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# Experimental chemoporoelastic characterization of shale using millimeter-scale specimens

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## ABSTRACT

The development of reliable experimental techniques for characterization of chemoporomechancial shale–fluid interactions is important for the design of drilling fluids that maximize shale stability. In this context, testing of millimeter-scale specimens is promising because small specimens require shorter test durations and are more readily available from offcuts of preserved core or potentially from drill cuttings or wellbore cave-in material than larger core plugs that are required for more conventional experimentation. Here we present experiments wherein we measure the axial displacement of 4 mm long by 4 mm diameter cylindrical shale specimens that are subjected firstly to a mechanical axial loading and then to an osmotic loading associated with a sudden increase in the salinity of the surrounding fluid. The response to both stages of loading is consistent with theoretical, chemoporoelastic predictions. In particular, the model predicts two types of behavior depending on the ratio between the reflection coefficient and the so-called chemomechanical coupling coefficient that quantifies the volumetric strain as a result of a change in ion content. Consistent with predictions, both monotonic shrinkage and initial shrinkage followed by partial recovery are observed in our testing campaign which includes 20 shales from a variety of geological settings. Quantitative characterization is also carried out by selecting chemoporoelastic parameter values that minimize the mismatch between the data and the model. The results show that the reflection coefficient and the chemomechanical coupling parameter are correlated with each other and with both the Cation Exchange Capacity (CEC) and the Specific Surface Area (SSA). Based on the consistency of the data from test to test and with the model, together with the fact that the key chemoporoelastic coefficients are sensibly correlated with CEC and SSA, we conclude that these millimeter-scale experiments are able to provide useful characterization for better understanding and predicting shale–fluid interactions.

## 1. Introduction

The interaction between shale formations and water-based fluids is a widely recognized source of challenges for the petroleum industry. For most of the history of this topic, the main concern has been the impact on stabilizing methods for drilling through shales en route to the reservoir layers (e.g. Chenevert, 1970a), although recent interest is also motivated by the need to minimize negative impacts on the transport properties of shale reservoirs associated with fluid leakoff from hydraulic fracturing (Bahrami et al., 2012).

The petroleum industry has been investigating the tendency of shales to swell in the presence of low-salinity water since at least the early 1940s (Power et al., 1942; Chaney and Oxford, 1954), with research efforts eventually leading to recommendations for modifying the composition of drilling fluids in order to promote shale stability through oil emulsion and salinity balanced drilling fluids (Mondshine and Kercheville, 1966; Chenevert, 1970a,b). Following these seminal works, there was an emphasis on characterizing the impact of various emulsion agents and salts on the behavior of the clay mineral constituents of shales through the 1970s and 1980s (e.g. Clark et al., 1976; Roehl and Hackett, 1982; Nesbitt et al., 1985; Bol, 1986).

Then, in the 1990s, the attention turned to incorporation of fluid–shale interaction into wellbore stability models. This drive

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gave rise to a number of thermodynamics-based chemoporomechanical models (Sherwood, 1993, 1994; Heidug and Wong, 1996), micromechanical models (Pride, 1994; Revil and Leroy, 2004), as well as partial mechanical models based on the concept of membrane efficiency (Mody and Hale, 1993). The concept of membrane efficiency birthed a new line of research aimed at its characterization in the laboratory (Yew et al., 1993; van Oort et al., 1996; Tan et al., 1998, 2002), with a particular impetus provided by the environmentally driven move towards completely water-based drilling fluids (Tan et al., 2002). The past decade, then, has brought several extensions of poroelastic or poroelasto-plastic theory that accounts for coupling among the hydraulic, chemical, mechanical, and in some cases thermal, fields (e.g. Ghassemi and Diek, 2003; Ekbote and Abousleiman, 2006b; Sarout and Detournay, 2011; Roshan and Rahman, 2011). Among the accomplishments of these models, they demonstrate that the membrane efficiency or equivalently the reflection coefficient in the context of the model used here, is the key quantity that must be characterized in order to make informed decisions regarding alteration of mud salinity in order to promote wellbore stability. Furthermore, and arguably most importantly, these past works demonstrated a connection between the membrane efficiency/reflection coefficient and engineering practice (see case studies in e.g. Tan et al., 1999, 2009), thus establishing a critical link between theory and the behavior of real wellbores that forms the practical foundation for our research.

In spite of a long history of theoretical and experimental developments, chemoporomechanical characterization of shale remains arduous. Firstly, each theory relies on values of relatively large number of parameters compared with – say – a linear elastic or even a classical poroelastic analysis. The difficulty is compounded not only by issues of availability of well-preserved materials, but also by the fact that the very low matrix permeability leads to test durations on the order of weeks to months when experiments are performed on specimens at the usual tens of millimeters scale (Tan et al., 2002; Sarout and Detournay, 2011).

One important line of research that is aimed at overcoming some of these difficulties focuses on developing experimental methods that can be performed on millimeter-scale specimens. Some key successes include the measurement of acoustic properties (Nes et al., 1998) and strength properties (Abousleiman et al., 2010). Another approach to addressing difficulties in shale characterization is to seek to correlate chemoporomechanical properties, which in general involve highly time consuming experimentation, with physicochemical properties that are more easily obtained (e.g. Josh et al., 2009). By combining demonstration of a new millimeter-scale experimental method with independent measurements of physicochemical properties for a suite of 20 shales, this research contributes to both of these approaches to shale characterization.

In this context, the main contributions of this paper are twofold. The first component is the demonstration of an experimental technique that is able to characterize several of the most important chemoporomechanical properties of a shale using millimeter-scale specimens and with total experimental run times on the order of a couple of days. In this regard we present a method which has evolved from the initial, indicative procedure that was presented in a conference paper several years ago (Bunger and Detournay, 2008), wherein our results were promising, but limited at that time to just a couple of questionably preserved specimens. We have now been able to apply a refined experimental protocol, which we describe in this paper, to the testing of 20 preserved shales representing a variety of depositional and thermo-tectonic environments. As a result, we are able to experimentally explore the behavior of shales with a wide range of clay mineralogy and physicochemical properties such as Cation Exchange Capacity (CEC) and Specific Surface Area (SSA). It is this testing of a wide range of shales, enabled both by availability of materials (courtesy of the members of a research consortium) and relatively short testing times, that leads to the second component of this paper's main contribution, which is the demonstration of correlations between CEC and SSA and both the reflection coefficient and chemomechanical coupling coefficient by which the latter quantities could potentially be usefully constrained based on CEC and/or SSA measurements.

## 2. Mathematical model

For an isotropic shale under isothermal conditions, a linear chemoporoelastic model is given by Sarout and Detournay (2011):

(1) Constitutive equations relating the volumetric strain $\epsilon$, the Biot fluid content $\zeta$, and the relative ionic content $\theta$ to the mean stress $\sigma$, hydraulic pore pressure $p_h$, and osmotic pore pressure $p_c$, given by

$$\begin{pmatrix} \varepsilon \\ \zeta \\ \theta \end{pmatrix} = \overline{C} \begin{pmatrix} \sigma \\ p_h \\ p_c \end{pmatrix}, \quad \overline{C} = \begin{pmatrix} C_d & bC_d & -\alpha bC_d \\ bC_d & \dfrac{bC_d}{B} & -\beta\dfrac{bC_d}{B} \\ -\alpha bC_d & -\beta\dfrac{bC_d}{B} & \gamma \end{pmatrix}, \tag{1}$$

where the constitutive parameters are defined below and the osmotic pressure is given according to the Van't Hoff law by $p_c = R_g T c_s$, where $c_s$ is the molar salt concentration, $R_g$ is the gas constant (8.31 J/mol K), and $T$ is the absolute temperature (van't Hoff, 1887). Naturally, $\sigma = \mathrm{tr}(\overline{\sigma})/3$ and $\varepsilon = \mathrm{tr}(\overline{\varepsilon})$, where $\overline{\sigma}$ and $\overline{\varepsilon}$ are the Cauchy stress and linear strain tensors, respectively. These tensorial quantities are related according to

$$2G\overline{\varepsilon} = \overline{\sigma} - \frac{3\nu}{1+\nu}\sigma\overline{I} + \frac{1-2\nu}{1+\nu} b\overline{I}(p_h - \alpha p_c), \tag{2}$$

where $\overline{I}$ is the second rank identity tensor and $G$ is the elastic shear modulus.

(2) Linear momentum balance, which, taking body forces as zero, is

$$\nabla \cdot \overline{\sigma} = 0. \tag{3}$$

(3) Strain–displacement relationship given, for displacement vector $\overline{u}$, by

$$\overline{\varepsilon} = \tfrac{1}{2}(\nabla\overline{u} + {}^{T}\nabla\overline{u}). \tag{4}$$

(4) Mass balance equations for the fluid and ionic phases, given by

$$\nabla \cdot \overline{q} = -\dot{\zeta}, \quad \nabla \cdot \overline{w} = -\dot{\theta}, \tag{5}$$

where $\overline{q}$ and $\overline{w}$ are the fluid and ionic fluxes, respectively.

(5) Coupled transport equations given by

$$\overline{q} = -\kappa\nabla p_h + \kappa\mathcal{R}\nabla p_c \tag{6}$$

$$\overline{w} = \mathcal{R}\kappa\nabla p_h - \frac{D_c}{N}\nabla p_c \tag{7}$$

where $N = R_g T/\upsilon_s$ with $\upsilon_s$ representing the molar volume of the salt ($\upsilon_s$=0.03 L/Mol for NaCl).

Upon the addition of appropriate initial and boundary conditions, Eqs. (1)–(7) comprise a complete system which can be solved for the unknown quantities $\overline{u}$, $\overline{q}$, $\overline{w}$, $\overline{\varepsilon}$, $\overline{\sigma}$, $\zeta$, $\theta$, $p_h$, and $p_c$.

We see, then, that characterization of the shale consists of obtaining values for the 10 phenomenological parameters upon