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Practical approaches for addressing shale testing challenges associated with permeability, capillarity and brine interactions

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HIGHLIGHTS

- Shales have negative pore pressure when not under stress, even if fully-saturated.
- Undrained compression creates positive pore pressure and mitigates testing issues.
- Sample water content pre-conditioning speeds attainment of desired effective stress.
- Testing protocols should be designed around the permeability of the shale.
- Testing protocols should consider osmotic forces, and capillary pressure.

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ABSTRACT

Shales present several challenges for mechanical testing. Modal pore throat sizes range from a few nm to a few tens of nm, and these small pore sizes result in extremely low values of permeability and of consolidation coefficient. They also allow high values of capillary suction, even in well-preserved fully-saturated shales. Total suction can be even greater than capillary suction, mainly due to effects associated with clay surfaces. Clays also result in chemico-osmotic forces that cause a hydraulic pressure difference between the shale pore fluid and the brine in an external pore line or reservoir. All these characteristics directly impact testing protocols. The first step in any test should be to apply sufficient confining stress to raise the pore pressure up to a positive, measurable value. Undrained isostatic compression, combined with undrained triaxial compression and with small sample sizes (and drainage screens when necessary), results in relatively short test durations that still allow for pore pressure equilibrium throughout loading and failure. A range of effective consolidation stress values can be attained by first equilibrating shale samples in varying amounts of suction, to vary the water content. If actively raising the sample pore pressure or saturation can be avoided, then methods can be used that do not require any brine contact.

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1. Introduction

The behavior of shales is important to many fields associated with energy and the environment, such as oil and gas drilling and production, CO₂ sequestration, and containment of radioactive or other wastes. To define the mechanical behavior of shales in the laboratory, or coupled poromechanical, thermo-poromechanical or chemo-poromechanical behavior, requires that the pore pressure throughout the sample be known and measurable. This is made difficult by three characteristics of shales: extremely low permeability, high capillary suction (capillary tension) when unstressed, and various interactions with brines.

One of the most important aspects of shales is that, when not under sufficient stress, the pore pressure within a sample is highly negative. This is true even for shales that are fully-preserved

and fully-saturated.¹⁻³ The presence of negative pore pressure within the sample can lead to testing artifacts and to incorrect test interpretation,^{4,5} since its value cannot be directly measured. One of the best methods for handling this is to apply sufficient stress to the sample to negate the capillary tension and bring the pore pressure up to a positive and measurable value.^{5,6} This paper presents examples of this approach, and illustrates how sample pre-conditioning with controlled relative humidity makes this a practical method.

Loading rates that are too fast for the sample permeability and the drainage conditions can lead to excess pore pressure within the sample, which cannot be measured or known.⁷ Chemico-osmotic forces can arise when using brine as an external pore fluid, and this leads to sample internal pore pressure being different than the externally-measured pore pressure.^{8,9} Solutions to these issues are available, and are presented in this paper. Test methods for triaxial testing are presented which result in the sample pore

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pressure being always known, always measurable, and always at equilibrium within the sample.

This paper consists of two main sections. The first section provides quantitative values for key shale properties such as pore size, permeability, consolidation coefficient, native-state (fully-preserved, unstressed) total suction, capillary suction, saturation, osmotic membrane efficiency, and osmotically-induced hydraulic pressure differences. Examples are given covering shales from 0.15 to 0.4 void ratio. The second section of the paper presents example approaches for mechanical testing which can both shorten overall test duration and avoid test issues associated with capillary suction and with osmotically-driven hydraulic pressure differences.

The results presented in this paper were obtained on claystones. Claystones are defined as mudstones (compacted and indurated mud) which are dominated by clay-size particles. The claystones used in this study are clay-supported (67% to 76% total clay), with silt grains generally not touching each other. The fundamental load-bearing particles in these rocks are clay 'grains' or clay 'aggregates', on the order of 0.1 μm thickness and at least one order of magnitude greater in width. Space exists outside these grains, which makes up most of the total water-bearing space in these rocks and also provides the permeability flow paths. Claystones will be referred to as shales in this paper. Basic properties of the studied claystones are listed in Table 1. Complete descriptions, including mineralogy and cation exchange capacity, can be found in Ref. 10.

2. Characteristics of shales which create testing difficulties

Two important characteristics of shales are (1) very small pore sizes, and (2) significant clay content. The small pore sizes cause extremely low values of permeability and consolidation coefficient. They also allow high capillary suction values and very strong capillary forces, even in samples which are fully or almost fully saturated, especially when confining stress is low or zero. Although the small pore size helps to increase the air entry pressure, most shales that have not been carefully preserved are found to be only partially saturated. As most shale tests require samples to be fully saturated and contain known and measurable pore pressure, various procedures need to be adopted to ensure full saturation while avoiding uncontrolled fluid content increase driven by the strong capillary forces.

The presence of clays adds additional requirements to such procedures. Various interactions with brines and water can occur, such as swelling, strength alteration, and hydraulic pressure difference associated with osmotic-type forces. While clays do increase the water retention capability of shales, which is desirable in avoiding desaturation, they add to the total suction. This increases the driving forces for uncontrolled water content increase and clay swelling. The significant clay-related components of total suction also make it difficult to quantify capillary suction, and make it difficult to exactly balance the 'pore water activity' with an external brine since this activity (escaping tendency) is affected by near-clay water and is generally lower than that of the free pore water (the suction is greater than the solute suction).

The remainder of this section will more fully explain the above points.

2.1. Typical pore size distributions

As a result of geologic burial and compaction, the pore sizes in shales (claystones) are extremely small. While the clay interlayer space is too small to be accessed even by high-pressure mercury, the pore structure that exists external to the clay grains (clay aggregates) is mostly accessible, and this represents the majority of the total pore space in a typical shale. Fig. 1 shows normalized pore size

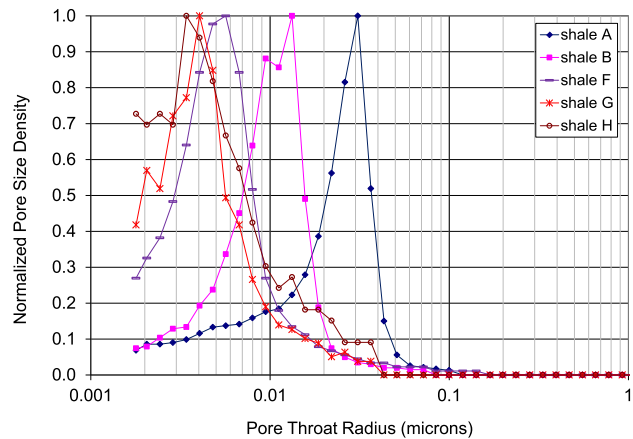


Fig. 1. Normalized pore size distribution for five example shales, from high-pressure mercury injection.

distributions from high-pressure mercury injection porosimetry (MIP) for five example shales. These five shales have the total void ratio and porosity values listed in Table 1 in their native, fully-saturated states. These native-state values are based on total water weight loss upon oven drying. Of the total porosity, the water that exists within the clay grains is thought to represent about 3% to 6% of total sample bulk volume for these and similar shales¹⁰; the remainder of the measured total porosity is external to the clay grains. The intra-grain space consists mostly of clay interlayer water, typically just one to two water layers in thickness, with the clays within each grain being physically aligned and forming crystallographically non-coherent stacks.¹⁰

The MIP data were corrected for system and mercury compression, and for apparent intrusion (due to surface imperfections and/or cracks) at low mercury pressure, prior to true intrusion. Samples were prepared for MIP by applying vacuum for 24 h at 63 °C, followed by 48 h at 38 °C. This results in some sample shrinkage. The estimated dried-state porosity of the MIP samples is listed in Table 1. These estimates were performed using the observed shrinkage trends (void ratio vs. total suction) for these shales¹⁰ combined with data for other claystones.¹² The modal pore sizes for these dried samples (Fig. 1) have likely been shifted slightly smaller than the modal pore sizes in a native, saturated state.¹¹

The modal pore throat sizes in Fig. 1 are seen to span about one order of magnitude, from ~ 3 nm to ~ 30 nm equivalent radius, and are smaller for lower porosity. While possibly shifted slightly smaller due to sample drying, it is clear that in-situ compaction due to burial results in smaller pore sizes as porosity is reduced. These five shales come from four different regions around the world, and they contain different types of clays.¹⁰ However, all of them contain between 67% and 76% total clay, which means that clay grains, and not silt grains, are the load-bearing matrix. The pores external to the clay grains are reduced in size as the geologic stress increases.

Values of in-situ stress for each of the shales are listed in Table 1; each shale is believed to be currently at or very close to its maximum past stress. Vertical stress is based on integrated bulk density logs and horizontal stress is based on hydraulic microfracture tests (casing shoe leakoff tests). Pore pressure is from the best possible estimates, in most cases constrained by direct downhole measurements in nearby permeable formations.

2.2. Permeability and pressure diffusivity

Shales have quite low values of permeability, and of pressure diffusivity (consolidation coefficient, c_v), mainly governed by

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