Experimental investigation of the limits of fluid squeeze out from an imbibed porous material

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Abstract. The resistance to flow of a liquid squeezed out of a soft, porous structure subjected to compression generates a high load capacity and represents the mechanism on which the XPHD lubrication is based. The process is dependent on permeability which, in turn, depends on porosity, variable during compression. This study investigates experimentally the effect of compression on the porosity of open-cell polyurethane foams. An experimental device is used to compress a disc made of a porous material imbibed with glycerine and the volume of the remaining fluid is measured. Two reticulated foams with the same internal structure but with different initial porosities were selected for the study. The results are consistent and show a good correlation when compared with theoretical predictions. It is shown that the deformation of the solid fraction closes pores before squeezing out all the fluid. A residual porosity and a threshold strain when the pores are closed are put in evidence.

1. Introduction

XPHD lubrication [1] is a research subject devoted to the capacity of soft and porous structures imbibed with liquids in order to generate a high load support under compression. The load support is generated through the resistance to flow inside the porous material. During compression, the resistance to flow and load support increase. The greater the compression level, the lower the porosity and corresponding permeability.

Hypothetically, the porosity and permeability decrease to as low as zero at high compression levels, which yields to unrealistic increases (up to infinity) of the fluid pressure and flow resistance forces. In reality, the rapid compression of the soft porous layers imbibed with fluids is characterized, by three stages:

(i) A first stage, when the load support is produced mainly by the resistance to flow, and the contribution of the deformation of the solid structure is negligible; this is purely XPHD regime.

(ii) Increasing the compression level of the compaction, the deformation forces of the solid fraction increase due to multiple contacts between micro-components of the structure (fibres, cell walls, particles etc.).

(iii) When the compression level reaches a certain threshold - expressed in terms of porosity or strain - the deformation force sharply increases, despite the porosity being still positive. As a result of the high compaction, most of the pores close and the fluid flow vanishes. However, depending on the properties of the solid structure, flow is still possible through some open "tunnels" between compacted structural micro-components.

Darcy’s law is widely used to describe low velocity flows inside porous materials; a key factor in this equation is the permeability. Various investigations of the permeability of porous materials and its dependence on the pore sizes are available. Most of this work was dedicated to the fluid flow through sand and rocks. Carman [1] and Scheidegger [2] are pioneers in the field, being among the first ones who proposed expressions for the prediction of permeability written as a function of porosity and a characteristic dimension of the porous structure. Subsequently, the interest was extended to soft foams for filtering applications. Soft reticulated foams have a high initial porosity (above 0.9) and large pores bordered by beam-shaped walls. During compression, the cell size decreases and correspondingly, the...
permeability as a function of strain. Gent and Rusch [3] are probably the first that found experimentally a correlation between permeability and cell size (diameter) for urethane foams. Two regimes were studied: a low flow regime (laminar flow) where Darcy’s law is applicable, and a high flow velocity regime - when inertial effects are dominant. The transition between the two regimes was found for a pore-based Reynolds number $Re_{p}=1$.

For most porous materials, the reduction of the pore diameter under compressive strain uniformly increases the density of the material. The cellular structure of the reticulated foams behaves in a complex manner when compressed. A distinct characteristic for open-cell foams is that they do not deform homogeneously, and the buckling process of the solid structure appears in layers which are perpendicular on the direction of compression. Gioia et al. [5] and later Li et al. [6] discovered experimentally this phenomenon. Li et al. [6] proposed a model based on two zones that divide the entire volume: a crushed zone, in which the compression strain equals the densification strain, and the elastic zone, which is at the critical state of crushing inception.

Dawson et al. [4] proposed a model for permeability variation with the relative compression level (strain) for low density cell foams; the model includes the variation of the relative density with compressive strain. The formation due to compression of bands of different densities was observed experimentally. Based on this observation, three regions were defined: a central densification region and two elastic regions above and underneath, in the vicinity of the compression plates. Carefully conducted experiments with Newtonian fluids have shown a good correlation with the model. However, the permeability of each specimen was measured only for compressive strains that were less than $\delta_{d}=0.6$.

A literature survey revealed that the elastic buckling of the solid structure occurs around the compression strain $\delta_{el}=0.075$ strain. Multiple layers of cells buckle and collapse and the thickness of the local densification bands of large deformations increase when the overall strain increases. The densification strain is approximately $\delta_{d}=0.6$ for high porosity polyurethane foams [4]. The densification layer has a low porosity and several studies that prove the presence of a residual porosity can be found in literature. Using a numerical model, Bardenhagen et al. [7] proved that the residual porosity inside a densification zone can be up to $\varepsilon=0.35$ for soft porous materials with an initial porosity greater than $\varepsilon_{0}=0.9$.

In this paper, an experimental study of the effect of strain on the porosity of soft polyurethane foams is presented. An original experimental device is used to evaluate the volume of the pores at various levels of compression. The experiments are performed on foams imbied with glycerine, and the volume of glycerine remaining after compression allows the evaluation of open pores volume.

2. Materials and experimental procedure

An original device was designed and manufactured for compression tests with low and medium forces on imbied soft porous materials, in order to measure the fluid squeezed out at various compression levels (figure 1). The device consists of a closed – bottom cylinder, on whose base a disc specimen made of a porous foam imbied with glycerine is placed. The disc is compressed by a piston consisting of two plexiglas discs spaced apart for better guidance by a metallic rod.

During compression, the squeezed fluid flows out of the cylinder through four large circumferential openings (slits) cut near the bottom of the side surface of the cylinder. The extent of these slits covers around 85% of the cylinder circumference to allow the fluid flow as unobstructed as possible. The height of the slits is slightly greater than the maximum, initial thickness of the samples; thus, the free radial flow is possible from the very beginning of the compression. An O-ring placed on the bottom disc of the piston ensures a pure radial flow of the fluid during compression.

The compression was done by placing the device on the frame of a tensile-compression machine and driving the piston with its vertical carriage. Simultaneous control of the load and displacement were possible on two standard testing machines: CETR-UMT-2 – a universal tester for low forces (equivalent to maximum 60kPa) and Zwick/Roell Z010 TN for high forces (up to 200kPa, but with greater capabilities up to 3000kPa).
The tested specimens were Ø64mm discs cut from two similar open-cell polyurethane reticulated foams with different pore sizes and slightly different thicknesses. The dimensions of the specimens were measured with vernier calliper with 0.05mm accuracy.

The porosity of each specimen was carefully measured using a simple volumetric method. A square sample of the foam was cut, measured and accurately weighed and then immersed in a glass graduated tube filled with water. The mass of water inside the glass tube was also weighed with an accurate balance. Inside the tube, the sample was compressed using a metal rod until all the air trapped inside the pores was removed. The weight of the water lost when the rod was removed was measured and the initial volume of water was adjusted accordingly. The final dislocated volume of water was measured on the tube scale. The porosity was calculated by dividing the volume of the solid fraction by the total volume.

The main characteristics of the two materials are presented in Table 1.

| Commercial name (Symbol) | Pore size* [mm] | Material initial density* [kg/m³] | Initial porosity, (ε₀) | Initial thickness, (h₀) [mm] |
|--------------------------|-----------------|---------------------------------|------------------------|-----------------------------|
| FILTREN® TM 25133 (F133) | 1.06÷1.66       | 1150                            | 0.9854                 | 11.5                        |
| FILTREN® TM 25450 (F450) | 3.40÷5.60       | 1150                            | 0.9870                 | 10.5                        |

* according to EUROFOAM Romania

**Figure 1** Experimental setup
A careful evaluation of the parasitic forces generated by the friction between the O-ring and the cylinder was made prior to the experiments. Tests were performed for two contact situations: dry surfaces and glycerine wetted surfaces, respectively. Because some departures from cylindricity were evident for the plexiglas components, four circumferential relative positions of the piston inside the cylinder were considered. Friction force was measured during simulated compression motion of the carriage starting from a height of over 13mm. These tests revealed that the presence of the glycerine does not affect sensibly the friction force, which is less than 15N where the compression of the porous material starts (~11mm), and reduces towards zero when the piston travels over the slitted zone. From figure 2, it is evident that the piston-cylinder friction does not affect significantly the force measurement during compression, except at the very beginning of each series of tests, when the strain is very low. Henceforward, the friction force was neglected.

![Figure 2: Cylinder-piston friction force variation during piston travel on compression stroke](image)

**Figure 2** Cylinder-piston friction force variation during piston travel on compression stroke
(Numbers refer to the four different circumferential positions of the piston)

After the assessment of friction force, precise weighing of each component (dry specimen, piston assembly, cylinder, collecting container) was done using a high precision balance (accuracy 0.01g).

The proper experiments followed a meticulous procedure. Each specimen was subjected to multiple series of compression tests at constant speed. An external load, transverse to the plane of the porous disc, was applied until a pre-set thickness of the specimen was reached. The piston displacement and the axial force were recorded continuously for all the compression tests. The experiments started with a full compression of each specimen in dry conditions (before imbibition) and the stress-strain curves were generated.

The squeeze tests were done at consecutively decreasing smaller thicknesses down to a minimum thickness $h_{\text{min}}=0.4\text{mm}$. Before each compression cycle, the specimen was imibed with glycerine up to its full capacity and weighed. After reaching the pre-set thickness, the collecting container and the specimen were weighed separately. Thus, the amount of glycerine expelled out and the rest of glycerine in the porous matrix could be evaluated by subtracting the original mass of the collector and specimen. The piston and the cylinder were also weighed in order to deduct the amount of glycerine that adhered to their surfaces. Finally, the mass of the remaining glycerine in the porous material was obtained by two ways: (i) directly from the weight of the porous disc and (ii) from the mass of the glycerine collected in the container and that which adhered to the surfaces of the piston and cylinder. If the difference between these two masses was greater than 5%, the test was repeated.

It must be noted that theoretical saturation of both specimens was practically impossible: the materials were not fully imbibed. The F450 material was imbibed with a maximum of 32g of glycerine, which represents 72% of the theoretical value (44.3g). On the other hand, F133 was imbibed with 45.1g, which represents 92% of the theoretical maximum (48.8g). However, we believe
that the partial imbibition does not affect the results for high strain. The pressure produced during compression squeezed out both the air and the glycerine and vanished the voids.

Another source of errors are the liquid losses during the manipulation between the container with glycerine, where the specimens are imbibed, and the location in the cylinder. It was estimated that these losses were no more than 1-2 droplets where a typical droplet weighting about 0.25g.

3. Results
Foams have different types of stress-strain responses in compressions tests, but for the majority, a similar behaviour can be found. A graph presenting the stress-strain variation can be divided in a region of linear elasticity for low stresses, followed by a plateau with a low stress variation, which is delimited by a region of densification with a sharp stress rise. During the elastic response phase, the compression stress increases with cell-wall bending deformations and the macroscopic deformation is uniformly distributed throughout the whole volume [8]. When compression is increased further, the cell walls collapse. Cell wall buckling, cell wall breaking and the formation of plastic hinge in the cell wall, or their combination, could be the reason of cell wall collapse [8].

Figure 3 depicts the stress-strain curves for each of two materials compressed in dry conditions. All compression tests were made at a low compression speed of 0.5mm/s to obtain cvasi-static conditions. The internal forces during deformation are mainly produced by cell-wall bending, and further by collapsing, once the elastic limit is reached. As expected, the differences are hardly distinguishable, as the initial porosity and density is similar for both materials. On the same graph, the maximum stress values recorded for each consecutive compression test are included (shown with markers). These peaks follow practically the same stress-strain evolution for the dry material, which means that the squeeze force is neglectable. A buckling strain of $\delta_b=0.07$ and a densification strain of $\delta_d=0.75$ were found, based on this graph. These values are in accordance with similar values that can be found in literature [4].

![Figure 3 Stress-strain variation for dry and imbibed specimens](image)

The volume of pores as a function of strain can be estimated based on the remaining volume of fluid inside the compressed specimen. This is valid only because we assume that all the open pores are filled with glycerine and there is no air is inside the porous layer. Figures 4 presents the experimentally determined variation of the volume of pores with strain for both specimens; one can remark the linear variation for both cases - a result which was expected.

Using the model of Li et al. [6] the thickness of the densification region and the ratio between the thickness of densification and elastic zones were calculated and graphically represented in figure 4.

One can remark the rapid increase of the densification layer thickness, which becomes dominant for $\delta>0.65$. 
Further, the volume of pores can be calculated using solid fraction conservation (figure 5). Because some fluid can be entrapped inside the densification zone, a correction was included, considering a residual porosity of 0.35. A similar procedure was followed for both foams. From figure 5, one can remark that the predictions made according to the model proposed by Li et al. [6] underestimate our experimental results. This can be explained observing that for high compression strains, the quantity of squeezed fluid was high, and some uncontrolled reimbusition after compression may occur. In the case of the F450 foam, at low strains, the experimentally determined volume of pores was lower due to the lower initial level of imbibition, which was 72%. This means that some of the pores remained filled with air and the experiments could not put this in evidence.

One of the objectives of these experiments was to find the threshold porosity, where the volume of pores remains constant. This limit should appear when the densification region is dominant in the foam volume and the pores are closed. This can be seen from figure 6, where the volume of the glycerine remaining inside the compressed foam is shown as a function of porosity. Porosity variation was found using measured material thickness variation and the conservation of solid fraction assumption. For both materials, the volume of closed pores varied asymptotically to a value of 5cm$^3$ for F133 and 4.6cm$^3$ for F450, respectively. Correspondingly, the residual porosity results being 0.55 for F133 and 0.65 for F450.
4. Conclusions

An experimental study on the variation of porosity during compression with constant speed for two soft polyurethane foams has been reported in this paper. Using a compression-tension machine, the stress-strain correlation for dry materials was experimentally obtained and the elastic and densification strains where graphically found.

Further, the foams were imbibed with glycerine, and after each successive compression, the volume of liquid remaining in the foam was measured by weighting. An original experimental device was used to compress uniformly the foam. This helped with finding the remaining pore volume inside the foam for a known compression level, and further, a porosity–strain variation was found. The results were consistent and showed a good correlation when compared with theoretical predictions from literature. The differences were justified by errors produced by the experimental procedure: reimbibition may occur during the load release phase, and for the F450 foam, the initial imbibition was low and affected the results with respect to low strain.

Based on the correlation between measured volume of pores and porosity, a residual porosity and a limit strain when the pores were closed were put in evidence. The residual porosity was constant and independent of strain, and contributed to finding the limit strain when the densification region was dominant in the foam volume and the pores were closed.

5. References

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Acknowledgments
This work has been funded by the European Social Fund from the Sectoral Operational Programme Human Capital 2014-2020, through the Financial Agreement with the title "Scholarships for entrepreneurial education among doctoral students and postdoctoral researchers (Be Antreprenor!)", Contract no. 51680/09.07.2019 - SMIS code: 124539.