Towards a new method of porosimetry : principles and experiments

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1. Introduction

Current experimental methods used to determine pore size distributions (PSD) in porous media present several drawbacks such as toxicity of employed fluids (e.g. mercury porosimetry). Theoretical basis of a new method to obtain the PSD of porous media has been proposed in the literature [1, 2]. It is founded on the injection of yield stress fluids, which are characterized by a threshold stress below which they do not flow. The main idea in these works is that injecting yield stress fluids through a porous medium allows to obtain its PSD from the measure of flow rate at various pressure gradients $Q(\nabla P)$. This method has been successfully tested in some cases by Oukhlef [3], using numerically generated experiments. However, obtaining of PSD from $Q(\nabla P)$ data involves calculation of fractional derivatives and this induces that results under real conditions are fluctuating and meaningless. The aim of this work is to present a new approach capable of exploiting experimental data in a more simple and physically meaningful way. The basis of this method is briefly described hereunder and raw experimental results are treated in order to provide PSD and evaluate therefore the performance of the method.

2. The basis of the method

Rheological behavior of common yield stress fluids is well described by the Herschel-bulkley law $\tau = \tau_0 + k\dot{\gamma}^n$, where τ_0 is the yield stress of the fluid, k its consistency and n its flow index. The flow rate of a Herschel-Bulkley fluid flowing through a single capillary of radius r under a constant pressure gradient ∇P is given by :

$$q(\nabla P, r) = \left[1 - 2\left[\frac{(1 - \frac{2\tau_0}{\nabla Pr})^2}{\frac{1}{n} + 3} + \frac{\frac{2\tau_0}{\nabla Pr}(1 - \frac{2\tau_0}{\nabla Pr})}{\frac{1}{n} + 2}\right]\right]\left[\frac{r}{\frac{1}{n} + 1}(\frac{r\nabla P}{2k})^{\frac{1}{n}}(1 - \frac{2\tau_0}{\nabla Pr})^{\frac{1}{n}+1}\right]\pi r^2 \quad for \quad \nabla P > 2\tau_0/r$$

$$q(\nabla P, r) = 0 \quad for \quad \nabla P < 2\tau_0/r$$

Like in standard mercury porosimetry, the porous medium is modeled as a bundle of capillaries whose radii sizes are distributed according to a probabilistic law $p(r)$. Hence, the flow rate through a pore of radius r_i is $q_i = q(\nabla P, r_i)$. Injecting a Herschel-Bulkley fluid through a porous medium leads to N experimental data in the form $(Q_j, \nabla P_j)$ for $j = 1 \dots N$, where $Q_j = \sum q_i$ is the flow rate and ∇P_j is the corresponding pressure gradient. As only those pores whose radius is larger than the critical value $r_c = 2\tau_0/\nabla P$ contribute to the flow, it is assumed that there is no flow below the first detectable flow rate $(Q_1, \nabla P_1)$. Therefore, Q_2 reflects the contribution to the overall flow rate of all those pores joining the flow at pressure gradients between ∇P_1 and ∇P_2, and whose typical radius is r_1. The ratio of this contribution with respect to that of a single pore of this size, q_1, gives the number of pores of size r_1. The next data point, Q_3, reflects the contribution to the total flow rate of the previous pores plus those pores joining the flow between ∇P_2 and ∇P_3, whose characteristic radius is r_2. Here again, the relative importance of this contribution with respect to the individual contribution of a single pore of this size, q_2, gives the number of pores whose radius is r_2, and so on until the number of all scanned pore radii are calculated. By this way, the PSD is therefore obtained.

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3. Experimental and Results

The experimental setup is a classical one: the fluid is injected in the analyzed porous medium using a syringe pump and the stationary pressure loss is measured over a range of imposed flow rates. In this experiment the used porous medium is a sintered silicate (Aerolith ® 10) of cylindrical form (length = 10 cm, diameter = 5 cm). The fluid is a xanthan gum aqueous solution at a concentration of 7000 ppm. Its preparation consists in firstly mixing the water with NaN_3 (bactericide) at 0.02 % w/w and then slowly adding the xanthan powder while stirring. Once all the xanthan powder has been added, the fluid is kept under gentle agitation for 3 days at $20°$ C. The rheology of the obtained solution is then characterized by means of a stress-controlled rheometer (ARG2 from TA Instruments) and fitted to a Herschel-Bulkley law giving $\tau_0 = 8.7Pa$, $k = 0.25Pas^n$ and $n = 0.60$. Figure 1 shows the raw experimental data obtained during this injection (∇P is represented in abscissa axis because of its direct link with critical pore radius). Figure 2 displays results obtained by applying the approach summarized in Section 2 (magenta histogram). In this figure are also displayed the PSD results as obtained for the source porous medium by using the classic mercury porosimetry (blue histogram). It is observed that results obtained by the two methods are in very good agreement . This seems to indicate that it is possible to derive representative PSD by means of simple experiments. It is worth noting that such finding has to be assessed by performing experiments with different kinds of porous media.
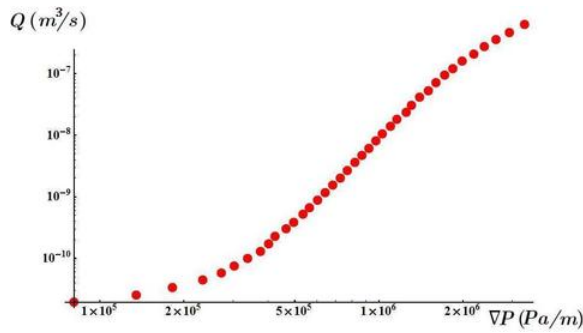

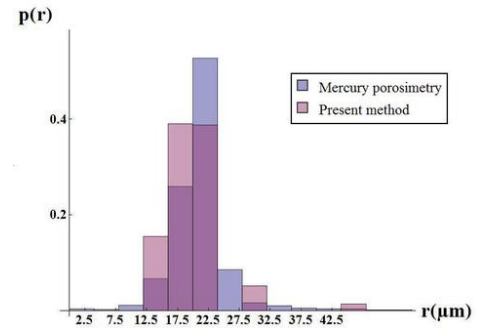Figure 1: Experimental data : $\nabla P(Pa/m)$ vs $Q(m^3/s)$

Figure 2: PSD. New method vs mercury porosimetry (IFP-EN).

4. Conclusion

A new method of PSD determination from yield stress fluids injection experiments is proposed. It is possible to get PSD from $Q(\nabla P)$ raw data with straightforward calculations. Besides, a real experiment has been carried out and its exploitation provides a PSD comparable with mercury porosimetry results. The reliability of this method has now to be tested over a wide range of porous medium samples and fluids, but these first results are encouraging.

5. Acknowledgements

References

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