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## Porosity and pore size distribution of deeply-buried fine-grained rocks: Influence of diagenetic and metamorphic processes on shale reservoir quality and exploration

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#### A R T I C L E I N F O

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#### ABSTRACT

Significant exploration risks are associated with the pursuit of deeply-buried shale gas reservoirs due to pore volume reduction and changes in pore size distribution. These changes in pore character result in decreases in gas in place and permeability. A suite of shale, low grade, pelitic metamorphic and a granite outcrop samples from various location in North America have been selected to span the later stages of diagenetic, epimetamorphic (epizone) and anchimetamorphic (anchizone) processes to evaluate the changes in the inorganic pore volumes and size distributions. Diagenetic/metamorphic ranking of samples were determined by the illite crystallinity method. Pore volumes reduce with increasing maturity/metamorphic grade. The loss of mesopore volume (2-50 nm) with increasing maturity is the cause of the reduction in porosity. The reduction in mesopore volume is interpreted to be due to the authigenic recrystallization and growth of the clay minerals. As maturity/metamorphic grade increases there is a relative increase in the macropore (>50 nm) and micropore (<2 nm) size fractions. The increase in micropore volumes may be attributed to the development of secondary porosity within the kerogen. At higher maturity/metamorphic grade (i.e., illite crystallinity < 0.2  $\Delta 2\theta$ ) porosity values range between 0.9% and 3.6% indicating that fracture porosity is not the only mechanism of gas storage in deeply buried shale (and pelitic metamorphic rocks) reservoirs. Matrix porosities in these higher maturity/metamorphic samples are comparable to matrix porosities of the Horn River shales of British Columbia and other shale reservoirs. Similar to the Horn River and Doig-Montney shales, the reduction in mesopore volumes may reduce the matrix permeability of these rocks and fracture stimulation will be an integral component of the completions program to access hydrocarbons.

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#### Introduction

As global exploration of shale gas intensifies there is significant risk associated with the pursuit of shale gas resources that are deeply buried (i.e., Haynesville Formation, Mississippi, USA; Mainland Resource Inc., 2009) due to diagenetic and metamorphic processes that reduce porosity, alter pore size distributions and reduce permeability. The decrease in porosity with increasing maturity within sandstones and shales is well documented within the oil and gas windows (i.e., vitrinite reflectance –  $R_o$  0.5–2.0%, Hunt, 1995; Schimmelmann et al., 2006). The change in pore characteristics results in lower gas capacity and hence lower permeabilities (Biryukov and Kuchuk, 2012). Porosity, as described in this study, refers to the total matrix porosity (i.e.,  $S_w = 0$ ) and excludes fracture

\* Corresponding author. *E-mail address*: garethchalmers@gmail.com (G.R.L. Chalmers). porosity. Porosity is a significant control on gas-in-place estimations for shale gas resources as illustrated for the regional evaluation of the Jurassic Gordondale Member (Ross and Bustin, 2007), Devonian Horn River strata (Ross and Bustin, 2007), Cretaceous Buckinghorse Formation (Chalmers and Bustin, 2008a) and the Cretaceous Shaftesbury Formation (Chalmers and Bustin, 2012a) in northeastern British Columbia. A positive relationship exists between porosity and total gas (free + sorbed gas) capacity of a shale reservoir (Chalmers and Bustin, 2008a; Appendix F). The free gas component increases relative to the sorbed gas component in deeply buried shales because of reservoir temperature increases with depth of burial. Sorbed gas is an exothermic reaction and an increase in temperature will reduce the volume of sorbed gas and increase the free gas content of a reservoir (Ross and Bustin, 2008) and majority of the free gas content will reside in the inorganic pore system. For example, areas that experience high geothermal gradients (i.e., Horn River Basin, British Columbia),

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the free gas component is a significant portion of gas-in-place (GIP) estimates for gas shale resources (Ross and Bustin, 2008) and any loss of porosity associated with increased burial and associated chemical and mechanical alteration (Foscolos, 1990; Kisch, 1991; Kubler and Jaboyedoff, 2000) may significantly reduce the gasin-place estimation of the reservoir. Requirements for economic production (i.e., permeability, drainage volumes, fracability) and gas-in-place estimations are dependent on effective porosity and the pore size distribution (PSD) of the reservoir and interconnectivity of the pore system. The evaluation of the PSD is fundamental in the understanding of a shale gas reservoir capacity and permeability as the PSD is a control on matrix permeability of shale reservoirs. Studies have shown that higher matrix permeabilities in both the Horn River (Chalmers et al., 2012b) and Triassic Doig-Montney (Chalmers and Bustin, 2012b) shales exhibit a balanced ratio between micro-, meso- and macroporosity compared to lower permeability shales. To adequately describe the PSD of fine-grained reservoirs, the IUPAC (1994) classification system which was developed for porous solids has been adopted for this study, as the majority of pore throats in shale reservoir pore systems are within the nanometre scale (Chalmers et al., 2012a; Milliken et al., 2013). In the IUPAC classification micropores are <2 nm, mesopores between 2 and 50 nm and macropores are >50 nm (Rouquerol et al., 1994). Shale pore classification system by electron microcopy (i.e., Loucks et al., 2012) do not include pores that are below 5-8 nm which are a significant portion of the total porosity of a shale reservoir (Chalmers et al., 2012a; Milliken et al., 2013; Mastalerz et al., 2013). Hence, electron microscopy does not capture changes that occur within the nanometre scale since all pores are described as "nanopores". A study of pore volumes using electron microscopy indicates that this technique is unable to quantify up to 98% of the total porosity of shale reservoir (Milliken et al., 2013). Gas adsorption and mercury porosimetry analyses and description of the pore size distribution using the IUPAC classification system have been successfully used to identify changes in PSD with parameters like maturity (Chalmers and Bustin, 2012a; Mastalerz et al., 2013), permeability (Chalmers et al., 2012b) and with TOC content (Chalmers and Bustin, 2008b; Ross and Bustin, 2007, 2008; Tian et al., 2013) and have been adopted for this investigation.

This study will evaluate the relationship between maturity, porosity and PSD to include maturities greater than 2.0%  $R_o$  to the point of incipient metamorphism (epimetamorphism; Kisch, 1991; Kubler and Jaboyedoff, 2000) as past similar studies have limited their investigation to maturities below 2.0%  $R_o$ . The inorganic pore volume and PSD of a suite of shales and pelitic metamorphic rocks are investigated to gain an understanding of the effect of diagenesis and metamorphic processes have on potential shale gas reservoirs within the dry thermogenic gas window ( $R_o > 2\%$ ; Schimmelmann et al., 2006) and into the anchizone and epizone. The organic pore volume is not considered in this study as samples are overmature resulting in kerogen that is inert residual carbon and no changes to the organic porosity are expected.

#### Methodology

We have analysed a suite of samples from various locations in North America (Fig. 1; Table 1) that span a range of diagenetic to metamorphic processes to understand the changes in the PSD and porosity that are associated with the inorganic content (TOC lean rocks; <1.5 wt%). Majority of samples in this study do not represent a producing reservoir as no producing reservoirs have experienced maturities above 2.0%  $R_o$ , therefore these samples are being used in the study to investigate how the inorganic pore system changes with diagenetic and metamorphic processes. Matrix permeability will also be affected by these changes but is not in the scope of this study. A granite sample is included in the suite of rocks to evaluate the porosity and PSD as fractured igneous rocks, like metamorphic rocks are also considered to have no matrix porosity (Biryukov and Kuchuk, 2012) with hydrocarbon production considered to be exclusively from fracture networks (i.e., Yemen; Legrand et al., 2011). The criteria for selection are based on: (1) pelitic origin; (2) TOC lean; and (3) span diagenetic to metamorphic processes.

#### X-ray diffraction and Rietveld analysis

Seventeen crushed samples (<250  $\mu$ m) were mixed with ethanol, hand ground in a mortar and pestle and then smear mounted on glass slides for X-ray diffraction analysis. A normal-focus Cobalt X-ray tube was used on a Siemens<sup>®</sup> Diffraktometer D5000 at 40 kV and 40 mA. The mineral composition was semi-quantified by Rietveld analysis (Rietveld, 1967) using Bruker AXS Topas<sup>®</sup> V3.0 software.

#### Illite crystallinity

A number of well-established analytical techniques measure the level of diagenesis and metamorphism of sedimentary rocks. The degree of diagenesis/metamorphism is measured on the organic component (i.e., vitrinite reflectance,  $T_{max}$ , proximate analysis; Diessel, 1992; Taylor et al., 1998) or on the mineral matter (i.e., illite crystallinity; Kubler, 1968; Kisch, 1974, 1980, 1991; Kubler and Jaboyedoff, 2000). Illite crystallinity is a reliable method to determine maturity or metamorphic grade for sedimentary and pelitic low grade metamorphic rocks in late stages of diagenesis and incipient metamorphism (Foscolos, 1990; Kisch, 1991; Kubler and Jaboyedoff, 2000). Metamorphic zone boundaries are defined by changes in mineral assemblages and begin at the end of dry gas generation which is termed the anchizone (Kisch, 1991). The greenschist facies minerals (i.e., chloritoids) define rocks within the epizone and the transformation of dickite to pyrophyllite (Kubler, 1967). Illite crystallinity is a reliable measure of diagenetic and metamorphic processes as the growth and recrystallization of illite, in particular, the increase in the thickness of the illite crystallites occurs with increasing metamorphic grade in sedimentary rocks (Abad, 2007; Meunier, 2005). This study has utilised illite crystallinity method (Kubler, 1968) as a measure of maturity for this suite of samples as this is most reliable method for overmature TOC-lean rocks. Illite crystallinity is favoured for fine-grained sedimentary rock that may lack the size and quantity of dispersed organic matter to provide valid vitrinite reflectance measurements and the S2 is too depleted for  $T_{max}$  values from programmed pyrolysis (Peters, 1986). S2 peak is the second peak in rock-eval pyrolysis that represents the hydrocarbon generative potential of the sample and this peak is used to determine the  $T_{max}$ and maturity of the rock (Espitalie et al., 1985). Samples used in this study are TOC lean (<1.5 wt%) and vitrinite and rock-eval pyrolysis cannot provide valid results to measure their maturity. The thickness of illite crystallites is measured by the peakedness of the first basal reflection (001) peak at X-ray diffraction spacing of 10 angstroms and reported as a peak width in degrees (Blenkinsop, 1988; Kisch, 1991; Abad, 2007). The boundary between diagenesis and the initiation of metamorphism (anchizone) is considered at  $0.42^{\circ} \Delta 2\theta$  illite peak and the boundary between anchizone and epizone at  $0.25^{\circ} \Delta 2\theta$  illite peak (Kisch, 1991).

Protocols that have been developed to obtain consistent results between laboratories (Kisch, 1991) have been implemented in this study. Samples were disintegrated using mortar and pestle and agitated by sonic bath in a distilled water and deflocculant Download English Version:

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