

OBTAINING PORE SIZE DISTRIBUTION OF POROUS STONE USING NON-NEWTONIAN FLUIDS

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Introduction

Pore space characteristics of building and geological materials have significant scientific and industrial importance due to their association with mechanical, transport, and hydraulic properties. Particularly, pore size distribution is crucial in determining rock susceptibility to weathering processes. Various techniques are employed to determine pore size distribution, but none of them are without limitations or drawbacks, namely the mercury intrusion porosimetry – despite being very popular – is the object of some critics due to the harmful effects of the mercury.

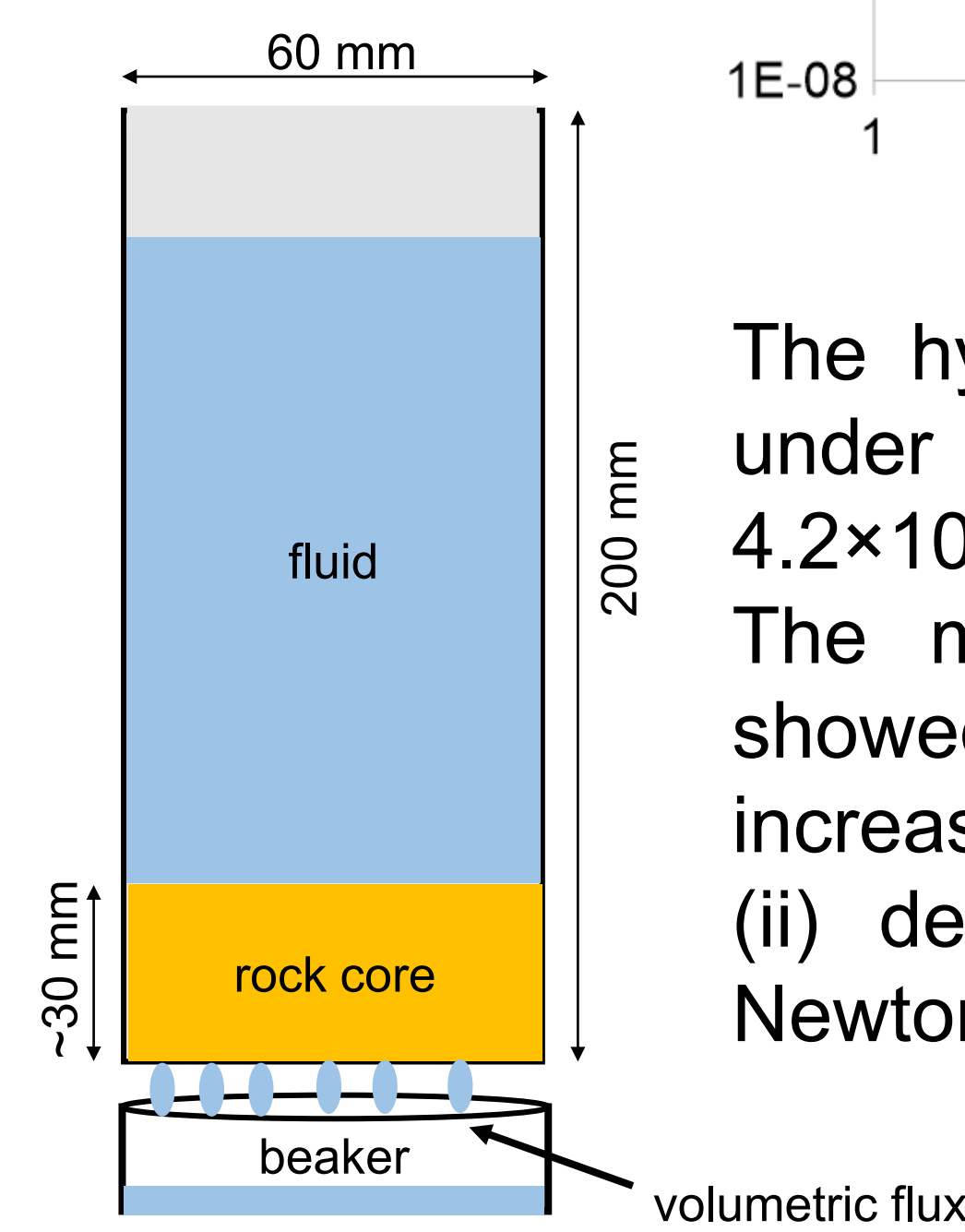
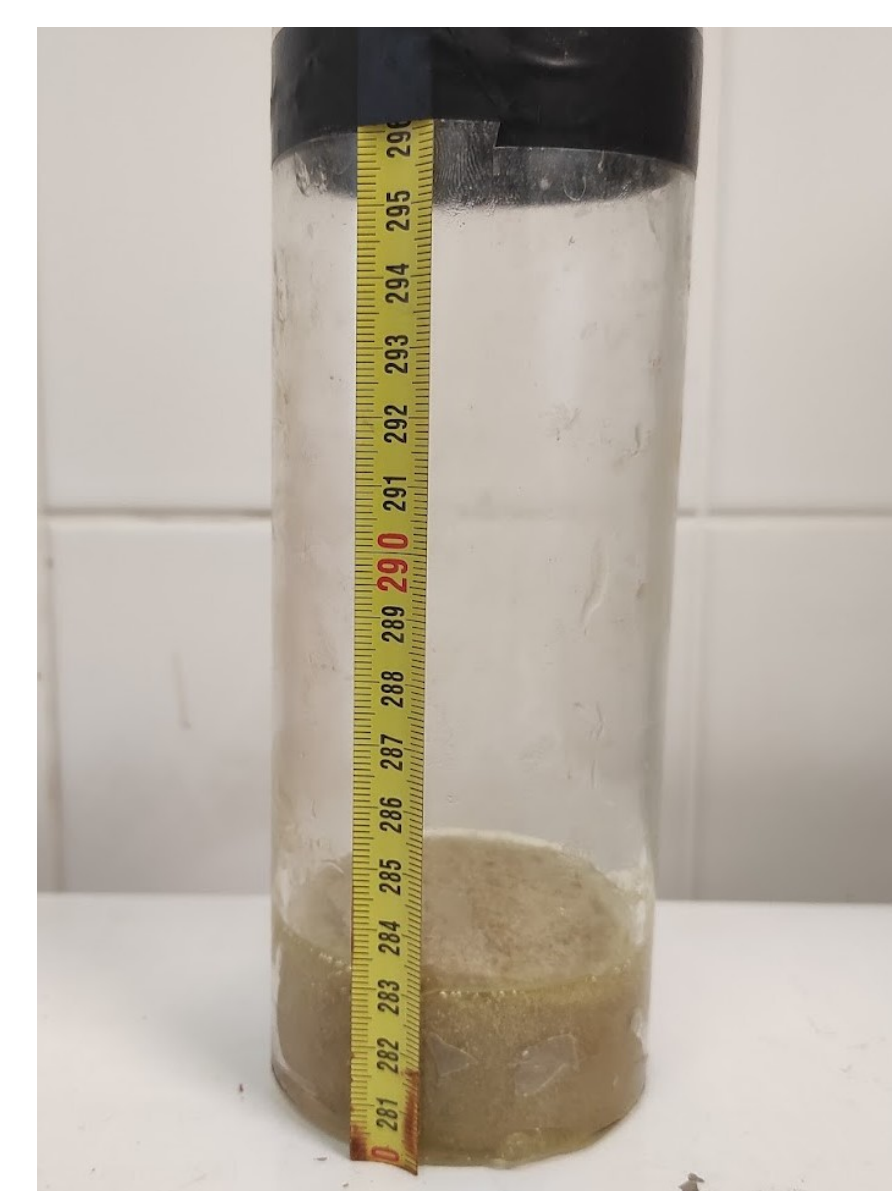
In recent years, interest in using non-Newtonian fluids to determine pore size distribution of porous materials has increased^{1,2}. The principle takes advantage of the viscosity change of non-Newtonian fluids with shear rate. As a result, saturated flow of different fluids and under different hydraulic gradients is distributed differently in the pore space. Thus, conducting several saturated flow experiments with different fluids and/or under different hydraulic gradients allows – using a numerical model – to determine an approximation of the pore size distribution.

Our goal is to test the feasibility of determining pore size distribution in rocks using saturated flow experiments with non-Newtonian fluids at relatively low gradients (<5) and using a simple laboratory technique.

Methodology

We used xanthan solutions (shear-thinning fluids) with concentrations of 0.3 to 1.7 g/l. Their viscosity was measured on a ViscoQC 300 viscometer (Anton Paar). Up until now, we have conducted experiments on three types of quartz sandstone from Bohemian Cretaceous Basin: two from Teplice formation (SR and ST sandstone) and one from Jizera formation (JE sandstone).

Samples in the form of a core ~3 cm high and 6 cm in diameter were inserted into a plexiglass tube (~20 cm long) so as the core's bottom was elongated with one of the tube's opening. The core was sealed into the tube using an epoxy resin, creating an impermeable space between the core wall and the plexiglass wall. For flow experiments, we used water and the xanthan solutions successively from the lowest to the highest concentration. We first saturated the sandstone core from below under 95% vacuum with vacuum-degassed deionized water. Then, we adjusted the fluid level in the tube so that the hydraulic gradient corresponded to a value of about 5.



Model

We adapted a simplified model of flow through a saturated porous medium, which is replaced by bundles of capillaries of given radii. In the volumetric flux, we consider the representation of capillaries of different radii R_i (where $i=1, \dots, N$) with a relative weight w_i . Characterizing the fluid by the xanthan concentration c , the total volumetric flux v under given hydraulic gradient H is the sum of the fluxes q corresponding to N different pore sizes:

$$v(H, c) = \sum_{i=1}^N w_i q(H, c, R_i)$$

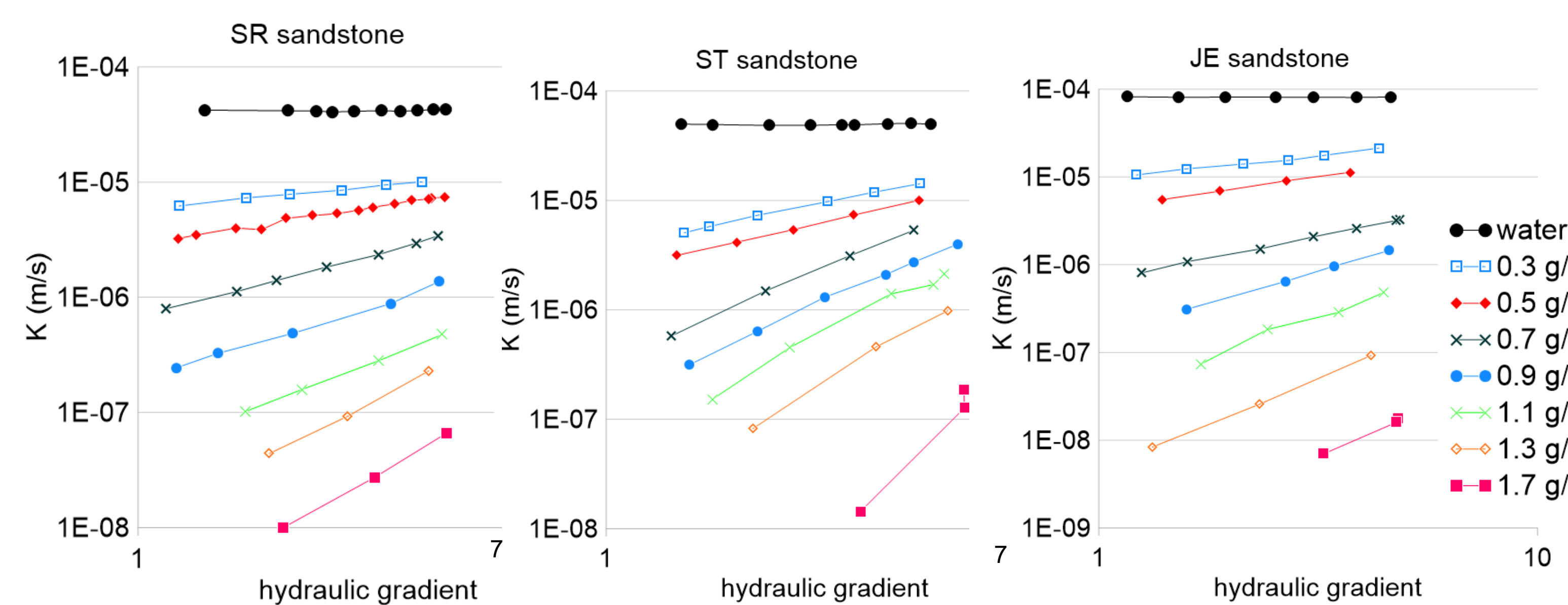
The set of radii R_i and their w_i represent the (discrete) distribution of the effective pore sizes. The method of their estimation is based on the fact that in the case of non-Newtonian fluids, the volumetric flux in the capillary is neither linearly proportional to R^2 (unlike Newtonian fluids) nor proportional to the hydraulic gradient H . Instead, in both cases the dependence is nonlinear, and the nature of the dependence is different for different fluids. Using power-law model for the viscosity, the volumetric flux in a capillary of radius R is given by:

$$q(H, c, R) = C_{geom} H^{1/n} R^{1+1/n}$$

where parameters C_{geom} , n are dependent on the polymer concentration c .

The index n is different at different concentrations, so for each fluid and for each hydraulic gradient we get a resulting flow with different distributions of the total flow into pore bundles with radii R_i . Ideologically, we draw mainly on the work of Abou Najm and Atallah¹. For brevity, we do not mention the role of tortuosity and total (effective) porosity.

Results



The hydraulic conductivity K for measurements with water was constant under different gradients (corresponding to Newtonian fluid) with values of 4.2×10^{-5} m/s for SR, 4.9×10^{-5} m/s for ST and 8.1×10^{-5} m/s for JE sandstone. The measurements with xanthan solutions of different concentrations showed that the apparent hydraulic conductivity (i) generally decreased with increasing concentration of the solutions (due to their higher viscosity) and (ii) decreased with decreasing hydraulic gradient (indicating the non-Newtonian behavior of the fluids).

By solving an inverse problem, we calculated the relative representation of the pore sizes from the measured data. The comparison with conventional mercury intrusion porosimetry (MIP) indicates that the numerical model demonstrates similar pore sizes and a reasonably consistent match with the MIP curves.

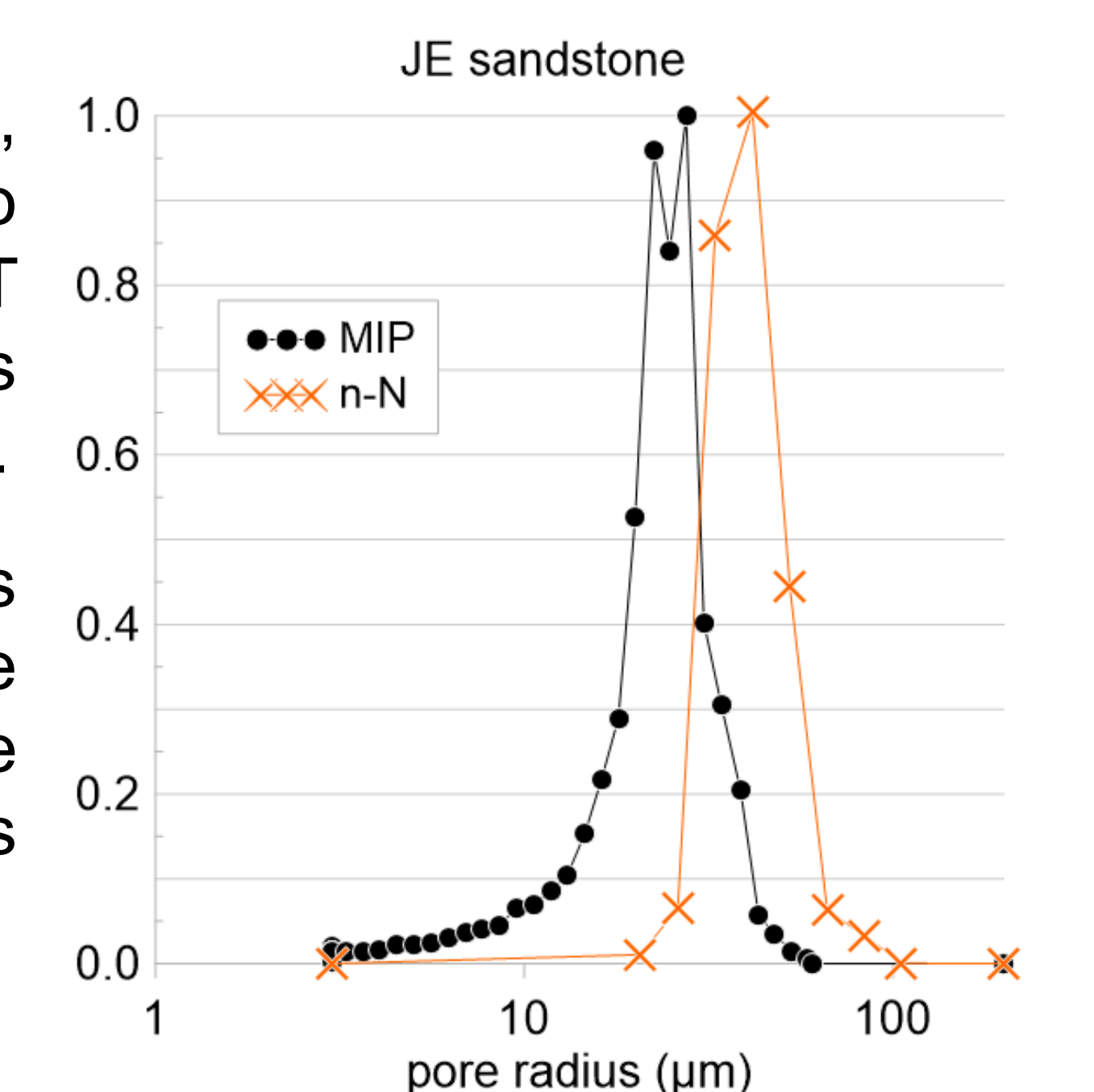
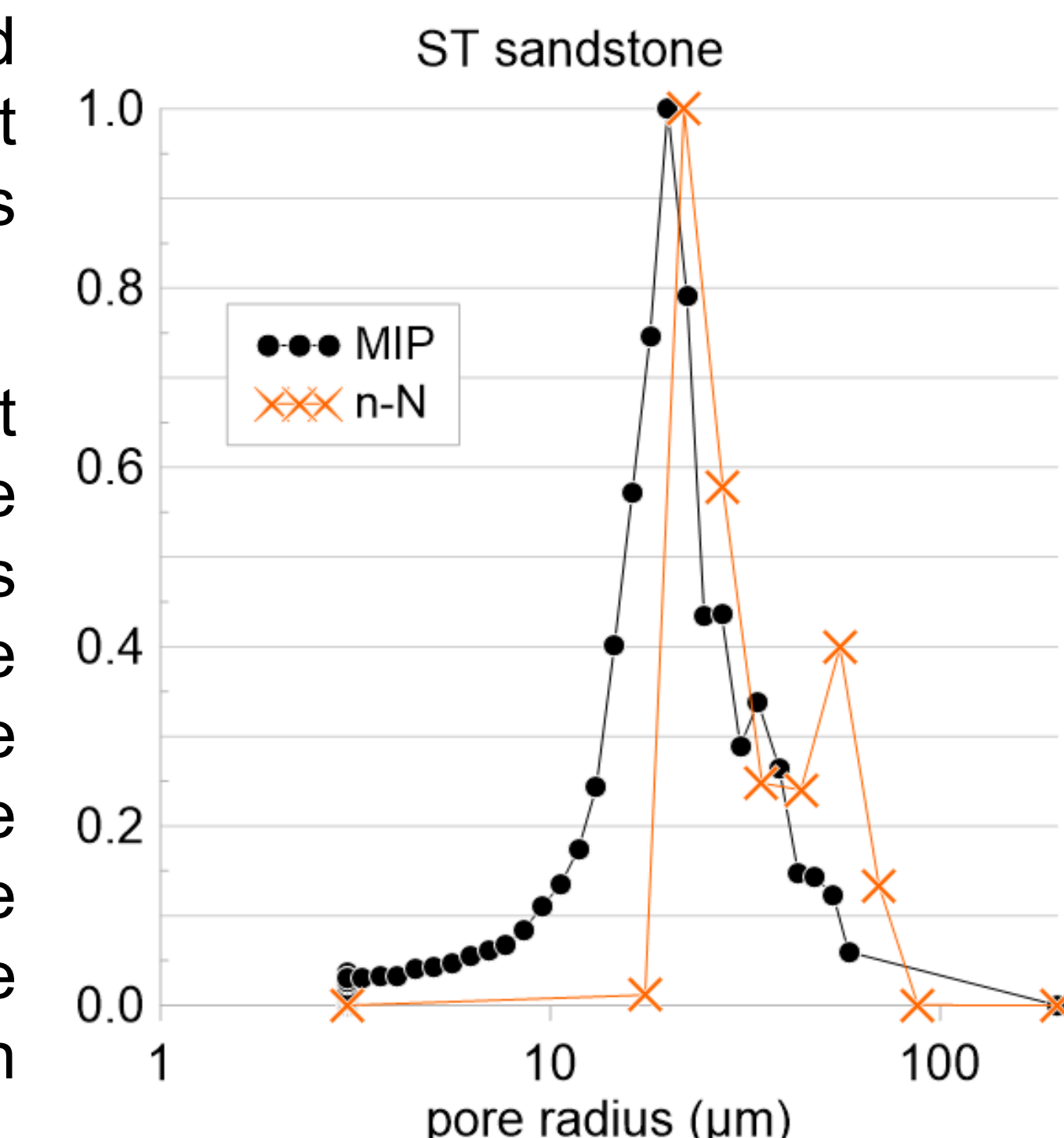
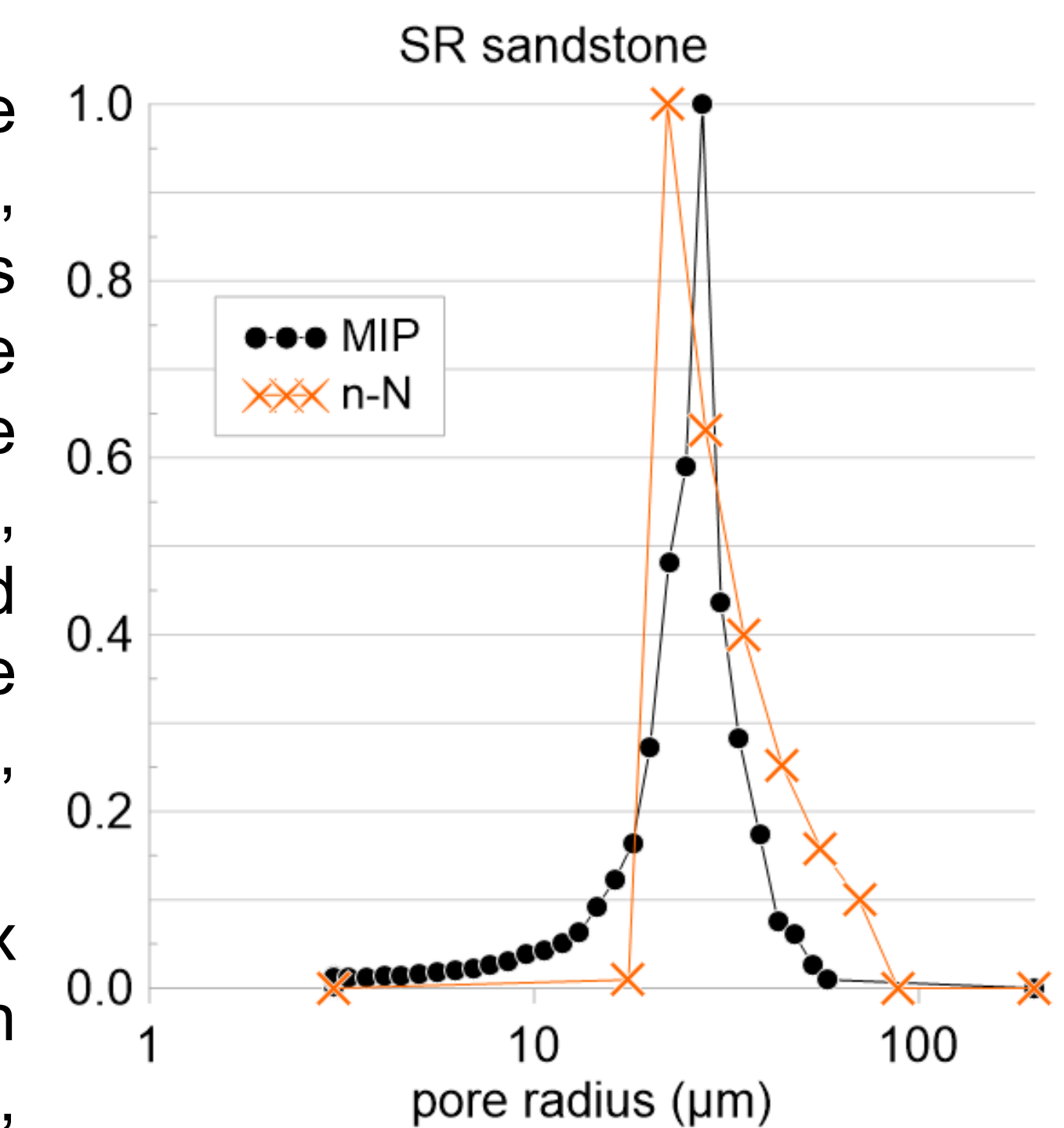
The pore sizes of both methods are within the same order of magnitude, and the peak widths of the curves are also fairly similar. The tops of the peaks, which represent the pore size with the highest abundance, obtained from the n-N fluids method are somewhat displaced from the peaks derived from the MIP method, and the discrepancy is 2–14 μm .

Pore size distribution is a complex characteristic that is dependent on the specific method used. Therefore, differences between curves obtained from different methods should not automatically be interpreted as inconsistencies or errors.

We would like to emphasize that although non-Newtonian fluids have been already used in previous studies to determine pore size distributions, the procedure presented here seeks to maximize the technical simplicity of the laboratory methodology and the results obtained are a pilot study in this regard.

We consider the results promising, and the method shows potential to serve as a cheap alternative to μCT and MIP methods in geosciences and various cultural heritage studies.

An ambitious goal for the future is the analysis of the reliability of the results depending on the choice (acceptable number) of experiments performed.



References

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2. Rodríguez de Castro A., Omari, A., Ahmadi-Sénichault, A., Savin, S., Madariaga, L-S., 2016: Characterizing Porous Media with the Yield Stress Fluids Porosimetry Method. *Transport in Porous Media*, 114, 213–233.