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Pore Structure Characterization of Shales from Kachchh and Kaladgi Basins

[1]Sagar Thakurdas*, [2]Dhananjay Meshram, [3]Bhawanisingh G. Desai, and [4]Uday Kulkarni

Author's Affiliations:
[1]Assistant Professor, Department of Geology, Nowrosjee Wadia College, Pune, Maharashtra 411001, India
E-mail: sagarthakurdas@gmail.com
[2]Professor, Department of Geology, Savitribai Phule Pune University, Pune, Maharashtra 411007, India.
E-mail: dcmeshram@unipune.ernet.in
[3]Associate Professor, Pandit Deendayal Energy University, Gandhinagar, Gujarat 382007, India.
E-mail:bhawanigd@gmail.com
[4] Principal, Moolji Jaitha College, Jalgaon, Maharashtra 425001, India.
E-mail: drudkulkarni@gmail.com

***Corresponding Author: Sagar Mahendra Thakurdas** Assistant Professor, Department of Geology, Nowrosjee Wadia College, Pune, Maharashtra 411001, India
E-mail: sagarthakurdas@gmail.com

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Abstract:
Estimating transport and storage properties of shale is a challenging task, due to the lack of understanding of their pore size characteristics. Matrix of shales have micro (pores less than 2 nm diameter) to meso-pores (pores with 2-50 nm diameter) within them. These nano scale pores are mainly associated with clay minerals. The pore structure characteristics of shales viz. surface area, pore size distribution and pore volume, from Kachchh basin of Gujrat and Kaladgi basin of Karnataka, were evaluated using N_2physisorption analyses. The samples were analyzed by applying Brunauer–Emmett–Teller (BET) theory for surface area calculation and Barrett–Joyner–Halenda (BJH) theory for understanding the pore size distribution and pore volume. The pore structural characteristics were compared with the mineral composition obtained from X-ray diffraction analysis. Both sample sets show Bi-modal pore size distribution. Bi-modal pore size distribution is indicative of the intra-layer clay pore space. Differential porevolume indicates that porosity is very low in Kaladgi shales in comparison to Kachchh shales.

Keywords: *Kachchh, Kaladgi, Shale, Pore size distribution, Shale, BET*

1. INTRODUCTION

Shale gas is an important energy source for the world, and its development is likely to continue to have a significant impact on global energy markets in the years to come. It being an unconventional resource of petroleum, has been a topic of research for oil and gas industry around the world. The production of petroleum from unconventional resources needs improvement in the techniques, as well as, greater insight into the reservoir rock and its petrophysical properties (Siswandani et al. 2015). Shale gas production is increasing exponentially and it is substituting the conventional fossil fuel in many parts of the

world to meet the ever increasing energy demand. As per EIA report (2014), the global natural gas consumption is likely to escalate by 50%, by 2035. One third of this demand is expected to be satisfied by the unconventional sources. It also states that the technically recoverable global shale gas resources stand at approximately 7299 Tcf spanning over 137 shale formations in 95 basins and 41 countries. India ranks third in terms of energy consumption in the world followed by China and USA (BP Statistical Review, 2016), but it has got inadequate energy resources. India has great interest in developing its Shale Gas resources to meet the ever increasing demand for energy.

Shales are inherently anisotropic and they are made up of clay-sized (less than 1/256 mm (4 microns in diameter) particles (Sondhi, 2011). Porosity in shales is associated with clays and the organic matter preserved within. Pores in shales are nanometer (nm) in scale. The pore throat is as small as 5nm (Loucks et al., 2009). Different types of microscale pores are present in organic matter, within clay mineral matrix, within pyrite framboids, within the preserved fossils and in the form of microcracks (Sondergeld et al., 2010).

1.1 Geological Setting and Sampling

Kachchh basin is located in the north-west of India and it demonstrates excellent exposures of the Mesozoic sediments. Though the basin has rocks range from Middle Jurassic to recent (Biswas, 1993), the Mesozoic sediments form the major part of the sediment thickness. Tertiary sediments are observed in the region surrounding the major uplifts (Biswas, 1980). These rocks are divided into strastigraphic units namely Jhurio, Jumara, Jhuran and Bhuj Formation. The older Jhurio and Jumara formations represent marine sediments, Jhuran is paralic and younger Bhuj sandstone formation is deltaic to fluviatile (Biswas, 1993). Tertiary sediments are observed in the region surrounding the major uplifts (Biswas, 1980).The sediment deposition has occurred dominantly in the shallow marine to deltaic environment. Middle Rudramata Member is a prominent shale member exposed in the Kachchh basin. It is organic-rich shale with varying Total Organic Content (TOC) Values ranging from 0.2 up to 7 % (Desai et al., 2018). The Shales were deposited in the river-dominated deltaic environment (Desai and Biswas, 2018). The deposition of these shales was governed by the river-dominated deltaic environment (Desai and Biswas, 2018). The samples were collected from 4 different locations (Kodki, Jhuran river, Lakhpat and Khari river) spanning over the Kachchh basin (Figure 1). The representative samples from these four locations are named K3, K4, K5 and K6. Stratigraphy of the Kachchh basin is given in table 1 and that of Kaladgi Basin is given in figure 1B.).

The Kaladgisuper group of rocks are divided into lower Bagalkot and Upper Badami groups based on the presence of an angular unconformity. The Lokapur subgroup of Bagalkot group is the thickest and it is spread all over the basin. The rocks belonging to Lokapur subgroup are seen in the form of doubly plunging synclinoria (Kale et al. 2011). The sampling site is located at the boundary of this syncline near Yadhalli village (Figure 1B). Yadhalli Argillite of Lokapur subgroup were deposited in Mud Flats (Patil-Pillai and Kale, 1999). The samples were collected from the Yadhalli Argillite formation and are named as Y1 and Y2, in this paper.

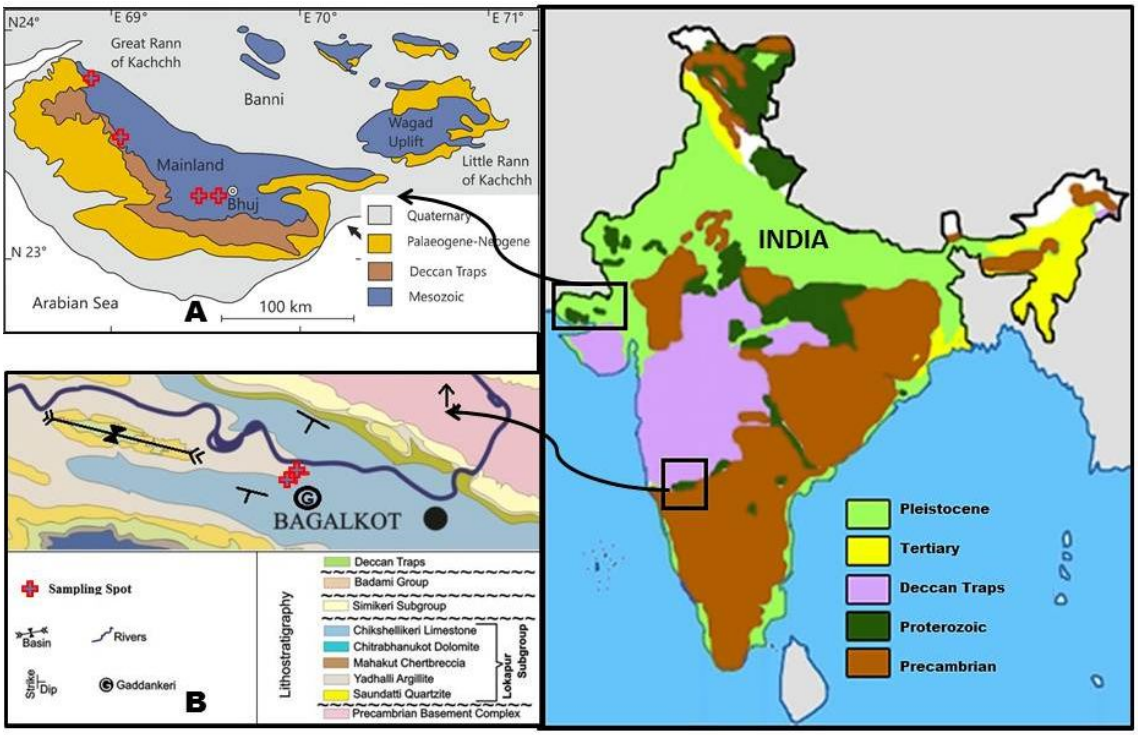

Figure 1: Geological map of India [A] Map of Kachchh Basin with sampling locations [B] Map of Kaladgi basin and its stratigraphy with sampling Locations.

Table 1: Mesozoic stratigraphy of Kachchh basin (after Biswas, 2016)

Age	Litho-Unit Thickness	Lithology	Environment
Kachchh Mainland			
Early Cretaceous	Bhuj Formation (400-900 m+)	**Upper Part:** Coarse grained, felspathic sandstone **Ukra Member** **Lower part:** Brown and reddish felspathic sandstone, ironstone and kaolinitic shale	Fluviatile to deltaic
Tithonian To Kimmeridhian	Jhuran Formation (375 - 850 m)	**Upper Member:** Pink and yellow sandstone with minor shale **Middle Member:** Grey shale Rudramata Shale member **Lower Member:** Shale and sandstone with calcareous bands	Infra-littoral Deltaic
Callovian to Oxfordian	Jumara Formation (300 m)	DosaOolite: Grey gypseous shale Middle member Lower Member	Sub-littoral
Upper Bathonian to Callovian	Jhurio Formation (325 m +)	Bedded white limestone Golden Oolite Limestone / Shale Limestone / Shale interbedded	Sub-littoral

2. METHODOLOGY

Shale samples from two different basins (Kachchh and Kaladgi) and two different eras (Mesozoic and Meso-Proterozoic, respectively) were analyzed and compared with respect to the mineral content, pore size distribution and pore volume. Mineralogical composition was obtained by X-ray diffraction analysis of selected representative samples using RIGAKU ULTIMA IV instrument with Copper (Cu) source.Thin section analysis of the same samples was carried out to understand the rock framework and occurrence of organic matter.

Pore size characterization using data obtained from low pressure N_2 gas adsorption technique was instrumental. Low pressure N_2 gas adsorption measurement is one the most effective methods in understanding the pore size distribution, microporosity and the surface area which can help in estimation of adsorbed gas capacity (Hinai, 2014). Cubical shale rock samples with 3x3 mm dimension were used for N_2 gas adsorption analysis in this study. Each sample weighed between 0.260g to 0.970g. This experiment was performed on a NOVA 2200e (Quantachrome) Surface Area & Pore Analyzerat the bathing temperature of 77.3 K.

The samples were degassed and dried at 180^0 C for 4 hours just before the analysis for removal of moisture and to ensure that the samples are clean. N_2 gas was allowed to adsorb into the degassed samples under the constant temperature to obtain the adsorption isotherms. Pore size distribution was obtained by using the Barrett, BJH theory based on N_2 adsorption isotherm.

3. RESULTS AND DISCUSSION

The yadhalli Argillites are fine grained, smoky blue to purple in color and it is soft and massive in nature. These appear to be ferruginous and siliceous shale deposits. Whereas shales from Kodki road cutting and Khari River are black, flaky, fissile and micaceous. Jhuran shale is fragile and black. It is important to note that the Kachchh shale is Mesozoic while shale from Kaladgi basin is much older belonging to Meso-Proterozoic age.

1. Comparative analysis of the pore size distribution data between Kachchh basin and Kaladgi basin shale samples obtained by BJH- N_2 adsorption method indicates that the pore throat size mainly lies between 3 to 4 nm and 10 to 20 nm, second mode being a major peak.
2. Kachchh shale show minor peaks at 3 nm pore throat and can be distinguished easily. Jhuran Shale (K3 and K7) show distinct peak between 3 to 4 nm. This indicates abundance of micro pores in the organic matter. The thin section analysis has shown presence of organic matter in these samples.
3. The comparison of differential pore volume suggests that porosity is very low in Kaladgi samples (Y1 and Y2) and it is evident from the hand specimen as well (Figure 3).
4. Shales from Kachchh basin have surface area ranging between 8.371 m^2/g to 26.783 In contrast, shales from Kaladgi basin show the surface area between 2.549 m^2/g to 2.777 m^2/g. The large surface area indicates the presence of high content of clays, organic matter and very fine rock matrix. The XRD data also supplements this fact. K4 has more clay percentage than other samples. The optical studies have also shown that the organic matter in K4 is comparatively higher in Kachchh Basin.

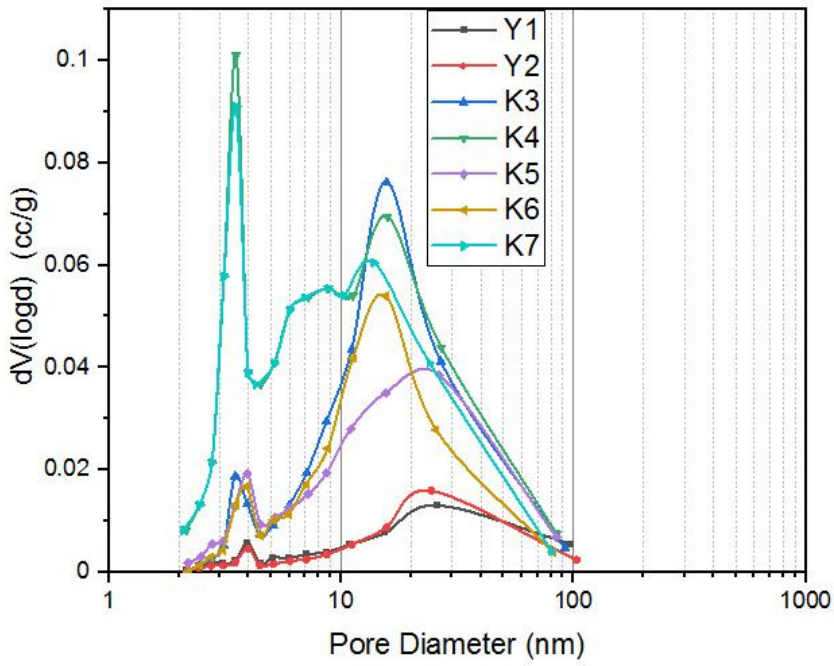

Figure 2: Comparative Pore-size distribution of Shales obtained from N_2 adsorption isotherm (BJH method). The shale samples show bimodal distribution with a major peak between 20-30 nm with a minor peak around 3-4 nm.

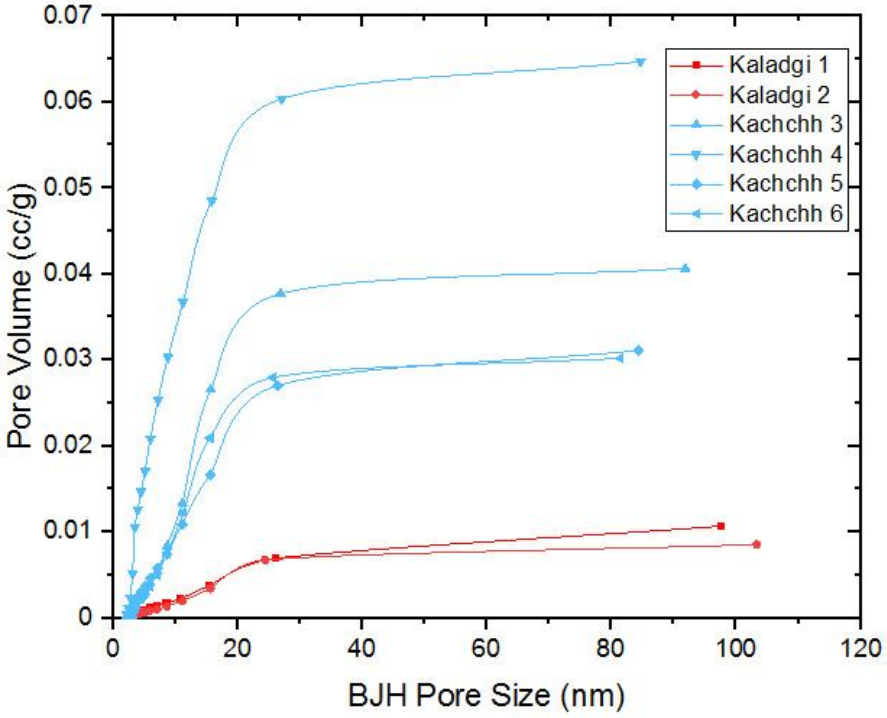

Figure 3: Plot of Pore volume Vs Pore Size

4. CONCLUSIONS

- The large surface area indicates the presence of high content of clays, organic matter and very fine rock matrix.
- Bi-modal pore size distribution observed in shales from both basins supplements the multi-scale porosity associated with clay structures. Mode of 3-4 nm pore throat size corresponds to interlayer and intra-particle pores, whereas mode of 20-30 nm corresponds to inter-particular mesopores.
- Differential porevolume indicates that porosity is very low in Kaladgi shales in comparison to Kachchh shales.

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