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Research paper

Evolution of porosity and pore geometry in the Permian Whitehill Formation of South Africa – A FE-SEM image analysis study

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A B S T R A C T

Since the advent of technologically efficient exploitation of economic hydrocarbon reservoirs in shales, increasingly more research has been devoted to identifying and characterizing pore systems within shales. However, only a handful of these studies focused on the development of porosity in thermally mature unconventional reservoirs. In this study, the evolution of porosity and pore geometry in the Permian Whitehill Formation is addressed with the aid of ultrathin sections (2 × 3 cm, 10–20 μm thick) and field-emission scanning electron microscopy (FE-SEM) on samples with mean random vitrinite reflectance values ranging from 1.03 to 4.07 %Ro. We document a strong positive covariation of porosity and total organic carbon content (TOC) in all localities. However for samples with vitrinite reflectance values greater than 2.88 %Ro porosity per unit TOC decreased by over 25% relative to samples with lower thermal maturities. The positive covariation of thermal maturity and total porosity recorded here is unsurprising and have been documented previously in many gas shales. However, the dramatic decrease in porosity restricted to samples from localities that experienced advanced maturation (Ro > 2.88%) is viewed as an evidence that porosity decrease is directly related to late thermal decarboxylation of organic matter. This is supported by the presence of pores and micro-fractures infilled by fibrous grains, including carbonates, clays, silicates, and phosphates, and residual fluid inclusions. These grains were likely generated from re-precipitation of framework grains previously dissolved by organic acids (carboxylic, phenolic) that were generated during thermochemical decarboxylation of the OM. Our findings do not only fill important gaps in the understanding of organic pore development, including processes that create, preserve, and destroy porosity, the porosities described here are also key to gas transfer from shale matrix to induced fractures during fracture stimulation programs.

1. Introduction

Traditionally, it is believed that hydrocarbons (oil and gas) are generated within organic matter-rich shales (i.e., source rocks) and then migrate into more porous units (i.e., reservoir rocks), such as sandstone, conglomerates or limestone and other naturally fractured rocks types (e.g., Demaison and Huizinga, 1991; Klemme and Ulmisheck, 1991; Magoon and Dow, 1994). However, appreciable hydrocarbons formed in some source rocks are retained after their formation, especially where the source rocks are highly impermeable or “tight”, resulting in an unconventional situation where source rocks also serve as reservoirs for hydrocarbons (e.g., US-NPC, 2007; Boyer et al., 2011; Wright et al., 2015). A gas shale is a peculiar type of unconventional gas-hosting rock in which the gas content can only be extracted by fracture permeability, either via artificial hydraulic fracturing (fracking) or via natural fractures (e.g., Jarvie et al., 2007; US-EIA, 2011). The cost-effective exploitation of economic hydrocarbon reservoirs in shale successions has resulted in a significant increase in funding of investigations on pore structures in shales (e.g., Loucks et al., 2009; Bernard et al., 2013; Milliken et al., 2013). Whether a shale can be suitable for gas production or not depends on the content and nature of its organic matter, porosity and ductility/brittleness (Jarvie et al., 2007; Passey et al., 2010). Characterizing the porosity in shales has been a rather difficult task, largely because of the small pore sizes of these rocks, much smaller than those in conventional reservoirs (Schieber and Zimmerle, 1998; Loucks et al., 2012; Camp et al., 2013). In addition, shales are dramatically heterogeneous with multi-level (from macro-down to nanoscale) variations in their structures and compositions (e.g., Curtis et al., 2012; Lazar et al., 2015). This means that each shale play is unique in several geological aspects and cannot be used with ease to evaluate another play, not even those in the same shale unit.

In addition, from a scientific standpoint, porosity is a multifaceted

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subject and is approached differently among disciplines. For instance, petroleum engineers and reservoir scientists quantify porosity using petrophysical (bulk) methods, such as helium porosimetry (e.g., Cui et al., 2004; Ross and Bustin, 2007; Mastalerz et al., 2013; Bahadur et al., 2015). While bulk characterization techniques effectively provide quantitation of pore throat dimensions (e.g., Dewhurst et al., 1998), they do not measure total porosity, which is critical for estimating the capacity of the reservoir. On the other hand, shale petrographers approach porosity development in shale successions by providing both quantitative and visual qualitative analyses of porosity by direct petrographic examinations of pores through the application of scanning electron microscopy and its auxiliary technologies (e.g., Loucks et al., 2009; Keller et al., 2011; Klaver et al., 2012; Camp et al., 2013; Milliken et al., 2013; Löhr et al., 2015). Direct observation of pores has a unique advantage of distinguishing pores within organic particles from those within the inorganic matrix of the shale sample. Although recent studies (e.g., Löhr et al., 2015; Sommacal et al., 2016) have shown that pores within OM have greater affinity for hydrocarbon than those within mineral grains, distinguishing OM-hosted- from mineral-hosted pores also allows porosity evolution to be related to factors closely associated with depositional, diagenetic, and catalytic processes (e.g., Curtis et al., 2012; Bernard et al., 2013; Schieber, 2013).

Pore networks within most gas shales are largely dominated by nanometer-size pores (e.g., Ross and Bustin, 2007; Curtis et al., 2012; Loucks et al., 2012; Milliken et al., 2013). IUPAC (1994) subdivided materials with nanometer-sized pores into three categories: macropores (> 50 nm), mesopores (2–50 nm), and micropores (< 2 nm). Characterizing nanometer-sized pores is challenging because conventional transmitted and reflected optical microscopy cannot image meso- and micropores due to the low power of magnification of these standard methods. While FE-SEM and its auxiliary technologies have the suitable resolution (Loucks et al., 2009; Curtis et al., 2012; Camp et al., 2013), this method is however not suitable for rock chips because of their irregular surface topography (Loucks et al., 2009; Keller et al., 2011; Schieber, 2013). Mechanically polished thin sections often contain artefacts such as abrasion marks and grinding debris (Schieber, 2013; Kaufhold et al., 2016), which can influence the detection of delicate features in SEM images. Ion-milling techniques, including those that involve the use of either a focused ion beam (FIB) or a broad ion-beam (BIB) of Ar + or Ga + to remove a small amount of material to generate an ultra-smooth surface (Loucks et al., 2009; Keller et al., 2011; Camp et al., 2013; Schieber, 2013), has greatly enhanced the understanding of shale features with SEM. However, ion-milling is limited due to the small field of view (about 40 × 30 μm) that can be imaged by SEM at sufficiently small pixel size at a time (Curtis et al., 2012; Kaufhold et al., 2016).

To date, the link between porosity and thermal maturity is inferred from studies that used samples with low thermal maturity range and where thermal maturity had been achieved through burial diagenesis (e.g., Cui et al., 2004; Ross and Bustin, 2007; Keller et al., 2011; Chalmers et al., 2012; Curtis et al., 2012; Bernard et al., 2013). Studies are rare on the development of porosity with increasing thermal maturity using samples with a wider range of thermal maturity and where thermal maturities were achieved through tectonic burial and thermal devolatilization during igneous intrusion. Samples from the Permian Whitehill Formation (WHF) in the main Karoo Basin of South Africa have thermal maturities that range between ~1.0 and > 4.0% Ro (Rowsell and De Swart, 1976; Cole and McLachlan, 1991). Works by Oelofsen (1981), Visser (1992) and Chukwuma and Bordy (2016) have shown that the WHF consists of subunits with remarkably uniform primary lithologic and sedimentologic characters across the Karoo Basin. In particular, the omnipresence of pyrites in both euhedral and framboidal forms, that appear to have formed due to the activity of sulfate-reducing bacteria at or near the depositional interface, is a strong evidence that uniform reducing (anoxic) conditions were persistent at all localities across the basin during the deposition of the lower two subunits (F1 and F2). Also, the 'consistency in the thickness of the biozones' (Oelofsen, 1981, p. 24) in the upper WHF subunits (F3, F4, F5) indicates that the depositional conditions were seemingly the same over the entire basin floor. There is, therefore, no evidence that depositional conditions varied significantly across the basin during the deposition of any of the five subunits of the WHF. This inference is important because it gives weight to the assumption that initially, each subunit of the WHF was identical in overall properties and that significant differences observed in abundance and distribution of porosity, particularly within organic macerals, are directly related to post-depositional (diagenetic and thermal maturation) processes, which were majorly controlled by the relative distance of each locality from the presumed heat source, the Cape Fold Belt (CFB). Rowsell and De Swart (1976) and Cole and McLachlan (1991) observed that the degree of thermal maturity (measured with reflectance of vitrinite, [%Ro]) show a progressive decrease from the southwest (heat source) to the northeast. In the study by the latter authors, samples from localities within about latitude 29 °S and more southerly were within late dry gas window (%Ro > 2.3) whereas those within latitude 31.5 °S and more northerly were within oil to the wet gas window (%Ro < 2.3).

The overall objectives of this study were to investigate the evolution of porosity and pore geometry with increasing thermal maturity and to assess the relationship between TOC and total porosity in reservoir shales. In order to realize these goals, visual qualitative and quantitative image analyses of the pore systems within the WHF were combined with suites of geochemical analyses. We examined high-resolution two-dimensional (2-D) FE-SEM images of ultrathin sections (2 × 3 cm, 10–20 μm thick) from samples taken from three major subunits (F1-F3; Chukwuma and Bordy, 2016) of the WHF across the Karoo Basin (Fig. 1). X-ray fluorescence spectroscopy, Rock-Eval pyrolysis, and elemental and stable isotope analyses provided data on the compositional geochemistry of the shale samples. The geology of the Karoo Basin and the WHF has been explained in detail in the literature [e.g., by SACS (1980), Cole and Basson (1991), Visser (1992), Catuneanu et al. (2005), Tankard et al. (2012), Geel et al. (2015)], and are not repeated here.

2. Samples, methods and analytical limitations

2.1. Samples

Samples used in this study come from 14 localities along the semi-continuous exposure belt of the WHF in the main Karoo Basin (Fig. 1A), representing a complete stratigraphic development of the WHF (Fig. 1B), including organic matter (ranging between < 1 and 16.5%), different burial depths and temperatures (with calculated thermal maturities ranging between < 1 and 4.6 %Ro) and thickness (ranges between < 10 to about 70 m). The sampling localities, including the type unit of the WHF (Cole and Basson, 1991), were targeted because they expose the same stratigraphic subunits of the shales but at different distances from the CFB (Rowsell and De Swart, 1976; Cole and McLachlan, 1991). The largely unweathered samples were taken with a STIHL E-Z Core Rock Drill fitted with a Pomeroy 40 × 2.5 cm core barrel. The samples are designated by abbreviations related to their localities, e.g., PAT for Prince Albert, LAG for Laingsburg, MAJ for Matjiesfontein, CAL for Calvinia, NUW for Nuwelande, LOE for Loeriesfontein, VAK for Vanwyksvlei, BTT for Britstown, STY for Strydenburg, HPT for Hopetown and CST for Christiana. The description of the lithology and sedimentary structures of the samples is provided by Chukwuma and Bordy (2016).

2.2. Whole-rock and organic carbon composition

The chemical composition of One-hundred-and-twenty (120) powdered samples was determined using standard X-ray Fluorescence (XRF) procedure (Injuk and Van Grieken, 1993; IAEA, 1997) in the