Study of Constant Filling Pressure Conditions in a System ”Nanoporous Medium - Non-wetting Liquid” in an Impact Process

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Abstract. In this work critical constant filling pressure conditions for systems ”nanoporous medium - non-wetting liquid” under impact were studied. To determine the conditions influencing on the occurrence of a constant filling pressure, a series of impact experiments have been carried out on four porous media with distilled water as non-wetting liquid. On the basis of the obtained experimental data for the systems under investigation, a method for determining the flow rate of liquid in the pores was developed and its values for the systems under investigation in a given range of impact energies were determined. It is shown that the liquid flow rate in the pores is one of the key parameters determining the presence of a constant critical filling pressure. It was also found that in the investigated energy range the liquid flow rate increases and goes to limit with increasing impact energy.

1. Introduction

The system ”nanoporous medium - non-wetting liquid” is a smart-system with an controllable response to an impact [1]. Such systems are studied for energy absorption applications more then ten years [2-6]. The response is determined by the characteristics of a porous medium (pore size, surface modification of pores, etc.) and a non-wetting liquid. Energy absorption is a result of filling pores with liquid. A liquid can fill pores only at overpressure, and the energy required to fill the pores is proportional to the intrusion pressure and the volume of pores. The high energy absorption (∼ 10 J/g) of porous media and non-linear response are the basis for the creation of damping devices for protection against impact [1]. A necessary condition for the protection of the object is not to exceed the maximum appropriate force acting on the object. To provide the required reliability of the damping device under development, it is necessary to determine such parameters of the device (liquid, it’s quantity, porous medium, it’s quantity, the material of the device itself, etc.) in which the required protection condition is satisfied. A feature of the systems under study is the existence of a regime of a nanoporous medium filling with a non-wetting liquid at a constant (critical) pressure [7]. This regime is allows to control the maximum force acting onto the protected object. The study of conditions influencing on the occurrence of the constant filling pressure regime under impact of the system ”nanoporous medium - non-wetting liquid” is aim of this work.
2. Materials and Methods

Four hydrophobized silica gels Fluka 100 C18 (cat. no. 60756), Fluka 90 C18 (cat. no. 60757) and Fluka 100 C8 (cat. no. 60755 and cat. no. 60759) distributed by Sigma-Aldrich were studied in this work as porous media. These porous media are different by their pore size, granule size and surface chemistry. Distilled water was used as non-wetting liquid for each porous media. In order to determine the main specific characteristics of the materials under study (specific pore volume $V_{por}$, porosity $\varphi$, absorbed energy $E_{abs}$, relative compressibility $\chi_{rel}$), quasistatic compression experiments were carried out for all used samples accordingly to method and experimental setup described in [8]. The studies were carried out in a high-pressure cell (HPC). The design of the HPC allows measurements with an increase of a internal pressure up to 1000 atm. The HPC consists of the housing, the housing cover, the plug with gaskets and the rod. Porous medium (is up to 20 cm$^3$) in a permeable container placed on a bottom of the housing and into the housing inserted the plug, the free volume of the housing fills by a non-wetting liquid (is up to 60 cm$^3$). After this the housing cover screws onto the housing and the rod inserts into the plug.

Measurement of the pressure in the HPC and a change of the internal volume of the HPC were carried out on the experimental setup. This experimental setup consists of two metal plates connected to each by rods and movable platform which can be moved between plates. The platform is moved in the vertical direction by the screw jack mounted on the bottom plate. The HPC is mounted on a strain gauge force sensor located on the platform, so that the HPC rod touches the upper plate. The upward movement of the platform leads to the rod inserts into the HPC and the pressure increase in HPC. In this case, the force sensor registers the force $F$ with which the load acts on the rod and, therefore, the pressure in the HPC $p = F/S$, where $S$ is the cross-section area of the rod. The measurement range of the force sensor is from 10 to 20000 N. The cross-section area of the rod is $S = 0.785 \text{ cm}^2$. The measurement of the movement of the rod entry and volume change of HPC $\Delta V = l \cdot S$, where $l$ is the length of the rod inserted in the HPC, is carried out using the displacement sensor. The measurement range of the displacement sensor is 0.1 m. When the platform moves down, the rod leaves the HPC and the pressure in it decreases. The signals of the force sensor and displacement sensor are recorded with a frequency of $\sim 1$ kHz.

The results of the processing of experimental data, as well as data from the distributor’s website, are shown in table 1.

Table 1. Characteristics of materials.

| Material          | $V_{por}$, cm$^3$/g | $E_{dis}$, J/g | $\varphi$ | $< L >$, $\mu$m | $< R >$, nm | $\chi$, 1/atm |
|-------------------|---------------------|----------------|-----------|-----------------|-------------|---------------|
| Fluka 100 C18 (60756) | 0.36                 | 6.7            | 0.45      | 20              | 5           | 0.00052       |
| Fluka 90 C18 (60757) | 0.26                 | 6.0            | 0.37      | 52              | 4.5         | 0.00028       |
| Fluka 100 C8 (60755) | 0.46                 | 9.0            | 0.52      | 25              | 5           | 0.00032       |
| Fluka 100 C8 (60759) | 0.41                 | 8.3            | 0.49      | 52              | 5           | 0.00028       |

The experimental study of filling porous media with non-wetting liquid under impact process was carried out at the experimental setup and according to the methods described in the work [7]. A sample of the porous medium in a permeable container was placed in a bottom of HPC and free HPC volume was filled by non-wetting liquid and rod was inserted into the plug. The experimental setup consisted of two plates, fastened by tubes. Steel ropes were stretched between the plates, over which load by mass 10 kg could freely slide. A strain
gauge force sensor was installed on the bottom plate, onto which a sealed HPC was installed. The internal volume of the HPC is changed due to the movement of the rod, inserted into the chamber 7 through the gaskets. The sensor made it possible to measure a force from 10 to 10⁴ N with an error of less than 5% at a force value of more than 100 N. The displacement sensor with a stroke of 15 cm and a measurement error of 0.5% used to determining length of rod inserted into the HPC. In this case, the force sensor measures the force \( F(t) \) with which the load acts on the rod and, consequently, the pressure in the chamber \( p(t) = F(t)/S \). The rate of pressure increase in the experiments was \( dp/dt = (1/8) \cdot 10^4 \) atm/s. The volume change of HPC was as \( \Delta V(t) = l(t) \cdot S \).

3. The Experiment

Systems were tested under the following conditions. Mass of porous media 2 ÷ 14 g with a volume of water 46 ÷ 58 cm³ and impact energy \( E = 4 ÷ 90 \) J. In Fig. 1 shows typical plots of pressure versus time \( p(t) \) and volume changes versus time \(-\Delta V(t)\) for the system Fluka 100 C8 (60755) (4g) - distilled water (53 ml) with a drop height of \( h = 30 \) cm equal to impact energy \( E = 30 \) J.

![Figure 1. Dependence plots \( p(t) \) (a) and \( V(t) \) (b) for the Fluka 100 C8 (60755) (4g) - distilled water (53 ml) system at a drop height of \( h = 30 \) cm equal to impact energy \( E = 30 \) J.](image)

The time interval from 0 to 8 ms corresponds to the compression of the air in the HPC, at times from 8 to 15 ms, the system "nanoporous medium - non-wetting liquid - high-pressure chamber" undergoes elastic compression, after which, from 15 to 25 ms, simultaneously with compression, filling pores with liquid, i.e. impact energy absorption. The time moment to \( t = 25 \) ms corresponds to the stopping point of the system: the rod speed becomes equal to zero. At \( t > 25 \) ms, the rod moves in the opposite direction and the pressure in the HPC decreases. With the known dependences \( p(t) \), \( \Delta V(t) \) and the absolute compressibility of the system \( \chi \), it is possible to calculate the rate of the liquid flow \( v_l \) in the pores at the initial time of filling. Since during elastic deformation, the pressure in the system and the change in its volume are directly proportional:

\[
\Delta V_{\text{def}}(t) = \chi \cdot p(t) \tag{1}
\]

then the dependence \( \Delta V_{\text{def}}(t) \) (Fig. 1(b)) is the elastic part of the volume change; therefore, the value \( \Delta V_{\text{fill}}(t) = \Delta V_{\text{exp}}(t) - \Delta V_{\text{def}}(t) = \Delta V_{\text{exp}}(t) - \chi \cdot p(t) \) (Fig. 1(b)) determines the change of the volume of the system due to the filling of pores with liquid, the time derivative of which gives the liquid flow into the pores \( J(t) = \frac{d\Delta V_{\text{exp}}(t)}{dt} \), i.e.:

\[
J(t) = \frac{d\Delta V_{\text{exp}}(t)}{dt} - \chi \cdot \frac{dp}{dt} \tag{2}
\]
For known values of the average granule size \( <L> \), average pore size \( <R> \), porosity \( \varphi \), and sample mass \( m \), we can determine, under the assumption of pore cylindricality, the total area of the mouths of channels \( S_o \) on the surface of all granules of the sample, then

\[
v_o(t) = \frac{J(t)}{S_o}
\]

\[
S_o = \frac{\pi \cdot m}{2 \cdot \rho \cdot \left(\frac{1}{\varphi} - 1\right) \cdot <R> \left[1 - \left(1 - \frac{4 \cdot <R>}{<L>} \right)^3\right]}
\]

here \( \rho \approx 2 \text{ g/cm}^3 \) for these samples. It is seen that the liquid flow rate in the pores is maximum at the initial moment of filling.

**Figure 2.** The stages of processing experimental data for the system "Fluka 100 C18 (60756) (4 g) - distilled water (53 ml)" with a drop height of \( h = 40 \text{ cm} \) equal to impact energy \( E = 50 \text{ J} \)

### 4. Results

In Fig. 3 shows graphs of the dependences of the liquid flow rate in pores \( v_o \) at the initial moment of filling on the drop height \( h \) for the materials under study. In each case, the initial velocity \( v_o \) reaches saturation with increasing \( h \), i.e. with an increase in the impact energy \( E \), when it follows that for the considered porous media there is the maximum possible velocity of the liquid inside the pores, which is a characteristic of the porous medium. This means that it is impossible to efficiently absorbing an impact energy at a critical constant filling pressure for a fixed mass of a substance with increasing \( E \).

Also, critical initial flow rate \( v_{ocr} \) were obtained, below which a section with constant pressure is observed in the system (Fig. 4).

The condition \( v_o \leq v_{ocr} \) is the condition for obtaining of a critical constant filling pressure in these systems under impact.

### 5. Conclusion

For each studied systems, two limiting flow rates of liquid in pores were found. The first is the maximum flow rate of liquid in pores - is a characteristic of a system "nanoporous medium and non-wetting liquid". The second one is the maximum flow rate of liquid in pores, below which there is a constant pressure occurred. It is a condition for obtaining a critical constant filling pressure in the system "nanoporous medium - non-wetting liquid" in an impact process. Thus, using a second limiting flow rate, it is possible to predict and control the behavior of studied systems under impact.
Figure 3. Dependencies $v_o(h)$ for the systems Fluka100C8 (60755) (4 g) - distilled water (53 ml) (a), Fluka100C8 (60759) (4 g) - distilled water (53 ml) (b), respectively.

Figure 4. Dependences of $v_o$ on $m$ for the systems "Fluka100C18 (60756) - distilled water" ($h = 40$ cm) (a), "Fluka100C18 (60757) - distilled water" ($h = 30$ cm) (b), respectively.

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7. References
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