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Microtexture and mineralogy of Permian Lower Gondwana shales from South Karanpura Sub-basin, India

Abstract

The Permian Lower Gondwana organic rich shales from South Karanpura Sub-basin of Damodar Valley Basin, India have been investigated to understand their microtextural characteristics and mineralogy for evaluating their shale gas properties. X-ray diffraction (XRD) analysis was used for investigating the bulk mineralogy of the samples. The XRD results show that the studied shales mainly comprise of quartz, kaolinite, illite, muscovite, with little amount of feldspar, siderite and rutile. The microtextural properties such as nature and distribution of pores in the shales were studied using various imaging techniques such as field emission scanning electron microscopy (FE-SEM), focused ion beam scanning electron microscopy (FIB-SEM) and 3D X-ray microscopy (XRM). The results show occurrence of interparticle and intraparticle pores in various modes such as organic matter hosted, mineral matrix hosted and fracture pores. Organic matter hosted pores are more abundant in the Barakar shales, whereas, mineral matrix hosted pores are more common in the Barren Measures shales. Organic matter occurs as both structured and amorphous forms. Mineral matrix pores occur mainly along phyllosilicate cleavage planes. Three-dimensional shale volume obtained from 3D XRM analysis permitted visualization of kerogen and pore connectivity across the volumes.

Introduction

Understanding the microtextural features within organic rich shales is one of the very crucial tasks in investigating their shale gas potential, thus a substantial amount of research has been carried out in this field (Loucks et al., 2009, 2012; Chalmers et al., 2012; Milliken et al., 2013). Unlike the conventional reservoirs like sandstones and carbonates, shale reservoirs are complex, they are fine grained and rich in clay minerals which results in small pore size (micro- to nanometer) and low permeability (micro- to nanodarcy). Therefore, many traditional analytical techniques applied for pore characterization of conventional reservoirs are not very helpful for shale gas study. High resolution imaging techniques like FE-SEM, FIB-SEM and 3D XRM have given promising results in visualization of the distribution of micro- to nanometer scaled pores within the shales (Loucks et al., 2009; Chalmers et al., 2012; Boruah and Ganapathi, 2015). Bulk mineralogy of the shales is considered as another important attribute while analysing their gas potential. Mineral composition of any rock controls its brittleness, which controls its potential to crack during hydraulic fracturing. Other reservoir properties such as matrix permeability, porosity and texture are also dependent on mineralogy (Kuila and Prasad, 2013). The microtextural properties of the Lower Gondwana shales from the Damodar Valley Basin are mostly unexplored. Therefore, present study attempts an investigation of microtextural characteristics of shales from Barakar and Barren Measures formations from the South Karanpura Sub-basin. Distributions of pores and other microtextural features were studied using image analyses and the nature of the pore-systems is described qualitatively. X-ray diffraction (XRD) analysis was used for investigating the bulk mineralogy.

Samples and methods

A total of forty five borehole shale samples from the South Karanpura Sub-basin were collected. Twenty nine samples belonging to the Lower Permian Barakar Formation were collected from two boreholes. A set of sixteen samples from the Middle Permian Barren Measures Formation were collected from one borehole.

For X-ray diffraction (XRD) analysis the samples were powdered. The powdered samples were scanned in a PANalytical Empyrean X-ray diffractometer, from 5° to 70° 2θ using copper filtered K_α radiation (40 mA, 45 kV). Data processing as well as mineral identification and quantitative analyses were done using HighScore Plus software.

For field emission scanning electron microscopy (FE-SEM), small sub-samples were prepared by cutting the core samples parallel to the bedding planes. The samples were then dried overnight in an oven at 50 °C. The sample surfaces were coated with gold film to prevent electrostatic charge buildup and then placed on sample holders using double sided adhesive, electrically conductive carbon tapes. Then they were analysed using a Carl Zeiss Ultra 55 FE-SEM system with Oxford EDX system.

Small off cut block samples about 1 cm^2 were polished to create a level surface using dry silicon carbide grinding paper. After polishing, the samples were coated with gold to provide a conductive surface layer to minimize the effect of electrostatic charging. Each sample was analysed using a Zeiss-Auriga Compact FIB-SEM for imaging. ORS Visual SI software was used for data analysis.

A Zeiss Xradia 520 Versa 3D X-ray microscope was used for non-destructive 3D visualization of the samples. The data set was imported into ORS visual SI software to generate a 3D digital rock volume of the shale samples.

Results and discussions

The XRD data suggest that the shales belonging to the Barren Measures Formation are characterized by quartz (21.9-39.2%), kaolinite (18.8-37.4%), muscovite (11.3-26.4%), illite (6.6-24.5%), feldspar (0-11.5%), siderite (0-7.7%) and trace amount of rutile (0.3-0.9%). On the other hand shales from the Barakar Formation comprise of kaolinite (27.2-52.9%), quartz (23.6-38.4%), muscovite (5-26.4%), illite (0.1-19.2%), feldspar (0.3-30.3%), siderite (1.2-1.4%) and trace amount of rutile (0.2-1%). Clay minerals have a positive influence on methane sorption capacity as they provide larger surface areas and more adsorption sites than other minerals (Zhang et al., 2017). However, presence of large amounts of clay particles enhances the ductile properties of shale. Furthermore, some clay minerals have strong affinity for water that causes clay-rich shales to have large water contents and therefore reduced gas storage capacity (Zhang et al., 2017). Besides, clay-water interactions also create significant swelling in shale formations, causing a huge reduction in pore space and flow paths for gas movement in shale matrix (Zhang et al., 2017). These facts reduce the productivity of clay-rich formations. However, the expanding effect is much higher in smectite than other clay minerals (Rogala et al., 2013). Illite has swelling effect but it is not as great trouble as smectite in case of shale hydro-fracturing (Rogala et al., 2013). The XRD results of studied shales show that kaolinite and illite are the dominant clay minerals and smectite is absent.

Pore spaces within the matrix and natural fractures together form networks for gas to flow through shale formations to induced fractures during production (Loucks et al., 2012). A detailed analysis of FE-SEM images has revealed different types of matrix related pores in Barren Measures and Barakar shales from the South Karanpura Sub-basin (Figure 1). The pores majorly occur as intraparticle pores in organic matter, interparticle pores in mineral matrix, intarparticle pores in clay minerals and micas, intercrystalline pores between pyrite crystals within pyrite framboids and microfractures. High abundance of layered silicate minerals like micas and kaolinite is observed in Barren Measures shales. Pores mainly occur as intraparticle pores in between the layers of the mica or kaolinite bands (Figure 1a). However, organic matter hosted intraparticle pores were also found, though these are less common. Organic matter content of Barakar shales is high, thus dispersed organic matter within the mineral matrix is very commonly observed in these samples. Based on the SEM image analysis, organic matter hosted pores are the dominant pore types in the Barakar shale samples. Intraparticle pores occur within organic matter particles; their shapes vary from ellipsoidal to irregular. Organic matter observed in the images of the studied samples can be grouped into two types based on their forms. Structured organic matter is identified mainly based on the preserved morphological features of the original organic matter (Figure 1c). On the other hand, unstructured (amorphous) organic particles occur as homogeneous particulate debris with discrete form (Figure 1d). At places the amorphous organic matter are highly porous and spongy.

Clusters of pyrite framboids are found dispersed within the mineral matrix. Intercrystalline pores occur within the pyrite framboids. Many pyrite framboids are found in the sample, and the intracrystalline pores in some of the framboids are filled by clay (Figure 1b).

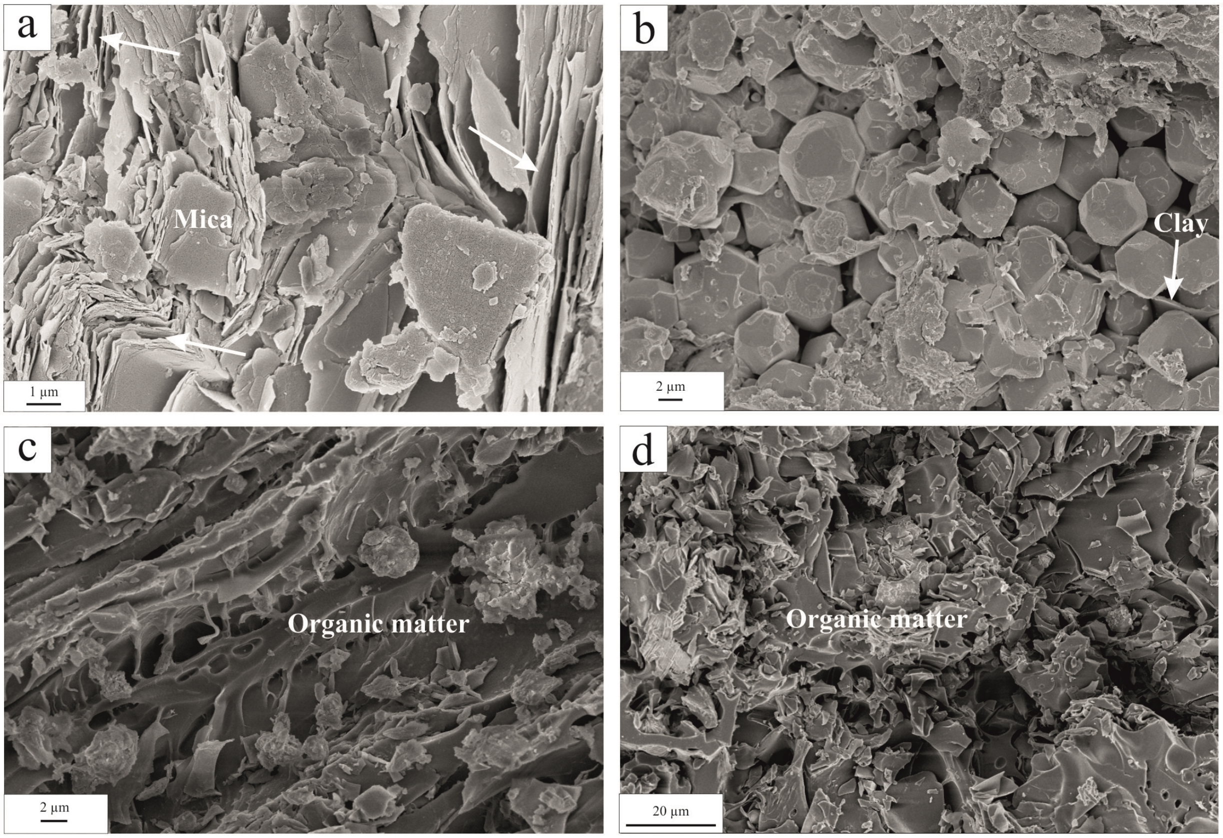

Figure 1. Different types of pores identified in the FESEM images of the studied shale samples. (a) pores within clay and mica, (b) pores within pyrite framboids, (c) structured organic matter hosting pores, (d) amorphous organic matter hosting pores.

The focused ion beam (FIB) improves image resolution by preventing most of the hardness-related topographic variations from forming on sample surfaces. The FIB, combined with FESEM devices, provides high resolution and access to site-specific observations. The images obtained from the studied samples show organic matter hosted as well as mineral matrix pores. Elongated and irregularly shaped organic matter, interparticle pores have developed at the edges of the organic matter, along the junction of organic matter and inorganic mineral matrix. Nanopres ranging in size from a few nm to less than 1 µm are observed in the samples. A common issue with FIB-SEM imaging is preferential milling causing an artifact called curtaining. Shale samples are heterogeneous with phases that have different material removal rates under the ion beam. This creates laminations in the cross-section, shown as vertical bands in the SEM image that may lead to segmentation errors. Another limitation of this technique is that the analysis volume is very small and that may not be representative of the larger sample.

3D X-ray microscopy (XRM) has been used in various geological disciplines. Use of this technique is rapidly increasing in oil and gas research. However, the application of this technique is comparatively new in shale gas research (Josh et al., 2012; Ma et al., 2017). In case of Permian shales of India, only one such report by Boruah and Ganapathi (2015) is available till date. The 2D images as well as the 3D reconstructed volumes show heterogeneity in the shales. Pyrite framboids and dispersed organic matter are easily identifiable in the images. 2D and 3D segmented images of different samples show connected and non-connected porosity. The total porosity values of the Barakar shale samples calculated from the

3D XRM ranges from 1.53 to 2.11%. The connected porosity ranges from 0.22 to 0.40% whereas the non-connected porosity ranges from 1.30 to 1.71%.

Conclusions

I. The XRD results show that shales from both the Barren Measures and Barakar formations comprise of quartz, kaolinite, muscovite, illite, feldspar, siderite and minor fractions of rutile. Clay group of minerals are higher in the samples than the quartz and carbonate groups. Clay content is known to have a positive influence on methane sorption capacities of shales.

II. Images obtained from the FE-SEM, FIB-SEM and 3D XRM analysis show various modes of occurrence of pores in the shales, such as organic matter hosted, mineral matrix hosted and fracture pores. The pores are both interparticle and intraparticle. Organic matter hosted pores are more abundant in the Barakar shales, whereas, mineral matrix hosted pores are more common in the Barren Measures shales. Organic matter occurs as both structured and amorphous in form and the associated pores are ellipsoidal to irregular in shape. Mineral matrix pores occur mainly along phyllosilicate cleavage planes that are separated by the bending of a grain, as well as interparticle spaces held open within layers of micas and/or clays. These pores are commonly elongated in shape. Three-dimensional renderings generated from the series of slices obtained from 3D XRM analysis permitted visualization of kerogen and pore connectivity across the volumes.

III. The total porosity values of the Barakar shale samples calculated from the 3D XRM ranges from 1.53 to 2.11%. The connected porosity ranges from 0.22 to 0.40% whereas the non-connected porosity ranges from 1.30 to 1.71%.

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