

Shale Petrophysics: Electrical, Dielectric and NMR Methods to Characterize Mudrocks and Discover Relationships to Mechanical Properties and Hydrocarbon Affinity

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INTRODUCTION

With the rise of unconventional gas plays in North America and elsewhere, shales have emerged from petrophysical obscurity to being mainstream targets for formation evaluation. Even so, breaking out of conventional thinking about how best to characterize shale resources is still a challenge that has only partly been met.

Petrophysical work-flows for shale characterization have been developed before, but mainly to assess geomechanical and seal properties rather than to estimate hydrocarbons in place and production characteristics. As major shale gas plays have developed from concept to maturity, it has been found that one size most certainly does not fit all when it comes to the type of down-hole and laboratory petrophysics that proves to be beneficial and cost effective. Some shale gas plays can be assessed effectively using petrophysical methods that are a well-crafted version of those applied to conventional reservoirs. Other shales have only given up their secrets after a period of trial and error, and some break-through petrophysical thinking. In this paper we look at some of the approaches to shale characterization that have been developed at CSIRO over the past 5 years, and assess their applicability to shale gas reservoir characterization.

PETROPHYSICAL CHALLENGES OF SHALE RESERVOIRS

The objectives when characterizing a shale gas resource are quite different from those in conventional formation evaluation (Table 1). While understanding the matrix of a conventional reservoir is important in order to work out its porosity and mechanical properties, most of the issues of quantification of reserves and assessment of the performance of the reservoir depend on the pore structure and the properties of the free fluids. On the other hand, shale reservoirs typically present with very low values of porosity, and rather than “pore fluids” the main interest is in organic content, its maturity, and the amount of gas that can be moved as the pressure varies. Flow properties of the formation as a whole are dominated by fractures, and the economics of a well by how fractures can be created and/or enlarged and linked up. This makes ease of fracturing (strength, brittleness, stress anisotropy) a primary concern. To understand matrix flow in shales, rather than thinking of millidarcies and capillary pressures in the psi range, one has to account for permeability as low as nanodarcies, and the flow may be predominantly by diffusion, with capillarity and osmotic effects being important for the retention of water and the interaction of the formation with different types of fracture fluids.

Table. 1. Formation Evaluation Objectives for conventional vs. shale gas reservoirs (not exhaustive).

Conventional clastic reservoir	Shale gas reservoir
1. Matrix properties: mineralogy and grain density	1. Matrix mineralogy: clays, non-clays, others
2. Porosity, total and "effective"	2. Organic content and maturity
3. Clays and their CEC for shaly sand resistivity analysis	3. Fractures: density, orientation, connectivity
4. Hydrocarbon saturation (often based on resistivity)	4. Matrix porosity: total or effective – definitions?
5. Permeability (generally dominated by matrix permeability, flow obeys Darcy's law)	5. Adsorbed gas content and desorption isotherms; gas in place.
6. Capillarity: pore size, wettability	6. Presence and mobility of water
7. Relative permeability	7. Matrix gas permeability and gas diffusivity: flow does not obey Darcy's law
8. Geomechanical properties for well completion, sanding prediction	8. Geomechanical properties: response to fracturing
9. Formation damage sensitivity	9. In situ stresses
	10. Compatibility / sensitivity to potential fracturing fluids

LABORATORY ANALYSIS: CSIRO'S WORKFLOWS FOR SHALE SAMPLE CHARACTERIZATION

Clennell et al. (2006) described a workflow to determine non-destructively the physical properties of mudrock core material before geomechanical testing was carried out. Our workflow integrates x-ray CT scanning at the whole-core and plug scales, low-field nuclear magnetic resonance (NMR) spectroscopy and the measurement of low and high frequency electrical properties. These methods have now been further refined, using improved laboratory instrumentation, sample handling procedures and data analysis techniques.

Core preservation and handling. Our research at CSIRO requires that we have intact shale material to work with. Careful attention to handling and storage is a pre-requisite for good characterization, and the requires for shales are more stringent than for most conventional reservoirs, due to the damage that occurs if drying and air-entry takes place (this is capillary damage plus possible salt damage/damage by unbalanced osmotic forces). Shale preservation operates on a number of levels from pressure core samples that retain original fluids including fugitive gas, through to core that is substantially damaged by drying, oxidation and so on. For our purposes, we try to ensure that we have material that retains all of its original pore water: otherwise we assume that it is likely to have suffered some degradation and treat the material as we would cuttings (i.e. only use it to study mineralogy and other properties not affected by capillary/salt damage). This requires sealing at the wellsite. We recommend the use of inert and low volatility paraffinic oils to store shale samples, as some process oils contain chemical constituents that could affect wettability, or even permeate into the samples if they have surfactant properties. The pore fluid used in testing needs to be matched to the formation water salinity if possible. We conduct a screening process with small fragments to try and determine this salinity and also to assess the range of salinities that the samples can equilibrate with in tests without sustaining hydration or slaking damage.

X-ray computed tomography and other microscale imaging. The first step in our analysis is the screening of whole-core sections received using a medical type x-ray CT scanner (Fig. 1). Top-down projection images are combined with transverse slices that penetrate the core liner. An obvious requirement is that the core liner be made from x-ray transparent material, such as PVC, fiberglass or aluminum. From the screening images (resolution is approximately 1 mm per pixel), orientation of bedding is determined, and larger fractures and inhomogeneities can be detected. The plugging program is planned based on these images. Often we wish to determine several properties from limited core material, so there is a tradeoff between what we can achieve non-destructively. At the greatest premium are "geomechanics" plugs, which must be perfectly shaped and oriented, and if possible have a 2:1 length to diameter ratio. These plugs are used for strength testing, during which small strain elastic properties and anisotropic P-wave and S-wave velocities are measured with a 5-axis

combination. The “geomechanical” plug format is also suitable for 4-electrode resistivity measurement or permeability testing, though the minimum length for these tests can be much shorter.

If the diameter of whole core is large enough, and we have sufficient intact material, it may be possible to obtain both bedding parallel “horizontal” plugs and bedding normal “vertical plugs”. While in conventional core analysis on a limestone or sandstone reservoir, one would be looking primarily for anisotropy in permeability, for shales understanding anisotropy is important for the geomechanical and seismic point of view, and to understand electrical anisotropy effects in logs. We routinely scan every core plug at the maximum resolution of the medical scanner (0.3 mm in x-y plane, 1 mm in z plane), with typical two orthogonal longitudinal scans and 5 transverse scans. This has proven invaluable when interpreting geomechanical and ultrasonic measurements, and also for assessing electrical and dielectric properties, when we often work with thin slices cut from the end of a “geomechanics” or “resistivity” plug.

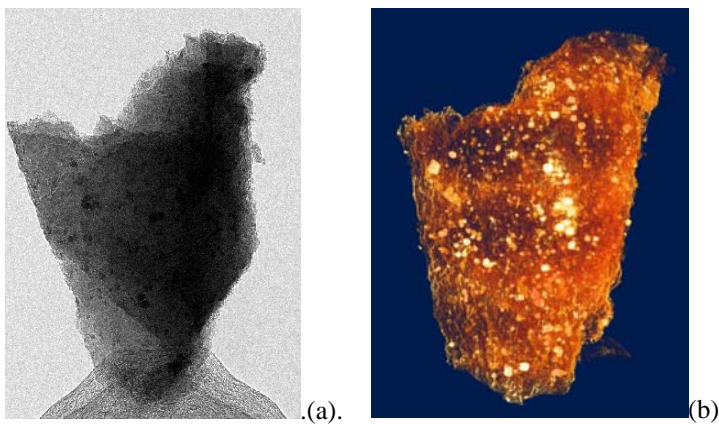


Fig.1 High resolution x-ray images of shale obtained with x-ray microscope system at CSIRO. The spatial resolution is better than 1 micron. (a) projection image as acquired (one of thousands obtained during stage rotation that build into tomogram). (b) rendered image showing general clay fabric alignment and with numerous pyrite microframboids (bright patches). Images: Sherry Mayo, CSIRO.

In the past three years, we have combined medical resolution x-ray imaging with high resolution x-ray microtomography conducted in-house at CSIRO using two conventional-source instruments, and also at synchrotron-source facilities. With ~1 micron pixel resolution one can image directly the organization of larger particles and structures in the shale (Fig. 1). Fine clay minerals (individual platelets can be ten times or more smaller) are not however resolved, meaning that some types of mudrocks appear featureless. While so-called nano-tomography x-ray imagers can improve on this resolution by perhaps an order of magnitude, the real breakthrough for understanding shale ultrastructure is proving to be focused ion beam/combined with field emission imaging in the scanning electron microscopy.

Nuclear Magnetic Resonance. In the last few years, the uptake of NMR in unconventional reservoirs (and for applications other than formation evaluation) has been notable. Back in 2006, we reviewed the situation with NMR applied to mudrocks and shales. A main finding then, and this is borne out by many further examples, is that the transverse relaxation time (T_2) response of shales is generally rather boring: a single peak at T_2 between ~0.5- 3 ms whose position and amplitude vary according to the general type of shale and its total porosity, respectively. If other peaks are present at longer T_2 , they indicate fluid-filled fractures or silty patches in the shale where larger pores are preserved. Some new types of multi-frequency, programmable NMR tools have been introduced in recent years. These could have some application to shale reservoirs, but in the main fancy pulse sequences are less useful than the ability to pick up total porosity by measuring down to very short T_2 , with adequate signal to noise. NMR logging has proven to be invaluable in some shale plays, while in others it has not proven to be

cost effective. In the laboratory, the limitations of permanent-magnets and inhomogeneous magnetic fields inherent to downhole tools can be sidestepped, and high-field NMR techniques can be usefully employed. Working at high magnetic fields enables enormous spectral resolution and signal to noise, and time resolution is much improved. This means detecting and differentiating fluids that are adsorbed or reside in very small pores. We have used a range of high field NMR methods to investigate wettability, hydration and fluid diffusivity in shales and clays. spectroscopy for both proton and ^{13}C species. Future research avenues include organic matter typing and investigation of wetting and non-wetting fracture fluids interaction with time-resolved NMR.

Electrical and Dielectric Spectroscopy. CSIRO has developed a range of methods for characterizing both preserved shales and shale cuttings using broad-band electrical/dielectric spectroscopy. The variation of resistivity with frequency from ~ 1 Hz to ~ 1 MHz depends on clay content, pore fluid content and its salinity. The most useful application of laboratory impedance spectroscopy at these lower frequencies is to calibrate downhole log data in shale sections and better understand what is leading to the resistivity variations encountered *in situ*. At high frequency (around 1 GHz) dielectric measurement offers a way to obtain water content, without knowledge of formation water salinity. In the GHz range, the permittivity can essentially be described by a mixing law relationship that depends only on the ratio of water (relative permittivity ~ 80) and mineral grains (relative permittivity ~ 4.5). The shape of the dielectric dispersion curve as frequency decreases to a few tens of megahertz is largely dictated by the clay mineral surface area and the amount of surface bound charges (Myers 1991). Therefore, with measurements over a range of frequencies (Fig. 2) it becomes possible to improve the definition of characteristics such as clay content and clay type which may correlate with mechanical properties, permeability and physicochemical response to fracturing fluids. New dielectric tools operating at multiple frequencies (e.g. Hizem et al. 2008), although not developed with shale reservoirs in mind, may prove very useful for characterizing these complex reservoirs.

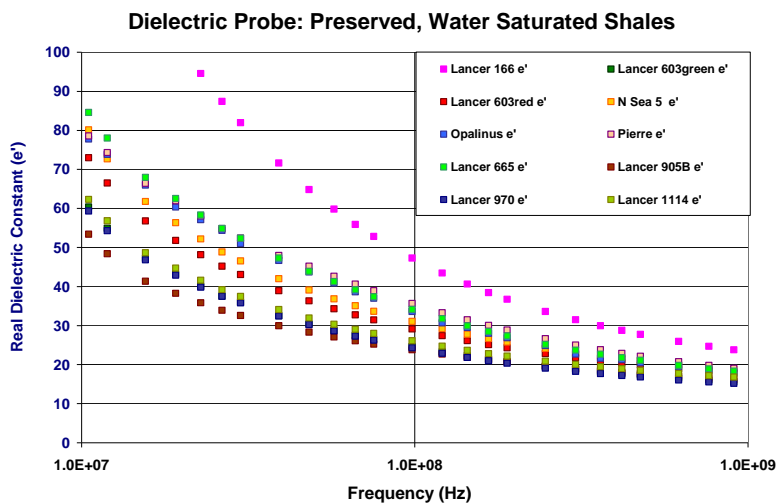


Fig. 2. Dielectric spectra of ten preserved shale samples from around the world. Most shales exhibit similar shaped dielectric curves but the amplitude varies from shale to shale. At high frequency, the relative permittivity (dielectric constant) is dictated by water content, while the degree of dispersion in permittivity is due to the amount of surface polarization, and so the dielectric constant at around 10-30 MHz correlates with the amount of surface bound charges on clay particles (i.e., CEC).

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