdot10^{-12}$**
				Relative expanded uncertainty	**9.3%**

Referring to the values reported in Table 1, it was found that the major individual contributions to the combined standard uncertainty are associated to the geometrical dimensions of the samples, i.e. the cross-sectional area A_s of the sample, and to the accuracy of tension output $Volt_{meas}$ (obtained as the standard deviation from 3 measurement repetitions).

The corresponding linear velocity of the fluid results $U = q_v/A_s = 1.32 \cdot 10^{-2}$ m/s, thus the Reynolds number, according to the exemplificative condition of equation (8), is $\mathrm{Re}_i = 8.4$, causing a linear error $e \approx 5\%$. It has to be said that, in general for porous tissue scaffolds, the effective porosity ε, and the pore sphericity φ, are far from unit, then the actual Reynolds number is expected to be considerably lower.

C. VALIDATION AND PROFICIENCY TEST

Recently, a bilateral proficiency test was carried out by comparing in-blind data measured by the acoustic permeameter and by a test-bench, compliant with ASTM F2952, employing a continuous water flow pump-based method[15]. Comparisons were carried out on different typologies of porous rigid scaffolds. The comparison provided highly compatible results, in terms of repeatability and reproducibility. In addition, from the analysis of uncertainties estimated by applying the two methods, a higher precision can be attributed to experimental results provided by the acoustic permeameter. This is mainly due to the fact that the acoustic permeameter uses a single transducer (the microphone), instead of two (a differential pressure gauge, and a flowmeter), as used in the ASTM-compliant test bench.

D. BRIEF SURVEY OF APPLICATIONS

The acoustic permeameter here described was employed for a large measurement campaign, in different fields of applications. Due to its versatility, e.g., being possible to vary the capacity of the test cavity volume V_0, the velocity of the fluid flow (by varying the angular frequency ω), or the surface area A_s, many different typologies of porous, fibrous or granular materials, can be analyzed.

I. TISSUE SCAFFOLDS ENGINEERING

As previously described, the acoustic permeameter is successfully applied to measure and quantify the intrinsic Darcian permeability of porous tissue scaffolds, developed in the field of regenerative medicine, for bone tissue engineering, in a wide range of permeability, in particular from 10^{-11} m^2 up to 10^{-7} m^2, that is the typical range of native human bone tissue permeability. Actually, it has to be said that this method is suitable only for porous rigid scaffolds in dry condition, and cannot be applied to ultra-soft or wet tissues. This method was applied to commercial calcium carbonate porous implants[16]; then, the method was progressively refined and extended to hydroxyapatite[17], and bioactive glass scaffolds[18] with foam-like architectures, as well as 3D printed scaffolds by vat photopolymerization[19].

II. AUTOMOTIVE ENGINEERING

The acoustical properties of materials used in automotive engineering, such as firewalls (or NVH absorbers) separating the engine compartment from the passenger compartment, and NVH absorbers used inside the passenger compartment, were estimated from measurements of airflow resistivity, also as a function of temperature[20].

III. POROELASTIC EFFECTS AS A FUNCTION OF STATIC LOAD

The effects of load compression, in poroleastic materials, used in insulating pads for floating floors, were investigated by measuring airflow resistivity as a function of different static load, in order to accurately estimate the dynamic stiffness, taking into account the air stiffness inside the open porosity. Indeed, the acoustical permeameter can be equipped by specific perforated devices, which apply different static loads on the surface of the samples[21].

IV. BUILDING PHYSICS

In building physics and architectural acoustic, sound absorption is a fundamental parameter for the characterization of the acoustical performance of indoor spaces. Sound absorption of porous/fibrous materials is directly related to the airflow resistivity, and a large variety of this kind of materials (both natural and artificial compounds) were successfully tested by using this acoustic permeameter [22]. In some case, the airflow resistivity has been measured also for granular inhomogeneous compounds of vegetable chips[23].

4. CONCLUSION

In this work, an acoustic permeameter, designed and built at INRIM, has been presented. The acoustic permeameter measures the pressure wave drop, in a sub-infrasonic frequency range, by means of a low-frequency pressure field microphone.

This system allows performing high accurate measurements of permeability and airflow resistivity. The accuracy of the system is established on the basis of a direct microphone calibration procedure, using the permeameter itself as a calibrator, based on the pistonphone principle.

Among several applications related to airflow resistivity and permeability, it is of interest the possibility to exploit this system for the measurement of the intrinsic Darcian permeability of porous tissue scaffolds used in regenerative medicine and applied biology. It has been shown that experimental results obtained with this system (in a way according to ISO Standard 9053-2), are compatible with results obtained with the specific Standard ASTM F2952.

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