



Source rock characteristics and pore characterization of Indian shale



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ABSTRACT

Indian shale samples, collected from Permian Damodar valley and Late Oligocene Assam basin were collected and studied thoroughly to know their hydrocarbon generation potential and pore characteristics. Rock eval pyrolysis, low-pressure adsorption (N₂), and x-ray diffraction analyses were performed on four Permian and three Late Oligocene shales considered as potential targets for shale gas exploration in India. The relationship between the source rock characteristics, mineralogy, BET surface area and pore volume were discussed. The study reveals a good to excellent hydrocarbon potential with high TOC content (4.8–37.3) of type III and type II+III kerogen. Thermal maturity data shows that Permian samples are mature (T_{max} 440 °C–465 °C), whereas Late Oligocene samples are immature (T_{max} 422 °C–434 °C). Mineralogy reveals a clay rich nature of the shale samples. BET surface area of the samples were between 0.7 and 13.6 m²/g and total pore volume ranged from 0.011 to 0.027 cc/g. Pore size distribution for all samples are found to be unimodal or bimodal (peaks at around pore radius 1.1 nm and at >3 nm). Kerogen type is observed to have a significant contribution on pore volume; type II+III shale samples (HI > 300) have higher average pore radius (6.62 nm) and higher average total pore volume (0.020 cc/g) than that of type III samples (4.2 nm and 0.018 cc/g respectively). Samples of higher surface area and total pore volume show smaller average pore radius. Clay minerals positively influence the BET surface area and total pore volumes of the samples. A negative relationship between quartz and TOC in the shale samples suggest a non-biogenic detrital source of quartz grains.

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1. Introduction

In the last few decades shale gas has globally emerged as an unconventional energy resource. Shale acts as both source and reservoir to the generated shale gas, primarily methane; it is generated from different organic matters trapped in shale being subjected to pressure and temperature with increasing burial. Type III organic matter is identified as a source for commercial hydrocarbon deposits in various basins throughout the world (Bertrand et al., 1986; Boreham and Powell, 1991, 1993; Thomas, 1982; Thompson et al., 1994). The generated methane remains trapped in the pore networks of minerals and organic matter. Although the success of shale gas production is causing a growing interests in gas-shale investigation, shale as an unconventional reservoir rock is highly complex. The complex nature of the shale reservoir is due to their heterogeneity in terms of organic inorganic composition,

porosity and pore size distribution, texture. Shale composition, pore systems, texture and kerogen characteristics differ widely between different shale plays and with different shale formations within the same play itself; hence, one shale play never behaves like another. For the past few decades, studies are being done in order to identify and characterize the pore systems, important textural and compositional features within shale, which play a significant role in shale's capability to generate, store, and produce hydrocarbon (Chalmers et al., 2009; Curtis et al., 2011; Schieber, 2010; Sondergeld et al., 2010; Wang and Reed, 2009). The discovery of ion milling and scanning electron microscopy (SEM) has helped in finding the pores in the organic matter in a precise manner (Loucks et al., 2009). The shale matrix is predominantly composed of micropores (pores < 2 nm dia) and mesopores (2–50 nm dia) and these pores are mainly associated with different clay minerals and organic matter (Kuila and Prasad, 2011). It is of utmost importance to analyze the nature of these clays and organic matter, so that their control on the pore-size distribution and pore network in shale can be critically understood.

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A variety of the geological factors such as, mineralogy, source rock characteristics, texture of shale, the types of clays and organics, govern the porosity and pore-size distribution and finally the matrix permeability of the reservoir (Chalmers et al., 2012a). The understanding of porosity development and its controls in shale is of utmost importance as this will help in understanding the producibility and storage potential of a reservoir. Different kinds of research works with porosity of shale are being performed on samples with different thermal maturities. These researches are focusing on different genesis of pores, varying types of pores, their distribution and controlling attributes of pore networks in shale (Bernard et al., 2012; Heath et al., 2011; Schieber, 2010). However, the development of porosity especially micro and meso porosity in the shale over different range of thermal maturities have not been thoroughly explored. Since in organic-rich shales, a significant portion of the total porosity is within the size ranges of 2–50 nm (mesopores), it is of utmost importance to understand how they get affected through varied geological factors and their development with gradual thermal maturity.

India with an objective to exploit its shale-gas resource has started to approach all the research areas related to shale gas. However these studies are in very nascent stage with very few published results. In this paper a multidisciplinary approach has been taken to understand the source rock characteristics and nature of porosity and pore size distribution of a few Indian shale samples from different age groups which are potential for future shale gas explorations. Four Permian and three Late-Oligocene samples were characterized in terms of composition and distribution of minerals and organic matter, porosity and pore size distribution. Assessment of source rock characteristics is based on the results of geochemical, petrographic, and vitrinite reflectance analyses. Here an effort has been made to study the controls of mineralogy and kerogen characteristics on the porosity and pore size distribution of Indian shales.

2. Materials and methods

2.1. Sample collection

Studied samples were collected from Damodar valley and Upper Assam basin. Four shale samples of Permian age were collected from Damodar valley basin and three samples of Late-Oligocene age were collected from Upper Assam Basin. The sample locations are shown in Fig. 1. All the samples were collected from Opencast/underground mine of different collieries. The sample name, corresponding coalfield, formation, age and depth are shown in Table 1.

2.2. Mineralogy

Shale samples were crushed into fine powder (–212 μm) for analysis. X-ray diffraction analyses were done in IIT Kharagpur, using Bruker D8 Advance instrument with Cu target and lynxeye detector. X-ray powder diffraction (XRPD) patterns were recorded for 2θ values in the range of 7–80° using Copper K α radiation. The clay and non-clay minerals peaks were distinguished, using basal spacings (d) and 2-theta for Cu K-alpha radiation. The quantitative analysis was carried out by Rietveld analyses using the software Topas.

2.3. Rock-Eval pyrolysis

All of the collected samples were wiped with wet towel to remove surface contaminants or dust particles. Then the samples were air dried for few minutes prior to crushing. The samples were

crushed to powder and screened through –212 μm sized mesh and then were well homogenized before carrying out the analyses. A Rock-Eval 6 instrument was used for the rock eval pyrolysis and TOC analysis of the samples. Different parameters like TOC content, S1, S2, S3 pyrolysis yields and temperature of maximum S2 pyrolysis yield (Tmax) were measured. Genetic potential (GP), hydrogen Index (HI), oxygen index (OI), and production index (PI) were calculated. Particulars on rock eval, parameters acquired and explanatory guidelines have been discussed by several workers (Lafargue et al., 1998; Peters and Cassa, 1994). Estimated vitrinite reflectance (EVRo) was calculated from Tmax using the equation below after Jarvie et al. (2001):

$$\text{EVRo \%} = 0.0180 * \text{Tmax} - 7.16 \quad (1)$$

2.4. Low pressure N₂ isotherm

Samples were crushed to <250 μm for low pressure N₂ sorption analysis (N₂ surface area measurement) using Micrometrics TriStar 3000 instrument. At first the degassing of shale samples were done in 150 °C for 12 h in a vacuum oven before being fed to N₂ adsorption analysis. At –196.15 °C nitrogen isotherms were measured under relative pressure range of 0.001–0.9 which ultimately provides the information on total pore volume, pore size distribution, BET surface area. The surface areas (m^2/g) were calculated using the Brunauer–Emmett–Teller (BET) method with the relative pressure range being 0.049–0.300 (P/P_0 , where P is the gas vapour pressure in the system and P_0 is the vapour pressure above the gas at the temperature of interest), following the equation:

$$\frac{1}{W \left(\left(\frac{P_0}{P} \right) - 1 \right)} = \frac{1}{W_m C} + C \frac{1}{W_m C \left(\frac{P}{P_0} \right)} \quad (2)$$

where W is the weight of the sorbed gas at relative pressure P/P_0 , W_m is the weight of the monolayer adsorbent (N₂), C is the BET constant which relates to the sorption energy between adsorbent and adsorbate. The total pore volume was calculated as the molar volume of adsorbed nitrogen at the relative pressure of 0.99. The micropore volume and external surface area was calculated using t-plot method (De Boer et al., 1963) of N₂ adsorption isotherm data. The micropore surface area was estimated by subtracting external surface area from total surface area. The N₂ data of the crushed sample were interpreted using multi-point Brunauer–Emmett–Teller (BET) and Langmuir analysis for surface area and Density Functional Theory (DFT) analysis for pore size distributions (Kuila and Prasad, 2013).

3. Results

3.1. Mineralogy

XRD analysis was done in order to understand the presence of different mineral phases and ultimately their controls on the pore size distribution. Samples have a varying combination of quartz, clay and carbonate (Table 2). The range of quartz content of all the samples is below 40%. The shale samples are found to be richer in clay with average clay content (smectite+illite+kaolinite) of 55.9% and average quartz content of 33.3%. The quartz content ranges between 6.2% and 55.8% while clay content ranges between 32.2% and 91.7%. Mineral percentages for all the samples are given in Table 2 and they exhibit a wide range of mineral compositions. It is

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