
Introduction

A significant proportion of the gas saturation in shale reservoirs is adsorbed on the rock surface, attached to organic matter, or to clay minerals. Adsorbed gas parameters along with other geomechanical properties are used to design an economically successful field development plan, ensuring maximum possible gas recovery within a specified period of time. Current practices for determining the adsorption capacity of these rocks such as the direct “canister” method have significant flaws, which results in some formations doing far better than expected while others struggle to pay-out. A framework is set for using of strain properties of core samples for estimating the gas-in-place of these source rock-reservoirs.

The matrix swelling behavior of shale samples, induced by the exposure to a high pressure adsorbant gas, such as methane or carbon dioxide can be expressed in terms of volumetric strain can be coupled with the excess sorption mass. A laboratory setup is proposed for core-scale testing under varying pressures enabling calculation of the axial and radial strains created by the swelling experimentally. The volumetric strain variation with gas pressure is similar to that of the Langmuir adsorption model.

This work can bear substantial future impact in the area of formation evaluation, as well as sweet-spot identification for drilling. The described indirect “manometric” method bears some strong advantages over the canister method, albeit exhibiting a higher complexity with a number of corrections necessary for adapting the test results prior to their use in field development. A stepping stone is established for further research into the development of reliable, practical methods for determining the maximum adsorbed gas capacity of shale gas reservoirs, hence providing insight on their optimal exploitation.

Measuring the Sorbed Gas Capacity of Organic-Rich Shales

Direct “Canister” Method. Current industry practices for determining the sorption capacity of these shale rocks mainly involve the direct “canister” desorption method (ASTM D7569/D7569M), which is used for determining the gas content capacity of microporous source rocks (shales). Developed for coals by Bertrand et al. (1970) originally for coals, a core is placed in a canister and capped immediately after being tripped-out of the borehole. The gas desorbed (expelled) remains trapped in the canister, raising its internal pressure. Once the canister pressure stabilizes, it is opened against a tube usually filled with water, displacing the free gas. The displacement level corresponds to the desorbed gas amount.

Nevertheless, several flaws associated with this testing method decrease the reliability of the acquired results. Gas desorbed during the coring process (“lost gas”) is not reflected in results obtained using the canister method. Furthermore, significant time-duration is required for sufficient gas to be desorbed from low-permeability, organic-rich shale samples. This time-duration is typically in the order of weeks, discouraging the use of this technique when quick decision-making is needed.

Indirect “Manometric” Method. The swelling behavior of shale samples, induced when exposed to a high-pressure adsorbate gas (e.g. methane/CH₄ or carbon dioxide/CO₂), can be quantified in terms of volumetric strain which corresponds to the effective adsorbed mass. The volumetric strain variation with the gas pressure is similar to that predicted by the Langmuir adsorption model, which is used in the direct method. This provides a platform for the use of laboratory strain measurements in organic-rich shale core samples for estimating the gas storage capacity of these source rock-reservoirs.

Proposed Core Testing Method

Experimental Setup. The setup (Figure 1) consists of a stainless-steel sample cell, where the sample core is placed, with a cantilever beam with attached strain gages placed around it. Moreover, four valves (two triple-port and two double-port), a high precision pressure transducer, two positive-displacement pumps, which enable steady-state pressurization, and a vacuum-pump. A reference cell is separately
attached to the sample cell for measuring the porosity of the core sample via helium. The experiments need to be isothermal, so a thermocouple recording the temperature is placed in the sample cell.

![Diagram](image)

**Figure 1 Proposed experimental setup.** A cantilever beam (orange) with strain-sensitive gages (yellow) is shown around the core inside the sample cell. This schematic does not include the reference cell used for measuring the porosity of the shale core prior to the sorption test using helium (He) gas.

**Laboratory Testing Procedure.** The independent variable controlled by the operator of these tests is the gas pressure and the primary output of interest is the strain value recorded by the gages on the cantilever beam (arc placed axially or radially around the core). The testing procedure below is to be followed:

i. Synchronize all data acquisition and control apparatus.
ii. Place the core sample in the sample cell and close the cap head.
iii. Open the valve of the He bottle and let the sample cell to fill with the He until the pressure stabilizes. Record the pressure.
iv. Open the valve connecting the sample cell to the reference cell and record the new stabilized pressure. It must be lower than the pressure recorded at Step (iii).
v. Bleed out the pressure, open the sample cell and take the core sample out.
vi. Attach the cantilever beam axially on the sample (along height) and place it back into the cell.
vii. Open the valve of the CH₄/CO₂ bottle to a pre-determined starting pressure and let the gas fill the sample cell (keep the valve on the line connecting the pressure and reference cells closed).
viii. Record the final stabilized displacement values detected by the gages for different pressures.
ix. Bleed out the remaining pressure, open the sample cell and rearrange the cantilever beam, this time radially along the core sample’s diameter.
x. Repeat Steps (vii) and (viii).

**Excess Sorption Calculation**

The sorption isotherms measured in the laboratory are referred to as “excess” sorption isotherms. This is defined as the incremental amount of gas adsorbed per unit pore volume compared to the amount of gas in pore volume in the absence of pore walls. The difference between the total adsorbant gas mass transferred into the sample cell, \( m_{\text{sor,total}} \) and the product of the He-measured volumetric storage...
capacity of the core’s pore volume, $V_{p,He}$ with the density of the adsorbate gas used (CH₄ or CO₂), $\rho_{g,ads}$ is equal to the excess sorbed mass denoted by $m_{sor,excess}$

$$m_{sor,excess} = m_{sor,total} - \rho_{g,ads}(p, T)V_{p,He}$$

where $\rho_{g,ads}$ is a function of pressure and temperature. Thus, a reliable equation of state (EOS) is needed for evaluating the appropriate $\rho_{g,ads}$ value in the $m_{sor,excess}$ calculation for every pressure reading (temperature being constant). For the purpose of coming up with robust predictive model, the Langmuir model is chosen for its simplicity and close fitting. An adapted, three-parameter version of the Langmuir model is fitted (Gasparik et al., 2014) to compensate for the higher pressures of the manometric sorption testing, compared to the lower (vapor) pressures the original Langmuir model was designed for

$$n_{excess}(T) = n_L \frac{p}{p + p_L(T)} \left[ 1 - \frac{\rho_{g,ads}(p, T)}{\rho_a} \right]$$

where $n_{excess}$ represents the excess sorbed amount in mmol/g at pressure $p$ and temperature $T$. $p_L$ is the “Langmuir pressure,” corresponding to the pressure at which half of the sorption sites on the pore walls are being occupied, $n_L$ is the maximum “Langmuir capacity” in mmol/g, also known as “Langmuir volume” denoting the adsorbed gas quantity fully occupying the “Langmuir monolayer” of density $\rho_a$ (Gasparik et al., 2014). Due to several intensive parameters controlling the sorption capacity in shales (total organic content, moisture, diagenesis, etc.) work is needed before reliable correlations are drawn.

**Matrix Swelling Quantification**

The swelling exhibited by organic-rich shales when exposed to a pressurized adsorbant gas can be quantifying using the radial and axial strains measured in the manometric tests. If transverse isotropy is assumed, where radially isotropic behavior is assumed for the shale core, $\epsilon_{vol}$ to can be evaluated by

$$\epsilon_{vol} = 2\epsilon_{rr} + \epsilon_{zz}$$

where, $\epsilon_{rr}$ and $\epsilon_{zz}$ are respectively the shale core’s radial and axial strains. This sorption-induced matrix swelling of shale is competing with poroelastic compressional forces induced by in-situ stresses via

$$\epsilon_{sor} = \epsilon_{vol} + \epsilon_p$$

where $\epsilon_{sor}$ is the proportion of the strain induced by adsorption and $\epsilon_p$ is the strain induced by poroelasticity. The value of $\epsilon_p$ can be determined by running the same core test procedure using a non-adsorbant gas, such as He. The fundamental idea behind Hol et al.’s (2012) model for net volumetric strain versus CO₂ content in coals can be used for shales and CH₄ gas as expressed by Chen et al. (2015),

$$\epsilon_{sor} = \left( \frac{p}{p + p_L} \right) \epsilon_m$$

where $\epsilon_m$ is the matrix strain of the core sample. The manometric method allows testing with either increasing or decreasing pressures, eliminating the need for modeling adsorption/desorption hysteresis.

**Application Evidence**

Strain data from tests published in the literature was used to derive correlations describing the relationships between adsorbate gas pressure, adsorption-induced matrix swelling, and CH₄ sorption capacity for organic-rich shales. Three samples are considered; two organic shales from Chen et al. (2015) and an activated carbon sample from Heller and Zoback (2014). It has to be noted that many studies mentioned increased difficulty in getting reliable adsorption-induced matrix swelling strain
measurements from shale samples. The volumetric strain seems to exhibit Langmuir-like behavior with pressure variation. This indicates that matrix swelling-induced volumetric strain can be correlated proportionally to adsorption volume as shown on Figure 2a. All experimental data sets indicate strong positive correlations between matrix swelling and sorbed volume (Figure 2b).

Figure 2 Left: Plot showing a logarithmic relationships between adsorption-induced matrix swelling and gas pressure for experimental results published in the literature. Right: Plot showing logarithmic relationships between the absolute sorption capacity and matrix swelling for the same data as in (a).

Conclusion

- The indirect “manometric” method enables sorbed gas (adsorption or desorption) capacity estimations via the coupling with strains induced from the matrix swelling at different pressures.
- The three-parameter Langmuir (monolayer) excess sorption equation presents a good fit of excess sorption isotherm data in organic-rich shales, while volumetric strains corrected for the strain caused by the poroelasticity effect are used to quantify matrix swelling.
- Even though the indirect manometric method eliminates some of the flaws of the direct/“canister” desorption method for sorption capacity estimations in shales, it exhibits a lot of complexities and further work is necessary to make it an industry standard.

References


