

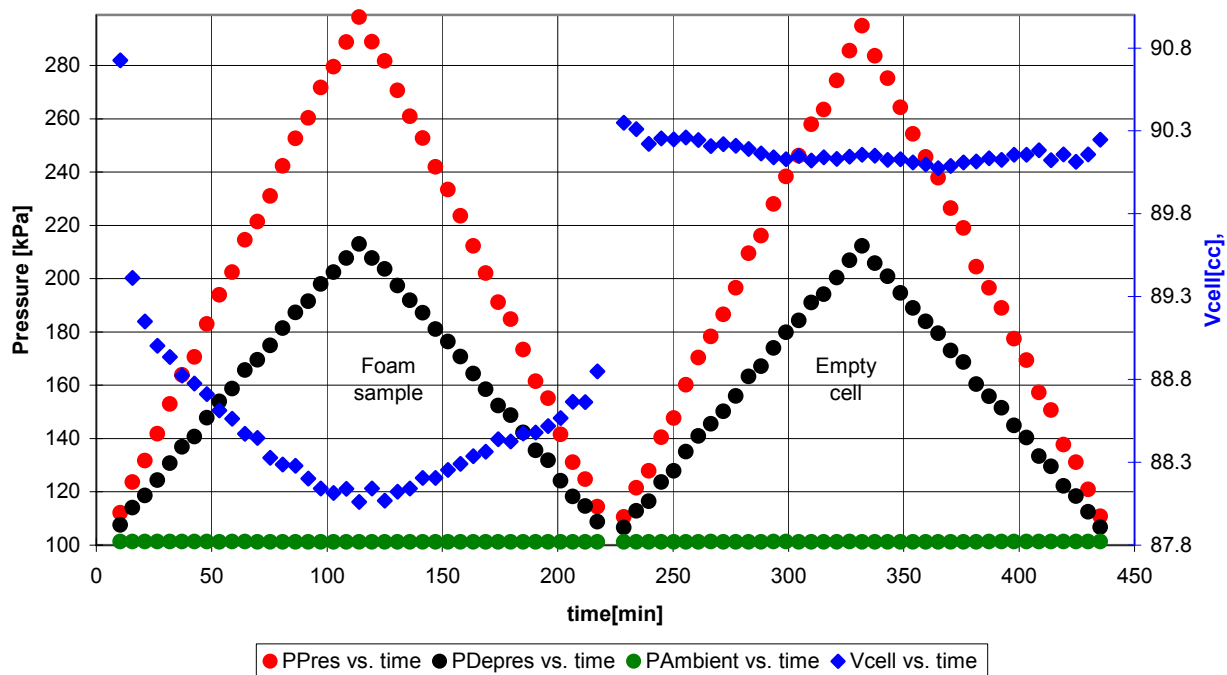
Determination of foam density (open - cell, closed – cell percentage) using the HumiPyc™ - density analyzer

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Profile of density vs. pressurization pressure instead of a single number

Density determination of materials of low rigidity (e.g. foams, powders) using a simple gas pycnometer is challenging in several aspects. Typically, to avoid excessive compression, the pressurization pressure is set just above ambient. However, this approach is problematic as the least optimal pycnometer pressure range is used (see the previous application note). It is often the case that foams of open-cell structure occupy large physical volume (geometrical volume V_g), but their “pycnometric” volume, V_p , is rather small. Therefore, the V_p , as a result of subtraction of two large numbers, further exacerbates the accuracy problem of subsequent density determination. Considering the diversity of foam properties, it seems inadequate to describe its density by just a single value. Actually, the density varies with pressurization pressure, temperature, and the history of the materials.

HumiPyc: Time course of exp. PycTestFoam0.SUM, T = 25 C



The capabilities of HumiPyc to automatically carry out programmed pressure steps (up to 1000) allows obtaining the density profile vs. pressurization pressure. As illustrated on the above picture, the volume of the sample cell (V_{cell}) was obtained at increasing and decreasing pressurization pressure, P_{Press} , values with the foam inside and the empty sample cell. A sample of packaging foam with open-cell structure (low firmness) was used for the measurements. It is clear that the sample volume exhibits strong dependence on

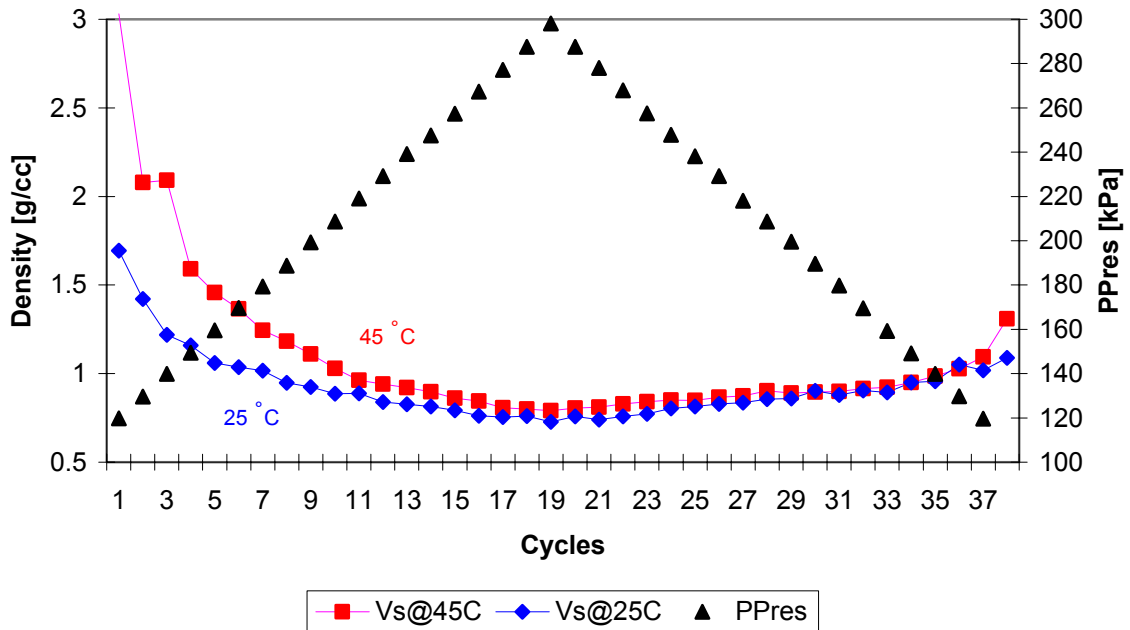
pressurization pressure and using low pressurization pressures (up to 120 kPa) yields results with larger error. It is also noticeable, that the linear up and down pressure ramp does not produce symmetrical response of the sample volume changes. Long relaxation times (typical to materials with closed-cell content) or in some cases partial destruction of the cellular structure can be the culprit.

Subtracting the cell volumes with the sample inside from the empty cell volumes at respective pressurization pressures yields the V_p volume of about 0.9 to 2 cc at 25 °C. The geometrical volume of the foam sample is 73.6 cc and its mass is 1.5226 g. The foam density can be easily calculated by dividing the mass by its volume.

Temperature control from sub ambient to 50 °C

Since density of many materials varies with temperature, the HumiPyc features uniform and stable temperature control from sub ambient to 50 °C. It allows for obtaining of additional information about the studied material by carrying out experiments at different temperatures. For example, the above experiment was rerun at 45 °C using the same foam sample. The density profiles at the two temperatures and at the up and down pressure steps is presented below.

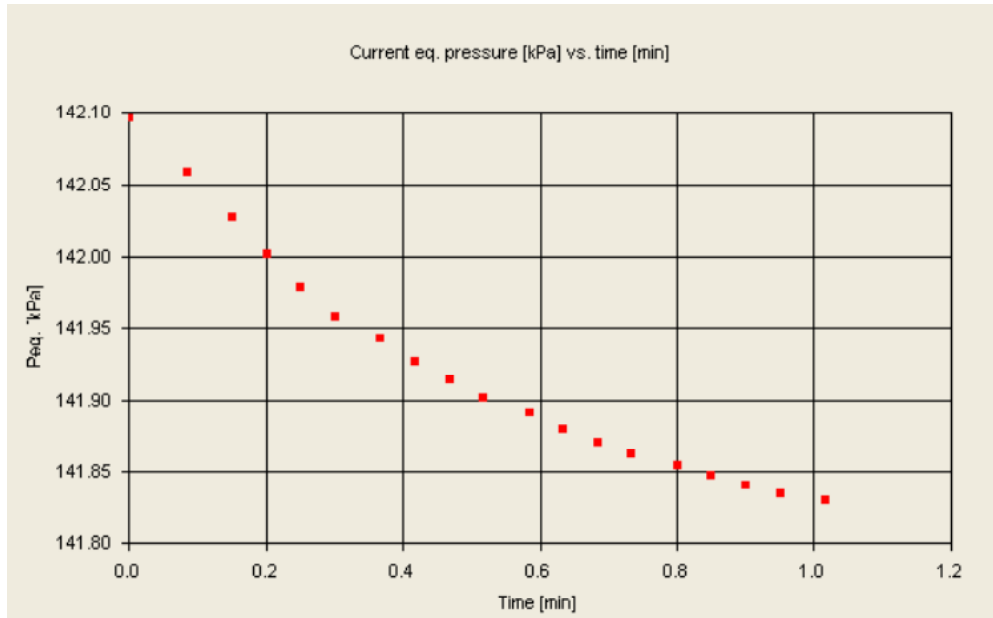
Density profiles of a foam sample at different temperatures and pressures



Selection of pressure equilibration criteria

Another aspect of accurate density determination is achieving a stable pressure value after pressurization or depressurization. Employing a fixed equilibration time rarely works well for real samples (other than metal or glass beads). In case of foam with some percentage of closed cells, the equilibration time can be very long and it might not be practical in all cases to use very strict criteria. However, the user should estimate the amount of error by using relaxed criteria and trying to get a larger number of data within allotted time. The HumiPyc employs two concurrent pressure stabilization criteria that are modifiable at run time: maximum allotted time or having three consecutive measurements within a specified pressure band. By

selecting the appropriate chart (presented below), the user can review the equilibration profile and adjust the parameters.



Open –cell, Closed-cell percentage, Corrections for cutting

Preparation of a foam specimen for subsequent measurements and determination of its geometrical volume requires precise cutting. Cutting a cylindrical shape sample using modified circular-hole cutters seems to provide good shape cutouts. The cylindrical shape allows for the most efficient filling of the sample chamber.

Using a sample of cylindrical shape, its geometrical volume, V_g , can be easily calculated:

$$V_g = \frac{\pi}{4} * d^2 * h \quad (\text{eq. 1})$$

where d is the sample diameter, h is the sample height, and π is a constant = 3.14159....

Using the HumiPyc, a profile of volume vs. pressure can be obtained (or a single measurement can be used) and the pycnometer volume, V_p , can be calculated.

Defining the open cell content, O_c , as

$$O_c [\%] = \frac{V_g - V_p}{V_g} * 100 \quad (\text{eq.2})$$

the close cell content, C_c , can be either simply calculated as

$$C_c [\%] = 100 - O_c \quad (\text{eq. 3})$$

or, from the definition

$$Cc[\%] = \frac{V_p}{V_g} * 100 \quad (\text{eq. 4})$$

Correcting for opened cells during cutting

Measurements of foam density require some sort of cutting operation which inadvertently destroys some amount of closed cells and makes them easily available to the gas access.

Since during cutting out the sample some initially closed cells are being opened, a correction for this effect can be attempted. Often the initial sample is divided into smaller pieces to expose additional area. Then the whole sample is re-measured again in a gas pycnometer (at the same experimental conditions) and a new volume, V_{p1} , is obtained. Obviously, the V_{p1} is expected to be smaller than the V_p . The exact correction formula will depend on the geometry of the sample and the way the sample was divided. To simplify the correction calculations, the additional cutting should avoid cutting through the original cutout surface(s).

In general, let say that the additional cutting has introduced area of A_1 value. That value has caused decrease of the sample volume by $V_p - V_{p1}$. If the originally exposed area during the sample is A_0 , the volume correction, V_{corr} , can be obtained from the relation:

$$\frac{A_0}{A_1} = \frac{V_{corr}}{V_p - V_{p1}} \quad (\text{eq. 5})$$

as

$$V_{corr} = \frac{A_0}{A_1} * (V_p - V_{p1}) \quad (\text{eq. 6})$$

If, hypothetically, the initial cutting had not exposed the surface A_0 , the original volume determination, V_p , would be larger by V_{corr} . Therefore, the equation 2 would need to be modified to reflect the correction:

$$Oc[\%] = \frac{V_g - V_p - V_{corr}}{V_g} * 100 \quad (\text{eq. 7})$$

An example of open-cell, closed cell, and correction calculations.

A specimen of closed cell (firm) foam was prepared by cutting a cylindrical shape from the foam sample. The photo below shows the specimen and the tool used. The specimen has diameter of 51.4 mm (2.02") and height of 39.3 mm (1.55"). The mass of the specimen is 2.3615 g. The geometrical volume, V_g , of the cutout is **81.5 cm³** (Equation 1).

Using the HumiPyc, the obtained volume V_p of the cutout is **15.96 cm³** (± 0.15). It needs to be pointed out, that due to difficulty of establishing good pressure stability for foams (and working far from optimal instrument conditions), the standard deviation is many times larger than for rigid substances.



To correct for the area A_0 exposed during initial cutting out of the specimen, the concentric cutout of diameter 35.7mm (1.40”) from the specimen was cut and it exposed additional area of A_1 value. The two parts are shown below.



The inner core was inserted into the shell and the volume of the whole sample, V_{p1} , was obtained as **14.04 cm³** (± 0.17). Since the top and bottom of the foam sheet is finished with the cells closed, those surfaces are not taken into account for correction calculations. In this particular case and due to the same cutouts height, the equation 6 can be presented in the form,

$$V_{corr} = \frac{d_0}{d_1} * (V_p - V_{p1}) \quad (\text{eq. 6a})$$

where d_0 is the diameter of the original cutout and the d_1 is the diameter of the inner core.

Plugging the data into the equation 6a yields the corrected volume V_{corr} equal to 2.76 cm³.

The percentage of the corrected open-cell content (Eq. 7) is 77.0%. The closed cell content is obviously 23.0 %. The corrected foam density can be estimated as 0.126 g/ cm³.

In contrast to the simple, manually or keypad-operated gas pycnometers, the fully automated density analyzer – HumiPyc offers substantial improvements for foam and powders characterization capabilities due to novel hardware and software design. Direct migration to the PC environment for experiment design, control, and data handling allows for presenting to a researcher a one of the most sophisticated density analyzers. Additionally, its multi techniques capabilities and detailed experimental data that are not available from other pycnometers allow for more complete characterizations of the materials in question.

It is not only to get the final number; it is to show the process and the number in it.