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Research paper

Microstructural characteristics of the Whitby Mudstone Formation (UK)

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ABSTRACT

When trying to improve gas productivity from unconventional sources a first aim is to understand gas storage and gas flow potential through the rock by investigating the microstructure, mineralogy and matrix porosity of unfractured shale. The porosity and mineralogy of the Mulgrave Shale member of the Whitby Mudstone Formation (UK) were characterized using a combination of microscopy, X-ray diffraction and gas adsorption methods on samples collected from outcrops. The Whitby Mudstone is an analogue for the Dutch Posidonia Shale which is a possible unconventional source for gas. The Mulgrave shale member of the Whitby Mudstone Formation can microstructurally be subdivided into a fossil rich (>15%) upper half and a sub-mm mineralogically laminated lower half. All clasts are embedded within a fine-grained matrix (all grains $< 2\ \mu m$) implying that any possible flow of gas will depend on the porosity and the pore network present within this matrix. The visible SEM porosity (pore diameter > 100 nm) is in the order of 0.5–2.5% and shows a non-connected pore network in 2D. Gas adsorption (N_2, Ar, He) porosity (pore diameters down to 2 nm) has been measured to be 0.3–7%. Overall more than 40% of the visible porosity is present within the matrix. Comparing the Whitby Mudstone Formation to other (producing) gas shales shows that the rock plots in the low porosity and high clay mineral content range, which could imply that Whitby Mudstone shales could be less favourable to mechanical fracturing than other gas shales. Estimated permeability indicates values in the micro-to nano-darcy range.

1. Introduction

Gas in shales is trapped in poorly connected pores and sorbed on the surfaces of particles present in the matrix of the shale host rock. The bulk permeability of shales is so low that gas production from such formations is simply not feasible without fracturing the rock by injecting fluids at high pressure. This creates a radiating network of pathways that penetrate the shale formation around the injection and/or production well. Fractures induced in this way improve the overall permeability of the formation, both directly and by connecting pre-existing natural fractures. However, while much progress has been made in developing fracturing methods, the permeability of the gas shale matrix present between fractures is so low that very close fracture spacing is often needed to achieve sufficiently rapid transport of gas from the matrix into the induced fractures (Curtis, 2002; Gale et al., 2007). Improvements are possible by increasing fracture density, but there is a limit and even the minimum achievable spacing may prove inadequate. Experience from the USA has taught us that petrophysical properties vary and a successful combination of petrophysical properties is hard to specify for gas shales, although almost all currently producing shale gas reservoirs are over mature, oil-prone source rocks; e.g.: Curtis, 2002; Jarvie et al., 2007.

In the Netherlands the lower Jurassic Toarcian Posidonia Shale Formation (PSF), the main hydrocarbon source rock in the North Sea, is considered as a possible gas shale (Herber and de Jager, 2010). The mean thickness of the formation in the subsurface of the Netherlands is 15–35 m, the average TOC value is 10% and the Hydrogen index (HI) is 800 (Herber and de Jager, 2010). The PSF is a generally dark grey, laminated and bituminous rock which represents peak transgression during a sea-level high stand and corresponds to an early Jurassic global oceanic anoxic event (e.g.: Trabucho-Alexandre et al., 2012). The PSF shales extend from the Yorkshire Basin (England; 'Whitby Mudstone Formation'), over the Lower Saxony Basin and the Southwest German Basin ('Posidonia Shale Formation') into the Paris Basin ('Schistes Carton') (e.g.:

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Jenkyns, 1985; Rullköter et al., 1988; Littke et al., 1991).

In order to try and improve gas productivity from the shale matrix it is necessary to find ways to better connect the in-situ pore network to the natural and mechanical induced fractures. A first step is then to determine the pore network characteristics and the porosity values of the rock. Since in the Netherlands the PSF does not outcrop, for this research time equivalent exposed PSF samples have been collected north of Whitby (UK). For microstructural investigations, porosity and mineralogy measurements we used; a combination of a Precision-Ion-Polishing-System (PIPS) and Scanning Electron Microscopy (SEM), light microscopy, gas adsorption (N_2, Ar, He), X-ray Diffraction, and X-ray Fluorescence.

2. Materials and methods

During the Jurassic the Cleveland Basin formed part of a system of shallow epeiric seas and small extensional tectonic basins linked to the North Sea Basin via the Sole Pit Basin (Ziegler, 1982; Powell, 2010). A Sinemurian–Aalenian paleogeographic map of the North Sea can be found in e.g.: Ziegler (1982), Littke et al. (1991), Knox et al. (1991), Hesselbo et al. (2000), Powell (2010). The organic-rich sediments deposited in Central and North Western Europe during the late Jurassic are known as the Mulgrave Shale (WMF), Schistes Carton (SC) and the Posidonia Shale (PSF) (e.g.: Jenkyns, 1985; Littke et al., 1991), where this depositional area was connected to the Tethyan ocean where black shales were deposited as well during the Jurassic (Littke et al., 1991). The Whitby Mudstones are similar to the fine-grained siliciclastic mudstone dominated successions deposited across North West Europe during the Lower Jurassic (Ghadeer and Macquaker, 2012) and consist predominantly of grey to dark grey mudstone and siltstone (Powell, 2010). Prior to exhumation the WMF has been curried to the early oil window (e.g.: Williams, 1986; Kemp et al., 2005), maximum burial depth of 2–4 km during the late Cretaceous (Hemmingway and Riddler, 1982; Williams, 1986; Green, 1989; Bray et al., 1992), and therefore these rocks could act as an useful analogue for shale reservoirs (Imber et al., 2014). Hydrogen indices in the Mulgrave Shale are high (Saelen et al., 1995), and some levels display unusually low $\delta^{13}C$ values (Jenkyns and Clayton, 1997; Kemp et al., 2005). The abundant framboidal pyrites in the section point towards anoxic and sulfidic conditions (Raiswell, 1982; Wignall and Newton, 1998). For an extensive summary on the geological history and geological setting of the Cleveland Basin and geological maps of the area around Whitby (UK) see e.g.: Powell (2010), Imber et al. (2014).

The WMF can be subdivided into the Alum Shale, the Mulgrave Shale and the Grey Shale. The Alum Shale Member, maximum thickness 37 m, consists of grey silty mudstones with bands of calcareous and siderite concretions (Powell, 2010). The Mulgrave Shale member is an oil-prone source rock, consisting of fissile, bituminous, dark grey mudstones with abundant ammonites (Powell, 2010), which is the lateral equivalent of the Posidonia Shale in Northern Europe (Leythaeuser et al., 1988; Rullköter et al., 1988; Littke et al., 1991; Powell, 2010; Ghadeer and Macquaker, 2012). The Mulgrave Shale is a circa 30 m high section with TOC values ranging between 1 and 15% and the highest Toc values (4–15%) can be found in the bottom 8 m of the section (Hesselbo et al., 2000; Powell, 2010; Ghadeer and Macquaker, 2011; Imber et al., 2014). The bottom 8 m of the Mulgrave Shale is subdivided in sections due to thick bands of calcareous concretions of varying sizes (top to bottom: Curling stones, Whale stones, Cannon balls) and a limestone band near the top of the section (e.g.: Raiswell, 1976; Powell, 2010; Ghadeer and Macquaker, 2011; Imber et al., 2014). Below the Mulgrave Shale you can find a bioturbated pale- to dark-grey mudstone with concretionary siderite and calcite cemented units with TOC values below 4% called the Gray Shale member (e.g.: Hesselbo et al., 2000; Powell, 2010). The transition from the Grey Shale to the Mulgrave Shale coincides with an increase of water depth in the basin (Saelen et al., 1996). Below the Whitby Mudstone one can find the Cleveland Iron Stone Formation. Ghadeer and Macquaker (2011) recognized six mudstone lithofacies types in the Cleveland Iron Stone Formation and the Whitby Mudstone Formation; 1. Sand- and clay bearing, silt-rich mudstones; 2. Silt bearing, clay-rich mudstones; 3. Clay-rich mudstones; 4. Clay-, calcareous nannoplankton-, organic carbon bearing mudstones; 5. Fine-grained muddy sandstones; and 6. Cement-rich mudstones, where lithofacies 3, 4 and 6 were encountered in the Mulgrave Shale member. Most siliciclastic clay-, silt- and sand-sized components are derived from detrital inputs to the basin (Ghadeer and Macquaker, 2011). In addition, some biogenic components are present like; bivalves, ammonoids, echinoderms, coccoliths, calcispheres and foraminifers in addition to organic carbon (e.g. Saelen et al., 1996; Wignall et al., 2005). The difference in temporal grain size and composition is for this formation linked to the balance of primary biogenic production relative to the dilution and length of the sediment transport path, which varied during deposition (Ghadeer and Macquaker, 2011).

WMF samples investigated originate from outcrops along the cliff coast north of Whitby (UK) near the villages Runswick Bay and Port Mulgrave. Samples were collected during fieldwork (May 2013, March 2014) using a geological hammer and chisel. All samples studied were collected within 5 km laterally and samples were taken throughout the circa 8 m thick Mulgrave Shale member of the WMF, the organic-rich section. A method of plastic bags and cling film has been used to store and transport the samples; hence samples became air-dried during sample transportation/storage and before sample preparation. Hand specimens taken in the field were numbered chronologically based on when the sample was taken. These numbers are used here to distinguish between the different hand specimens. One hand specimen though could have been used to be studied with a number of different techniques (Fig. 1). One can distinguish between the different subsamples by the letter added to the initial number; T for thin sections investigated with light microscopy, P for PIPS-SEM samples investigated with Scanning Electron Microscopy, D for samples used for X-ray Diffraction, F for samples used for X-ray Fluorescence, H for He gas adsorption samples and B for N_2 and Ar gas adsorption samples. Different subsamples investigated with the same method are indicated with small letters (a–z) after the first capital letter indicating the method.

2.1. Optical microscopy

Polished thin sections were prepared (ca. 2.5 × 4 cm) using conventional methods for 5 samples collected throughout the Mulgrave Shale member, all spaced 1–2 m apart vertically. Samples used for thin sections originate from the same sample blocks as the PIPS-SEM samples (top to bottom of the section: 4, 6, 15, 1, 23) and can be directly linked to the PIPS-SEM samples based on characteristic beds present in the hand specimen (Fig. 2). These samples were investigated using plain- and cross-polarized light under a Leica microscope under transmitted light; digital images were taken with a pixel size of 0.1 μm.

2.2. PIPS-SEM

The sample blocks were subsampled into 2 mm thick slices taken perpendicular to the bedding with a maximum diameter of 8 mm. These subsamples were glued onto a modified 8 mm diameter SEM stub. After mechanical polishing of the top surface parallel to the stub the samples were mounted in a Gatan Precision