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Integrated measurements of permeability, effective porosity, and specific storage of core samples using water as the pore fluid

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1. Introduction

Permeability (k), effective porosity (Φ), and specific storages (S_s) are the parameters which are often concerned when evaluating the transport properties of porous rocks. Ideally, the intrinsic transport properties of the rocks do not depend on the type of pore fluid; therefore both gas and liquid can be used as the pore fluid for the laboratory measurements, although the Klinkenberg effect on gas permeability needs to be corrected. Due to the high chemical stability and high performance convenience, $1,2$ $1,2$ $1,2$ gas has been favored by most rock-physicists. $3,4$ $3,4$ $3,4$ In addition, the compressibility and viscosity of gas are less sensitive to temperature variations than those of water, so the measurement error due to these factors is less likely to be introduced. When gas is the pore fluid, permeability can be measured by the steady-state or a transient method such as a pulse-decay and pore pressure oscil-lation technique,^{[5](#page--1-0),[6](#page--1-0)} and porosity by the gas expansion method, based on the Boyle–Mariotte equation for an isothermal gas.^{[7,8](#page--1-0)} By properly arranging the procedure of the measurement, permeability and porosity of a dry sample can be measured concurrently in a single experiment. 9

Under most natural conditions at the upper crustal depth, rocks

<http://dx.doi.org/10.1016/j.ijrmms.2015.08.007> 1365-1609/© 2015 Elsevier Ltd. All rights reserved. are expected to be water-saturated, so the gas properties are unlikely to be directly relevant except in some circumstances such as in the gas fields. Previous studies have shown that permeability of a sample to gas is higher (up to 1.8 orders of magnitude) than the permeability of the same sample to water, even after correction for the Klinkenberg effect. $9-13$ $9-13$ $9-13$ For some rock types like shale, strong physicochemical interactions may occur between the skeletal grains and the aqueous pore fluid, $2,11$ which influences the flow characteristics of these rocks.¹⁴ Against this background, it is important to determine the transport properties of porous rocks under water conditions. However, in laboratories liquid was seldom used as the pore medium for the concurrent measurement of these parameters.^{[15](#page--1-0)-[18](#page--1-0)} The most significant difficulty lies in the measurement of porosity. The current standard techniques for porosity measurement (mercury intrusion porosimetry, 2-D image analysis, CT, NMR imaging, and dry vs. wet weighing) cannot be readily applied under high confining pressure. The use of a highprecision volumetric pump allows for the quantification of the pore volume change in response to the change in confining pressure,^{[15](#page--1-0),[19](#page--1-0)} but it cannot tell the absolute, effective porosity of the sample.^{[16](#page--1-0)} To solve this problem, some measured the initial water porosity at atmospheric pressure by means of a standard method stated above.^{[16,20](#page--1-0)–[23](#page--1-0)} Alternatively, some measured the gas porosity of a dry sample at an initial pressure and then transferred to water condition at the same pressure. 8 However, these strategies encounter problems if incohesive rocks such as fault rocks are involved, or the sample measured contains swelling mineral(s) or

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generally contains abundant phyllosilicates. Adsorption of water can cause clay clusters to expand in size, even in the absence of swelling clays, and reduce the pore space.^{24,[25](#page--1-0)} Delamination and migration of particles, which then block the small pore throats, is also possible in the presence of abundant phyllosilicates, $12,26$ because the clay minerals usually have lower frictional strength when wet than dry, 27 thus facilitating reorientation of particles during compaction. Taken together, it is important to develop a method for determination of the absolute porosity under in-situ hydrological conditions.

Recently, a fluid-flow apparatus was developed at the Institute of Geology, China Earthquake Administration. By employing several techniques, this system allows for the integrated measurements of permeability, effective porosity, and specific storage, under elevated confining pressures.

2. Experimental method and procedure

As a conventional fluid-flow apparatus, the system consists of a pressure vessel, along with the standard units for controlling confining and pore pressures, as shown in Fig. $1⁸$ $1⁸$ $1⁸$ With an intensifier, the confining pressure can reach a maximum of 200 MPa, with fluctuations varying within 0.3 MPa. A maximum fluid pressure of 40 MPa can be used for the steady-state flow method, and a maximum mean value of \sim 36 MPa for the oscillatory fluid pressures when using the transient method. To maintain constant temperature in and around the sample, the pressure vessel, along with the connecting tubing, valves and fittings that are crucial for the measurement, are enclosed in a polystyrene-foam chamber in size of \sim 1 m³ (see the box in Fig. 1). The temperature probe used (Fluke 1523) has a resolution of 1 mK. Using a digital temperature controller, lamp heater and fan (Fig. 1), the temperature within the chamber can be controlled at a desired value (25–30 °C), with fluctuations less than 0.1 K throughout an experiment. In the measurement, by choosing different "fluid reservoirs", either gas

or liquid can be used as the pore fluid (Fig. 1). Independent pipelines are used when the gas and liquid work as pore fluids. In the present study, only liquids were employed.

2.1. Method for individual parameters

2.1.1. Permeability (k)

The versatile measurements of permeability were enabled by employing two syringe pumps (" P_f self-controlled unit"), as illustrated in Fig. 1, which can provide a constant flow (5–40,000 μL/ min) and a sinusoidal oscillating pressure $(20 s <$ period $<$ 2400 s). In regards to the steady-state flow method, the flow rate was determined by a balance or a high-precision flowmeter (Fig. 1). The pore pressure oscillation (PPO) method has been well de-scribed in previous publications.^{[28,29](#page--1-0)} Therefore, only a brief description is given here. A pressure signal of the sinusoidal oscillation was imposed at the upstream end of the sample, and the response at the downstream end was recorded ([Fig. 2](#page--1-0)a). A Fourier analysis was performed on the two signals to obtain the amplitude ratio ($0 < \alpha < 1$) and phase delay ($\theta < 0$). From these two measured parameters, two dimensionless parameters were calculated using a numerical analysis based on the equations given by Fischer and Paterson,^{[6](#page--1-0)} from which the permeability (k) and specific storage (S_s) of the sample were calculated. The relative uncertainties for k and S_s were further determined following the method given by Bernabé et al.^{[29](#page--1-0)}

The storage of the downstream reservoir (B_d) is the most important parameter for the PPO method, and in previous studies was either calibrated by subtracting the upstream storage from the storage of the entire system, 6 or calculated through multiplying the system volume by fluid compressibility.^{[5](#page--1-0)} Our verification tests indicated that these methods could introduce relatively large errors in B_d , and thus in the measurement results, particularly for S_s . In our system, B_d was directly measured using a specifically designated microvolumeter, which basically consisted of a valve and a LVDT (Fig. 1). The variable B_d could be achieved (if needed) by

Fig. 1. Diagram showing the fluid-flow system for integrated transport property measurements.

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