PRESSURE/MASS METHOD TO MEASURE OPEN POROSITY OF POROUS SOLIDS

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1. INTRODUCTION

The open porosity of a porous solid is a key parameter in the physical and acoustical modeling of porous media, especially to relate the effective properties of the fluid saturating the interconnected pores to the effective properties of the porous solid.^{1,2} In the past, several acoustical indirect methods have been proposed to measure this parameter^{3,4} However, direct methods are generally Examples of direct measurement methods preferred. include the pioneer work of Beranek,⁵ and its improved version standardized as ASTM D 2856-94. The method relies on the equation of state for ideal gases at constant temperature. However it is time consuming and requires several calibration of the device. Other direct measurement methods include the improvement of Beranek method proposed by Leclaire *et al.*⁶ and by Champoux *et al.*⁷ and the missing mass method proposed by Panneton and Gros⁸.

In this paper, a method is presented to measure the open porosity of porous solids. The method needs only a simple apparatus and its accuracy is predictable from the knowledge of the total bulk volume of the tested porous sample.

2. THEORY

The open porosity of porous solid is expressed as

$$\phi = 1 - V_s / V_t , \qquad (1)$$

where V_s is the volume of the solid phase, and V_t the total bulk volume of the porous aggregate. In general, V_s is unknown and needs to be determined. To determine its value, the four tests presented in fig.1 are used together with the perfect gas law relation. If the process between and during tests is very slow and room conditions are constant, isothermal condition can be assumed. The volume of the solid phase V_s can then be expressed as [see ref⁹]

$$V_{s} = \left(\left(M_{2} - M_{1} \right) / \left(P_{2} - P_{1} \right) - \left(\left(M_{4} - M_{3} \right) / \left(P_{4} - P_{3} \right) \right) \right) RT$$
(2)

where R is the specific gas constant, T is the temperature in Kelvin and M_i the different masses measured on the balance.

3. ERROR ANALYSIS

3.1 Applicability of the method

As shown, to apply the proposed method, volume V_s needs first to be evaluated. Usually, the target pressures to use are

low pressure P_{lo} and high pressure P_{hi} so that $P_1 \cong P_3 \cong P_{lo}$, and $P_2 \cong P_4 \cong P_{hi}$. Also, it can be easily shown that the difference $M_i - M_{i-1} = m_i - m_{i-1}\Big|_{i=2,4}$. Furthermore, $m_2 - m_4$ vields the mass of gas occupied by the solid phase of the porous sample at high pressure (i.e., $\rho_{hi}V_s$, where ρ_{hi} is the mass density of the gas at high pressure), and $m_1 - m_3$ yields the mass of gas occupied by the solid phase at low pressure (i.e., $\rho_{lo}V_s$, where ρ_{lo} is the mass density of the at low pressure). Consequently, gas $M_2 - M_4 - M_1 + M_3 = (\rho_{hi} - \rho_{lo})V_s$ and ρ_{lo} corrects for the fact that the low pressure condition is not the perfect vacuum condition. Not accounting for $m_1 - m_3$ would only add a bias error proportional to P_{lo} in the evaluation of V_s and ϕ ; however it does not prevent the applicability of the method.

From the previous analysis, the most severe condition that remains for applying the method is the readability of the high pressure mass difference (i.e., $m_2 - m_4 \ge \varepsilon$, where ε is the balance readability). This condition gives the maximum porosity the method can determine for a given set of operation conditions

$$\phi_{\max} = 1 - RT\varepsilon / P_{hi}V_t \quad . \tag{3}$$

Figure 2 gives the minimum bulk volume per balance readability as a function of open porosity for different operating conditions. It shows that larger bulk volume, larger high pressure, and gas with larger density are desirable to increase the applicability of the method, and to detect open porosity closer to unity (typical for sound absorbing porous materials).

3.2 Precision of the method

Using the total differential method and assuming that the errors are random and follow a normal distribution, the expected error committed on the open porosity is

$$\delta\phi = \pm \left(1 - \phi\right) \sqrt{\left(\delta T/T\right)^2 + \left(\delta V_t/V_t\right)^2 + \left(\delta Z/Z\right)^2}, \qquad (4)$$

For a cylindrical test sample, the error on its bulk volume is given by $\delta V_t = \pm V_t \delta_L \sqrt{(2/D)^2 + (1/H)^2}$, where *D* and *H* are its diameter and height, and δ_L is the uncertainty on the dimension measurement. δZ is the error related to variable *Z* defined, following Eq.(2), as $Z = V_s / RT$. Considering that the uncertainty relative to the masses and pressures are given, respectively, by the balance and the manometer readability ε and p, and Assuming P_1 and P_3 are approximately equivalent and equal to low pressure P_{lo} , P_2 and P_4 are approximately equivalent and equal to high pressure P_{hi} , and test chamber volume $V >> V_s$, one obtains

$$\delta Z \simeq 2\sqrt{\varepsilon^2 + \left(pV/RT\right)^2} / \left(P_{hi} - P_{lo}\right). \tag{5}$$

The error predicted by the Eq. (4) is valid only if $V_t \leq V$.

Figure 3 shows the expected absolute error on open porosity as a function of bulk volume per test chamber volume for three different open porosity values (0%, 90%, and 99%). The error generated by the individual uncertainties on the mass and pressure, the bulk volume, and the temperature are also plotted. It is noted that the precision of measurement is better when using larger bulk volume. It is also noted that for the three cases, the error is mostly controlled by the uncertainty on the mass and pressure reading. Since the error is mostly controlled by the uncertainty on the mass and pressure reading, the error on the open porosity can be estimated by the approximated expression given by

$$\delta\phi \simeq 2\sqrt{\left(\left(RT\varepsilon\right)^2 + \left(Vp\right)^2\right)} / V_t \left(P_{hi} - P_{lo}\right). \tag{6}$$

One can observe that it fits well with the global error in the valid range of bulk volume $(V_t / V \leq 1)$.

4. EXPERIMENTAL TESTS AND RESULTS

To validate the gas porosimeter and its precision, two tests have been performed. The first test consists in applying the method to measure the porosity of high porosity samples (95%) of known solid phase volume $V_{\rm s}$. The second test consists to applying the method to measure the porosity of low porosity samples (45%) of known solid phase volume V_s . Details on test setup and procedure can be found in ref⁹. Figure 4a compares the measured standard deviation (i.e., measured error) for each of the ten samples to the theoretical prediction. It is observed that the measured error fits well with the one predicted by Eq.(6). Figure 4b shows the measured mean porosity for the 30 individual tests as a function of the bulk volume to test chamber volume ratio to better visualize the scattering of the measurements around the theoretical value. One can clearly observed that the precision on the measurements improves with the bulk volume to test chamber volume ratio.

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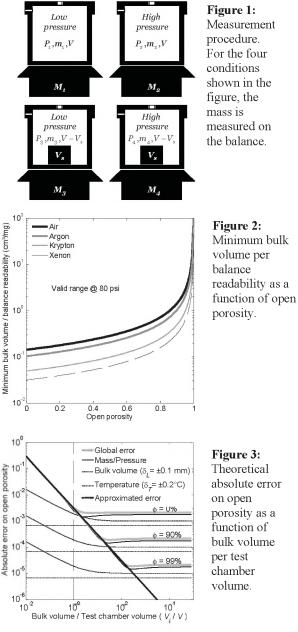
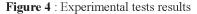


Figure 3: Theoretical absolute error on open porosity as a function of bulk volume per test chamber volume.



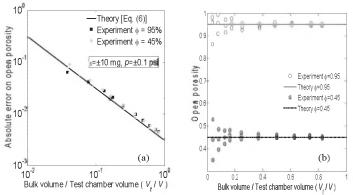


Figure 1:

Measurement procedure. For the four conditions shown in the figure. the mass is measured on the balance.