Mineralogical assemblage of Cambay Shale of North Cambay Basin, Gujarat, India

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Abstract

Shale Gas exploration and production plays an essential role in ensuring energy security and economic stability. Tapping these reserves using cost-effective technologies can really change the India's energy supply and value chain. Cambay Shale of Cambay Basin has been proved as the most prominent Indian Shale reserve and in this work an attempt has been made to understand the geographical and mineralogical distribution of this Shale. A sample study on Cambay Shale of North Tectonic Block of Cambay Basin has been ascertained to understand the mineralogy, porosity and maturity with respect to depth. The study involves the use of integrated measurement techniques mainly high-pressure porosity (HPP), scanning electron microscopy (SEM) and Fourier transform electron microscopy (FTIR). Approximately, 13 samples were analysed to understand the mineralogy of this shale and it was observed that it is very clayey in nature with major traces of Illite and minor traces of rutile and kaolinite. The other measurements (i.e., SEM and HPP) were done on two specific samples which confirm the presence of clay-rich porous minerals with high organic matter content.

Keywords Pore size · Mineralogy · SEM · Shale porosity

Introduction

Shale gas prospects are emerging rapidly as a promising energy source globally (Dayal et al. 2013). However, its exploration and exploitation in India is in nascent stage. A resource potential of around 2000 TCF of Shale Gas has been prognosticated in Indian Shales and according to preliminary data analysis by Directorate General of Hydrocarbons (DGH), Gondwana, Cambay Basin, Krishna—Godavari, Cauvery, Assam-Arakan, Rajasthan, Vindhyan and Bengal are the most promising shale gas plays in India. The present study is an attempt to understand the mineralogical characteristics of one of the prominent shale basins (i.e., Cambay Basin) of India.

Shale brittleness and its association with mineralogy and rock fabric plays is one of the factors suggested important with respect to a shale becoming significant resource play

(Dargahi et al. 2013). Brittleness plays a significant role in effective hydraulic fracturing for shale gas production and is related to mineralogy, mechanical properties and pore features in shale (Guo et al. 2012). A high clay abundance indicates low material brittleness and fracability. Therefore, it is important to understand the clay composition in shale which is quite challenging as shales are chemically complex and heterogeneous in nature. It has been observed through the open source literature (Hosterman and Loferski (1981), Dargahi et al. 2013, Kendall and Norton (1974), Yang et al. 2015, Abouelresh et al. 2016, Guerra et al. 2017, Akbar et al. (2017), Yang et al. 2018) that physical properties, chemical properties, water content clay type, distribution and size are the key important parameters for identifying mineralogical and elemental distribution and their effect on shale brittleness and fracability.

Numerous characterization techniques have been used to understand the mineralogy qualitatively and quantitatively. Thin section analysis, X-Ray diffraction (XRD) and Fourier transform infrared spectroscopy (FTIR) are the primary quantitative methods used for the mineral characterization (Ballard 2007). Gamero-Diaz et al. (2013) presented a detailed mineralogy based description for organic mudstones using inorganic mineralogy, primarily X-ray diffraction and

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geochemical log data. Similar investigations which include FTIR and XRD have been done on Perth Basin for Clay mineralogy evaluation (Dargahi et al. 2013). According to their investigations, XRD can be used to identify and quantify minerals but FTIR is efficient in providing complementary information on minerals and amorphous organic matter. A recently introduced new technology, i.e., diffusive reflectance infrared Fourier transform spectroscopy (DRIFTS) is very effective technique for simultaneously quantifying Kerogen and mineralogy of samples. This has been used to assess the core and cutting samples of wells in North and South America (Herron et al. 2014). Linear Swelling Meter, Cation Exchange Capacity (Abouelresh et al. 2016) and Quantitative Evaluation of Minerals Using Scanning Electron Microscopy (QEMSCAN), QEMSCAN coupled with CT Scanning (Ahmad and Haghighi 2012), Pulse Neutron Spectrometry Tool (Jacobson et al. 2009) have also been used to assess the distribution of different minerals in a porous samples. Therefore, FTIR, XRD and SEM are the most commonly and frequently used characterization technique for mineralogical assessment (Buller et al. 2014, Abouelresh et al. 2016, Rodriguez et al. 2015, Guerra et al. 2017, Akbar et al. (2017), Yang et al. 2018). However, for the identification of elemental composition of shales, X-ray fluorescence or energy dispersive XRF is the most commonly used technique (Guerra et al. 2017; Turner et al. 2015; Abouelresh et al. 2016; Rodriguez et al. 2015). Following the most frequent and common use of FTIR and SEM characterization technique in shale samples, the same techniques are used in the Shale samples of Cambay Shale of Cambay Basin, India to understand the mineralogy, porosity and pore structure. The data and results presented in this study can be used as a fundamental starting point for continuing research on the nature, pore size distribution, pore characterization of Cambay shale system.

Presently, there is no commercial shale gas production in India. Oil and gas production from these shale reserves can lead to reduction in India's dependence on imported crude oil which will result in foreign exchange savings. Understanding of Shale reservoir characteristics (such as Geophysical, Petrophysical, Geomechanics, etc.) can be useful in assessing the shale gas commercial production. Brittleness plays a significant role in effective hydraulic fracturing for shale gas production and is related to mineralogy, mechanical properties and pore features in shale. This paper is a pilot scale study to understand Cambay shale mineralogy which if augmented with geo-mechanics data can help in assessing and simulating the producing capacity of this shale at the commercial level.

Study area: geological settings and stratigraphy

The Cambay basin a rich petroleum province of India is a narrow, elongated rift graben, extending from Surat in the south to Sanchor in the north (Fig. 1) (Biswas 1977). It began evolving following the extensive outpour of Deccan Basalts during late Cretaceous covering large tracts of western and Central India (Bhandari and Chowdhary 1975). The basin is divided into five tectonic blocks—Sanchore, Ahmedabad, Tarapur, Broach and Narmada. The areal extent of this basin is about 55,000 sq. km (Onland) and has several sub basins filled with different sediments of age Paleocene to Early Eocene. The sedimentation in this basin is controlled by pre rift, syn rift and post rift stages. The Early Tertiary sediments ranging in age from Paleocene to Early Eocene represent syn-rift stage of deposition that was controlled by faults and basement highs in an expanding rift system (Balakrishnan et al. 1997). The Middle Eocene witnessed a regressive phase with oscillating conditions of deposition and development of deltaic sequences in the entire basin (Biswas 1977).

During Early Eocene age, a thick dark grey shale sequence, known as Cambay Shale was deposited which is the main hydrocarbon source rock of this basin. Paleocene to Oligocene age is the true age of source rock intervals of Cambay basin and are mainly dominated by sandstones, siltstones, shales (Kundu and Wani 1992). There is a lithological heterogeneity which give rise to hydrocarbons entrapments and development of secondary porosity (Hasan et al. 2013). Stratigraphically, the Deccan trap which is probably cretaceous to Palaeocene age is the tectonic basement (EIA 2013) of Cambay Basin. The Olpad formation overlying the Deccan trap consists of Alluvial Fans and deltas. The Olpad formation is overlain by older and younger cambay shale formation. The Cambay Shale showed marine sedimentation and is the episode of deposition of coarser clastics. Overlain to this is the Kalol formation which is a prolific producer in the Cambay Basin. Kalol has many cycles of deposition, Kalol IX + X is the producer which is producing for the last 40 years. Kalol is an intercalation of sandstone and siltstone within Shales and at places coal is found (Fig. 2).

Hydrocarbon production has declined during the recent years. To overcome the deficient hydrocarbons, exploiting shale gas reserves is the best alternate as they have enormous amount of gas and can continue to produce at a steady rate for decades (Frantz and Schlumberger 2005).

Fig. 1 Geological settings of Cambay Basin (EIA 2013)

Samples and methods

Samples

The cutting samples were collected from Cambay Shale of North Tectonic Block of Cambay Basin, Gujarat, India. Approximately 13 samples were collected from the well drilled through Cambay Shale over the cored interval of 1250–1378 m. Characterization technique, i.e., Fourier-transform infrared microscopy (FTIR) was performed on all these 13 samples to investigate the mineralogy. Other measurements like Scanning Electron Microscopy (SEM) and

High Pressure Porosity (HPP) were done on only selected few samples. The test summary is given below in Table 1.

Methods

Mineralogical investigation by FTIR

FTIR measurements were performed on 13 Shale samples using Thermo Fisher Scientific FTIR Instrument. A FTIR spectrophotometer consists of a source, interferometer, detector, sample holder and a computer. The instrument

ERA	BASIN		CAMBAY	
	PERIOD	EPOCH		
CENOZOIC	QUATERNARY	Holocene		
		Pleistocene	Gujarat Alluvium	
	TERTIARY	Pliocene	Jambusar Fm Broach Fm Jhagadia Fm <i>Kand Fm</i>	
		Miocene	<i>Babaguru Fm</i> <i>Tarkesvar Fm</i>	
			<i>Dadhar Fm/ Tarapur Shale</i>	
			<i>Kaloj Fm</i>	
		Eocene	<i>Kadi Fm</i>	<i>Younger Cambay Shale</i>
			<i>Older Cambay Shale</i>	
		Paleocene	<i>Olpad Fm</i>	
	CRETACEOUS	Upper	<i>Deccan Traps</i>	
		Lower		
MESOZOIC	JURASSIC	Upper		
	TRIASSIC			

Fig. 2 Stratigraphy of Cambay Basin (EIA 2013)

record information about the sample placed in the Infrared beam coming from the source at desired frequency. All types of minerals like Clay, Quartz, Kaolinite, Illite, etc. can be identified through this instrument (Ballard 2007). Figure 3a, b illustrates the minerals identified in the samples at depth intervals mentioned in Table 1.

Scanning electron microscopy (SEM)

SEM observations were conducted using Helios Nano Lab 650. The samples were first milled inside of the Dual Beam FIM/FE-SEM system with a 30 kV focussed gallium ion beam. Imaging was performed using backscattered electrons at an accelerating voltage of 1 kV (Abouelresh et al. 2016). The images are shown and discussed in Fig. 5a, b. This measurement was performed on two samples at depth interval of L1 (3600–3610 m) and U9 (1327–1328 m).

High-pressure porosity (HPP) measurement

In the high-pressure porosity measurement test, the sample is first cut in to length-1.25 inch and diameter of 1 inch

Table 1 Experiment matrix

Depth (m)	U1	U2	U3	U4	U5	U6	U7	U8	U9	U10	U11	U12	L1
	1250–1255	1265–1270	1275–1280	1285–1290	1295–1300	1300–1303	1312–1315	1326–1326.5	1327–1328	1334–1337	1377.5–1378	1378–1378.5	3600–3610
Test per-formed	FTIR	FTIR	FTIR	FTIR	FTIR	FTIR	FTIR	FTIR	FTIR, SEM, HPP	FTIR	FTIR	FTIR	FTIR, SEM, HPP

Fig. 3 **a** Illustrates the percentage of Minerals identified in the samples U1 to U12 and L1. The percentage of Clay Minerals is high as compared to other minerals. **b** Gives the distribution of clay minerals, i.e., Illite, Smectite, kaolinite, Chlorite and Mixed Clays with respect to depth intervals (U1–U12 and L1). This shows Illite is the dominant clay mineral as compared to other minerals present in the sample

(Fig. 4a) and then placed in oven for overnight drying. After drying, the samples are put into desiccator and then placed in the HPP set up (Fig. 4b) for porosity investigations. The samples taken for this investigation are L1-Horizontal and Vertical and U9. Results are tabulated in Table 2.

Results and discussions

Shale mineralogy

The mineralogy variation with respect to depth over the cored interval in the well drilled through Cambay Shale has been shown in Fig. 3a. Amongst all, the clay mineral shows the highest percentage (in the range of 54–80%). The second most dominating mineral is Quartz varying from 3 to 25%.

Figure 3b shows the distribution of clay minerals (mainly Illite, Kaolinite, Smectite, Chlorite and Mixed Clays) with respect to depth. Illite is the dominant clay mineral contributing approximately 62–65% to the overall clay content of the dataset. The percentage of the carbonate minerals is quite low as compared to other minerals present like quartz and feldspar in the dataset. Apatite (<4% wt.) and pyrite (others) occurs in most of the samples, however, the percentage is quite low. The results shows that the Cambay shale is very clayey in nature with Illite as the prime dominant clay factor.

Porosity measurements

Porosity of Cambay shale samples have been determined experimentally by high pressure porosity test at different

Fig. 4 a HPP sample size: 1.25 inch length and 1 inch diameter and b HPP sample cup

depth intervals (Fig. 4a, b). The porosity of sample—U9 and L1 is 16 and 11% at depth interval 1327–1328 and 3600–3610 m respectively. Table 2 shows the porosity results.

Scanning electron microscopy

Both samples appear to be clay heavy with U9 (Fig. 5a) having highest clay content. The clays in U9 (Fig. 5b) appear to be Illite, however, sample L1 (Fig. 5a) has a very high chlorite content. Sample L1 (Fig. 5a) also has a very high rutile content and has a larger average grain size with larger organic particles versus U9 (Fig. 5b) which has smaller grain and organic sizes. Siderite is the other mineral present in the U9 (Fig. 5b) sample. The sample L1 (Fig. 5a) show clay rich porosity and high organic matter content.

Shale producibility potential

Shale gas in India is in nascent stage. The initial drilling and hydraulic fracturing by Oil and Natural Gas Corporation Limited (ONGC) showed interesting results in the context of Shale production capacity and source rock maturity. Shale Oil was indicated during post hydraulic fracturing activation in Cambay pilot well. Cumulative oil knocked out till 21.03.2017 from this well was 2.35 m³. The observed kero-gen typing was II and III with moderate to good conversions. In total, ONGC has drilled 7 wells for shale gas exploration and have obtained gas kick/oil shows while drilling. Some private operators have also confirmed the same. Recently in 2018, ONGC received Ministry of Petroleum and Natural Gas (MOPNG) nod to drill 11 more shale wells in Cambay Basin at Mehsana, Ahmedabad and Bharuch in Gujarat to understand the shale from reservoir as well as source rock point of view. The total estimated cost of the project is Rs. 366 Crores. In India, Shale has a potential of producing oil/gas at the commercial level. This requires extensive research and advance exploitation techniques.

The kerogen typing of the samples drilled over the cored interval of 1250–1378 m is mainly Type II and III. A Vitrinite reflectance plot with respect to depth (Fig. 6a) and production index (Fig. 6b) has been plotted to identify the shale thermal maturity and oil producing zones in the drilled sections. The samples with Ro (%) in the range of 0.6–0.8%

Table 2 Porosity measurement by HPP

S. no.	Reference name	Depth (m)	Temp. (°C)	HPP porosity (%)	Bulk density
1	U9	1327–1328	24.9	16	2.04
2	L1-vertical	3600–3610	24.9	11	2.37
	L1-horizontal	3600–3610	24.9	11	2.37

Fig. 5 **a** SEM Image at Horizontal Flow Width 319 μm and depth interval of 3600–3610 m (L1). This figure shows that the sample is rich in clay content (i.e., 61%) with porosity 11%. Quartz is the second dominant mineral present in the sample. Silica in the SEM image confirms the same. **b** SEM Image at Horizontal Flow Width 91.2 μm and depth interval of 1327–1328 m (U9). This figure shows that the

sample is very rich in clay content (i.e., 75%) with high porosity which is very high as compared to other sample. Illite is the dominant clay mineral here. Quartz and Feldspar are the second dominant mineral present in the sample. Silica in the SEM image confirms the same

indicate the oil window with moderate levels of conversions (Fig. 6b).

Conclusions

This study is aimed to investigate pore structure, porosity and mineralogy of Cambay shale samples at varying depth intervals. It was observed that the shale is highly porous and less permeable in nature. Streaks of siltstone are sandwiched in between Shales which may be contributing to the overall high porosity of the gross shale thickness. This porous shale is also clayey in nature as inferred by FTIR measurements. Illite is the dominant clay mineral at deeper depth contributing approximately 65% to the overall clay content of the dataset. The percentage of carbonate mineral is quite low as compared to the other minerals present, i.e., quartz and feldspar. The clay mineral assemblage

indicates low content of Kaolinite, Smectite, Chlorite and high content of Illite which shows late diagenesis and possible alteration of Smectite and chlorite into Illite. Scanning electron microscopy (SEM) also confirms the presence of these minerals and clayey nature of this shale. If a clay conditioner is used in the drilled zones or sections, advance techniques like hydraulic fracturing may be used to extract oil from this oil-rich shale.

The study if augmented with more data can be used to understand the shale fracability potential in India. Currently, there is no commercial production of shale gas in India. Realising the importance of shale gas and oil for meeting the energy demands of the country and the need to expedite exploration and assessment of domestic reserves, it is the need of the hour to exploit these untapped shale resources. The pilot scale study will be upscaled as and when more cores are cut and procured by the present research team.

Fig. 6 **a** shows the variation of Vitritine reflectance (R_o , %) of the shale samples drilled through the cored interval of 1240–1378 m. This property is the indicator of shale thermal maturity and used in the evaluation of gas shale and shale-oil plays. **b** is plotted to under-

stand the maturity of the drilled shale section. The plot Production index (PI) vs Maturity (measured Vitritine reflectance in oil, % R_o) indicates oil window with moderate levels of conversion

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