PORE TYPE CHARACTERISTICS OF THE HUAI HIN LAT FORMATION IN SAP PHLU BASIN, NORTHEASTERN THAILAND: CONTRIBUTION TO THE UNDERSTANDING OF GAS STORAGE IN FINE-GRAINED ROCKS

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Abstract

In this article, pore characteristics were studied in the exposed fine-grained sediments of the Huai Hin Lat Formation, Sap Phlu Basin in northeastern Thailand. The proxies of a variety of pore types which are important for shale gas reservoir evaluation were presented and classified using field emission scanning electron microscopy. The proxies represent a variety of pore shapes and sizes, dominantly of nano- to micrometer scale, which are dispersed in the rock matrix. The detected pore types can be classified into 10 types, which include organic matter pores, flocculation pores, intergranular pores, pores between crystals, pores at the rim of rigid grains, intercrystalline pores, intraplatelet pores, pores within fossil fragments, pores within a covering of pyrite crystal, and microfracture and microchannel-related pores. Most pores are associated with the organic and inorganic constituents formed by diagenetic alterations and thermal maturity (R0). However, the microfracture and microchannel-related pores were formed by external forces. Overall, the micropores can contribute to good gas storage in the Huai Hin Lat Formation of the Sap Phlu Basin. The microfracture and microchannel- and other microstructure-related pores are also considered to be good migration pathways for generated hydrocarbon.

Keywords: Shale gas, unconventional hydrocarbon, FESEM, fluio-lacustrine facies, micropores, porosity

Introduction

Fine-grained rocks have been under intensive research recently, particularly those containing abundant organic carbon. The studies were designed to gain a more comprehensive understanding of these fine-grained rocks as unconventional petroleum (gas, oil, or natural gas liquids) reservoirs. At present, the unconventional petroleum reservoirs which produce petroleum are also referred to as shale oils and shale gas reservoirs (Fishman et al., 2012) or unconventional...
shale gas reservoirs.

Unconventional shale gas is a long-term energy source for the future (Slatt and O’Brien, 2011). It has become an increasingly important source of natural gas in the United States over the past decade and interest has spread to potential gas-rich shales in Canada, Europe, Asia, and Australia (Sondergeld et al., 2010; Tian et al., 2011). A higher concentration of organic matter can provide excellent original organic richness and high generation potential, resulting in higher gas-in-place volumes (Bowker, 2007) which can be stored in shale source rocks as gas was adsorbed to or within the organic matter (Jarvie et al., 2007) as in-situ reservoirs. Clarkson et al. (2012) suggested that the unconventional tight gas reservoirs are difficult to characterize and routine methods developed for conventional reservoirs are not appropriate for tight shale gas reservoirs as tight gas relates only to permeability. The non-routine methods to characterize the permeability heterogeneity and pore structures of the tight gas reservoirs are applied to an unconventional shale gas study. Therefore, significant advances in understanding shale depositional and diagenetic processes, macroscale to microscale sedimentary structures, and stacking patterns of different lithofacies in sequence to parasequence scales have been achieved (Slatt and O’Brien, 2011).

A recent hypothesis suggests that porosity is created within kerogen as a result of the volume change during and after petroleum generation (Jarvie et al., 2007; Fishman et al., 2012). Theses secondary pores can store the generated petroleum (Curtis et al., 2012). The pores observed in the organic matter were also the result of expelled oil and gas. Fewer nanopores of organic matter indicate a lower thermal maturity and higher secondary pores show increasing thermal maturity (Curtis et al., 2012). Therefore, organic porosity can be considered as an important storage for a shale gas system. Diagenetic processes and mineral phase changes can also contribute to micro-fissures. For an understanding of the storage of gas molecules through shales, a study of organic matter pores including the pores from other processes is recommended.

The purpose of this investigation was to establish the porosity characteristics for the Huai Hin Lat Formation and their hydrocarbon potential. The results of the study can be summarized as following: 1) characterization and classification of the pore types that exist in the Huai Hin Lat Formation, 2) comparison of the characteristics of the pores observed in the Huai Hin Lat Formation, and 3) evaluation of their potential for gas storage and migration pathways.

**Studied area and Stratigraphy of the Ban Nong Sai Measured Section**

The studied area, a part of the Sap Phlu Basin, is in the western margin of the Khorat Plateau covering part of Pak Chong district, Nakhon Ratchasima province and the nearby Khao Yai National Park. It belongs to a part of the Dong Phraya Yen range, representing a portion of the Indosinian fold belt, which is a southward continuation of the Pak Lay zone of Laos. The Ban Nong Sai section is located at 47P 1619790 N and 785346 E between the village of Nong Sai and Khlong Muang (rural road No. 2048), Nong Sarai sub-district, Pak Chong district and is represented on the topographic map (Figure 1). The result of the collision and fusion of 2 principle continental blocks: Sibumasu (also called Shan-Thai) (Chonglakmani, 2011) in the west and Indochina in the east was probably responsible for the strongest unconformity or the Indosinian I event in northeastern Thailand (Booth and Sattayarak, 2011). The subsequent regional uplifting and conjugated shear faulting brought about a number of narrow and elongated basins. These fluvo-lacustrine Late Triassic basins rest unconformably on the Permian and older rocks and commonly are floored by basal conglomerate and/or volcanic rocks.

Generally, the completed sequence of the Huai Hin Lat Formation is composed of: 1) thick sequences of basal siliciclastics resting unconformably on top of the Permian chert and fossiliferous limestone beds, changing laterally to fluvio-lacustrine sands with interfingering lacustrine shale, 2) dark gray lacustrine shale and argillaceous limestone accumulated as the transgressive depositional cycle, and 3) yellowish brown fluvial sediments at the top.
Figure 1. The geologic map showing location of Ban Nong Sai measured section.
The exposures of the Huai Hin Lat Formation in the Sap Phlu Basin containing well-preserved *estheria* faunas and leaf fossil floras in marlstone (Department of Mineral Resources, 1999, 2007) have been reported in both geological maps of Thailand and Nakhon Ratchasima province. The Huai Hin Lat Formation of the Ban Nong Sai section in Nong Sarai sub-district, Pak Chong district area has been the subject of earlier stratigraphic studies. The section is approximately 14 m thick and consists chiefly of dark black calcareous shale, greenish grey to black calcareous mudstone, grey marlstone, and grey limestone. These characteristics can be correlated with the Dat Yai Member of the Ban Dat Fa section (Chonglakmani and Sattayarak, 1978) of the Na Pho Song Basin in the northwestern Khorat Plateau of Nam Nao district, Petchabun province. Some beds of the dark black calcareous shale and greenish grey to black calcareous mudstone contain well-preserved invertebrate fossils. The most common fossils found in these beds are *Estheria* sp. which is the chief characteristic of the shale beds of the Huai Hin Lat Formation. Haile (1973) also reported the occurrence of microflora in the gray shale unit near the Power House of the Nam Phrom Dam in Chaiyaphum province where 7 species of pollens and spores had been reported.

**Field Methods and Sampling Strategy**

Most natural outcrops of the organically rich fine-grained rocks are normally too weathered for reliable analysis. However, the exposures of the Huai Hin Lat Formation in Ban Nong Sai, Sap Phlu Basin are newly excavated for agriculture (Figure 2(a-b) and are, therefore, suitable for detailed investigation (Figure 3). The samples are fresh and were taken from all beds of the measured Ban Nong Sai section of which the sampling locations are in the middle of each bed (Figure 3). The organic geochemical, mineralogical, and microscopic analyses were performed on unweathered samples (Figure 2(c)) were collected from outcrops for assessment of the unconventional shale gas potential of the Huai Hin Lat Formation in the Sap Phlu Basin.

![Figure 2. Rock exposure of the measured Ban Nong Sai section; (A) Lower and middle parts, (B) Middle and upper parts, and (C) Unweathered sample of the measured Ban Nong Sai section.](image)
Figure 3. Lithologic column of Ban Nong Sai section (Sap Phlu Basin) in correlation with Dat Fa section, after Chonglakmani and Sattayarak, 1978.
Organic Geochemical and Mineralogical Analysis

The total organic carbon (TOC) was measured by liquiTOC (Elementar Analysensysteme GmbH, Hanau, Germany) at the State Key Laboratory of Biogeology and Environmental Geology of the Ministry of Education, China University of Geosciences (Wuhan). Samples were treated with dilute hydrochloric acid in pure oxygen and burned under static conditions (960-970°C). The samples with the organic carbon were oxidized to generate carbon dioxide which was measured and calculated to the TOC. The thermal maturity (Ro) was measured by reflectance microscope at the Petroleum Exploration and Development Research Institute of the Jianghan Oilfield Company (SINOPEC), Wuhan city, Hubei province, China.

Mineralogic and clay fractions data were measured with an X’Pert PRO Dy 2198 (PANalytical B.V., Almelo, Netherlands) based on the X-ray diffraction (XRD) method at the State Key Laboratory of Geological Processes and Mineral Resources, China University of Geosciences (Wuhan). Firstly, the samples were pulverized to powdered particles, then placed in aluminum holders, and scanned by XRD using a D/max-3B diffractometer (Rigaku Corp., Tokyo, Japan). XRD measurements were scanned from 2° to 32°2Θ at 0.2° min-1 using CuKα radiation and a graphite monochromator. For clay fraction analysis, a portion of each sample was suspended in distilled water after particle separation by ultrasonic disaggregation. The particles <2 μm size fraction were recovered by ultracentrifugation and air-dried prior to making oriented slides by standard smear techniques.

Results

Organic Geochemical Analysis

Aplin and Macquaker (2011) described mudstones (fine-grained rocks) as an important source rock and considerable effort has been expended in investigating the controls on the preservation of organic carbon in these rocks. The TOC is very high through the studied section, ranging from 1.9 to 7.1%, with an average of 4.9% (Figure 4). The amount of organic carbon in sediments depends on the primary productivity in the photic zone. High contents are commonly associated with: (1) high primary organic matter productivity, (2) shallow to moderate water columns, and (3) restricted biological stirring (Bohacs, 1998). High rates of organic matter preservation are associated with a reducing condition which is indicated by elevated trace elements such as molybdenum, vanadium, and uranium.

The sediments of the studied section are mature, as indicated by the Ro ranging from 0.82 to 1.04 with an average of 0.92% (Figure 4). Tian et al. (2011) showed that the pore sizes are enlarged during thermal maturation. The micropores are abundant, enlarged, and morphologically changed (striped, elliptical, and connected), as shown in the FESEM images. They are caused by conversion of kerogen to hydrocarbons (Loucks et al., 2009).

Mineralogical Analysis

The detrital minerals (zeolite, quartz, feldspar, calcite, and dolomite) and authigenic minerals (clay minerals: smectite, chlorite, and illite) were measured for assessment of pore characteristics.

Clay and calcite are the dominant minerals present in the formation, as shown in Table 1. The clay and calcite contents range from 20 - 65 wt.% (average 42.61 wt.%) and 13 - 43 wt.% (average 27.91 wt.%), respectively. In 100 wt.%
clay, chlorite and illite are the dominant fractions (average 83.91 and 12.39 wt.%, respectively). The other minerals consisting of quartz, dolomite, and feldspar average 8.96 wt.%, 5.52 wt.%, and 13.13 wt.%, respectively. The general increasing in weight percent of clay with maturity is accompanied by a general decrease in weight percent of K-feldspar (Fishman et al., 2012).

**Microscopic Analysis (Pore Characteristics)**

The micropores in the fine-grained rocks of calcareous shales, calcareous mudstones, marlstones, and argillaceous limestone of the Huai Hin Lat Formation in the Sap Phlu Basin are of various types and sizes (Figure 5). Pore sizes less than 1 μm across are referred to as nanopores (nanometer-scaled), whereas those that measure a few microns are micropores (Loucks et al., 2012; Fishman et al., 2012).

The FESEM technique cannot quantify porosity because of the lack of any direct porosity measurements. Nevertheless, it can show pore characteristics and identify the pore types, as follows:

1. **Organic Matter Pores**

   Organic matter pores are present in the organic matter particles of the rock samples. They are natural and are different from the artificial pores which have sharp boundaries.

   The pores in the organic matter are commonly isolated, round to oval, rectangular, elongate, and irregular in shape and size (Figure 6(a)). They are commonly oval to elongate and are a few microns to 2.5 μm in length and commonly subparallel to parallel. The rocks of the studied section are very high in TOC, therefore they may constitute the large volume of organic pores in the strata of the Huai Hin Lat Formation.

2. **Interparticle Pores**

   Interparticle pores occur between grains. The origin of these pores is varied and their geometries differ significantly as a function of both primary pore preservation and diagenetic alteration. The interparticle pores are abundant in young or shallow buried sediments, commonly well connected and forming effective (permeable) pore networks. Four interparticle pore types can

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**Figure 4.** Thermal maturation (Ro), total organic carbon (TOC), and mineral trend for Ban Nong Sai section.
be distinguished in the studied section. These are pores produced by flocculation, intergranular pores, pores at the rim of rigid grains, and pores between crystals.

2.1. Flocculation pores

The floccules are clumps of electrostatically charged clay flakes which sink downward. The floccule can form a cardhouse structure of individual edge-face- or edge-edge oriented flakes and/or domains of face-face-oriented flakes (Slatt and O’Brien, 2011). The interparticle pores of the studied samples are commonly opened or gapped pores of an incomplete cardhouse structure (Figure 6(b)). The pores range from approximately 50-90 nm across in size and up to 2 μm in length. Most pores are arranged in nearly parallel alignment and show the imperfect or unclear cardhouse structures of oriented clay flakes.

2.2. Intergranular pores

Intergranular pores are the matrix-related pore types or interparticle pores between grains of rock matrix that are varied in shape (Figure 6(c)). Besides, the pores may be formed by dissolution or diagenetic processes because of their round to oval, elongate, cubic, and irregular shape. The pore sizes range from 10-160 nm across but some are about 0.25 - 1 μm.

2.3. Interparticle pores at the rim of rigid grains

These interparticle pores occur between the edges or rim of rigid grains or crystals and the rock matrix. They may be formed by the shrinkage of different minerals, for example,

### Table 1. Mineralogic and clay fraction XRD data of Ban Nong Sai section

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**Remarks:** pyrite was not shown in list (detected < 1 wt.%) and zeol*, feld*, calc*, dol*, smec*, and chlo* are zeolite, feldspar, calcite, dolomite, smectite, and chlorite, respectively.
the pyrite crystals in the rock matrix. Most are approximately 570 nm in width and the length depends on the dimension of the cubic-forming pyrites. In addition, this pore type also probably appears between the rim of the rock matrix and rigid quartz minerals (Figure 6(d)).

2.4. Interparticle pores between crystals

This interparticle pore type is associated with the characteristics of crystal-forming minerals. The pores occur in spaces between the crystals of minerals (Figure 6(e)). The shapes are varied and depend on the types and sizes of minerals. They are round to polygonal, elongate, and irregular in shape. The pore sizes range from 20-250 nm and up to 1.5 μm in width and length.

3. Intraparticle pores

Intraparticle pores occur within detrital grains or crystals, clumps of crystal, and mineral particles. Some of these pores are primary in origin but most are probably diagenetic. Four common types of these pores are recognized by their origin such as pyrite framboinds, clay particles, fossils, and covering pyrite minerals.

3.1. Intercrystalline pores

Intercrystalline pores are intraparticle pores which occur within frambooidal pyrite (Figure 6(f)). These porous grains may have internal pores between microcrystals of secondary origin and are usually dispersed within the shale matrix (Slatt and O’Brien, 2011). The micro-pellet pyrite has internal pores ranging in size from approximately 20-100 nm across. The frambooidal pyrite contains pore of a few microns to 1.4 μm. These pore sizes vary from regular to irregular shape forms and are mainly connected as a network of larger pores.

3.2. Intraplatelet pores

Intraplatelet pores occur within clay
aggregates/particles. The authigenic clay platelets are generally linear and parallel to one another (Loucks et al., 2012). These pores are commonly slot-like or interspersed between curved clay mineral plates (Figure 7(a)) and range in size from a few microns to 2.2 µm across and up to 10 µm in length of the striped curve between these flakes. The other clay minerals were flaky, irregular lath, and silk thread-like aggregates and the pores are generally a few microns across and up to 1 µm at least in length. Moreover, the mineralogical XRD data show a high percentage clay fraction of 20-65 wt.% (average 42.61 wt.%). The clay is mainly chlorite and illite which control the observed pore patterns.

3.3. Intraparticle pores within fossil fragments

Intraparticle pores related to fossil fragments occur within fossil fragments or body-cavities of fauna and flora (Loucks et al., 2012). Intraparticle pores within fossil fragments occur between the outer covering parts of the fossils and are approximately 1.5 µm across. The interior chamber of a few microns in size is commonly opened (Figure 7(b)).

3.4. Intraparticle pores within covering pyrite crystals/grains

This pore type occurs within crystals or grains of pyrite. These pores are likely formed by the primary origin of individual pyrite crystals.
The voids between the inner and outer coverings of crystals (Figure 7(c)) have a range from approximately 300-400 nm in width and 2-2.5 μm in length.

4. Microfracture and microchannel-related pores

This pore type consists chiefly of channels occurring within the rock matrix. It includes microchannels and fractures (Figure 7(d-e)) which were formed by tectonic forces. These features are subparallel to the bedding plane. They were likely the original fractures preserved in the rock matrix, not artifacts produced by fracturing, handling, and preparation of the samples.

This type is very important in shale gas systems that are not completely occluded by cement (Loucks et al., 2012). These fractures have been proposed as storage and transport mechanisms for hydrocarbon (Dewhurst et al., 1999) and have a significant contribution to hydrocarbon production (Loucks et al., 2012). The fractures in the Huai Hin Lat Formation are approximately 220 nm in width and the length is arranged in long strips. Although the mineralogical XRD data show a high percentage of carbonate minerals of 13-43 wt.% (average 27.91 wt.%) which cause the overgrowth of calcite in the pores of channels/fractures, the pores are still observed and it is still possible for...
the Huai Hin Lat shales to be the pathways for the migration of gas and liquid.

Identification of pore types and their variability as a function of thermal maturity are crucial for understanding the overall porosity in the formation. The ability to produce, store, and transmit hydrocarbons of self-sourced reservoirs like gas shales are dependent on these characteristics. The pores within fossil fragments and organic matter are larger and more abundant in argillaceous limestone and marlstone. Pores associated with pyrite are more abundant in calcareous shale and calcareous mudstone. Moreover, calcareous shale and calcareous mudstone commonly contain larger, longer, and microfracture and microchannel-related pores which indicate that both types of rocks are more brittle.

Discussion

Muds comprise a variable mixture of clay minerals (illite, smectite, chlorite, and, to a lesser extent, kaolinite), quartz, feldspars, carbonates (calcite and dolomite), zeolite, and sulfides (pyrite was detected < 1 wt.% and does not appear on the list) including amorphous material and organic matter (Aplin and Macquaker, 2011). The micropores, mainly of organic matter pores, interparticle pores, and intraparticle pores, are generated as the result of burial diagenesis. In addition, microfracture- and microchannel-related pores are also present. These pores are an important factor for successful petroleum production in both conventional and unconventional resources.

This document is the first study of pore types, pore size ranges, and pore distribution in the Huai Hin Lat Formation of the Sap Phlu Basin. It is an important step towards understanding where gas is stored and how it might migrate from the fine-grained sediments into the induced fractures which enable production (Loucks et al., 2009). For storage capacity, only pores are needed, whereas for production the pores should be interconnected (networked) so that the hydrocarbons can flow through. We could classify the pore types into 2 groups, the group with gas storage capacity as a function of organic matter pores and inter- and intraparticle pores, and the group with permeability pathways as a function of microfracture- and microchannel-related pores.

Gas Storage Capacity

Pores occur during burial (Emmanuel and Day-Stirrat, 2012) or post-deposition (Pitman et al., 2001). They may have been altered by the process of diagentic alteration or thermal maturation ($R_o$). The pores may be connected and may have enlarged extensively.

1. **Organic matter pores**

   Horodyski (1980) described micro-algal mats which grew in-situ on the seafloor. These algal mats are also found in fluvio-lacustrine facies of the Huai Hin Lat Formation. Their organic matters are probably similar to algal mats which were described by others in modern and ancient lacustrine. The research over several decades has shown that the amounts of organic carbon buried in the geologic record were dependent on the primary productivity (Aplin and Macquaker, 2011) and redox condition. The organic-rich samples in this study are analyzed, as shown in Table 1. The values of the TOC and $R_o$ range from approximately 1.9% to 7.1% and 0.8 to 1.0, respectively. Therefore, the rocks of the studied section are good to excellent in organic richness and the micropores have probably increased because of evaluated thermal maturity.

   The decomposition of organic matter by thermal maturation leads to the formation of hydrocarbons and intraparticle organic nanopores in the organic matters (Loucks et al., 2009). These pores correspond to the increase of temperature during burial which starts at 0.6% $R_o$ of low thermal maturity and 10% by weight of the conversion (Peters, 1986). The observed micropores are abundant which conforms to the high TOC in the samples. Moreover, in the higher thermal maturity section, they may have developed larger gas storage sites.

2. **Inorganic matter**

   The generated micropores in the formation are dependent on the mechanical and chemical stability of minerals. This inorganic matter is associated with both inter- and intraparticle pores which occur during syn- and post-depositional processes.
Phyllosilicate clays are associated with feldspar by a chemically altered process. Pytte and Reynolds (1988) suggested that clays are easily ductile, compact, and deformed depending on the increase in temperature which is associated with deep burial ($R_0$). They are rearranged parallel to subparallel as clay platelets or clay sheet-cleavages. The pores are arranged in narrow, elongate, and parallel to subparallel alignments. Therefore, interplatelet pores within clay aggregates including pores produced by flocculation are abundant and well preserved in the rock matrix and they provide a high potential for storage capacity.

Carbonate (Figure 7(f)) and feldspar minerals are easy to dissolve and deform in diagenetic processes. These pores are generated by extreme dissolution at high thermal maturity and can be good storage sites. In the case of connecting micropores, they will form porous networks and provide higher gas storage capacity.

Pyrite and silica are referred to as altered sulfide and quartz minerals, as shown in Table 1. Fishman et al. (2012) suggested that pyrite occurs in syn-depositional to early post-depositional processes. The syn- and post-depositional bacterial decomposition of the mats probably resulted in the formation of anaerobic conditions which in turn led to precipitation of pyrite framboids. The pores were generated by diagenesis of pyrite minerals. They are intergranular pores, pores between crystals, pore at the rim of a rigid grain, intercrystalline pores, and pores within the covering pyrite which are abundant in the rock matrix. These pores can probably store hydrocarbon but not contribute much to permeability. However, in the case of fossil fragment pores, they are probably connected and aligned along the beds which may provide higher permeability.

**Permeability Pathways**

Montgomery et al. (2005) described how methane molecules were stored and adsorbed by attachment to the surfaces of organic and mineral material and non-adsorbed (absorbed and free) in pore spaces. If these pores are touching and interconnected, gas then moves to other places in the reservoir along these permeability pathways. Usually in the subsurface, existing pores are filled with fluids due to the depositional environment; only later formed and isolated pores might be not filled with such fluids. In fact, all the pore characteristics cannot provide the permeability pathways unless the microfracture and microchannel related-pores are interconnected.

Abundant microfracture- and microchannel-related pores are dependent on overall mineral compositions, for example, they are more abundant in siliceous beds than clay-rich beds. They provide good potential for permeability/migration pathways for the Triassic Huai Hin Lat reservoir. Although the elongate micropores are small, they are sufficient to allow gas to migrate through their fractures to accumulate in suitable zones. The micropores along the rim of rigid grains of individual cubical pyrite are interconnected through the rock matrix forming the microfractures and microchannels. The samples with more interconnection will provide more permeability.

**Conclusions**

Most pores, such as mineral and fossil fragment pores, are mainly associated with their depositional processes. The inter- and intraparticle pores are associated with precipitated minerals but the interplatelet pores of aggregated clays and the organic matter pores are related to the thermal maturity level. The microfracture- and microchannel-related pores depend on the overall mineral compositions and structure of the reservoir formation.

This research is needed to fully define the interrelationships between pore formation, distribution, and fluid flows in reservoirs. In other words, we need to understand the relationships between pore types and the quantity of various minerals in a formation and the processes forming the micropores. The pores caused by flocculation, organic matter alteration, and inter- and intraparticle processes are small and narrow, but they are sufficient to store the hydrocarbon. Abundant micropores within organic matter are generated by high thermal maturity and an increasing formation temperature. The other micropores of interplatelet clay aggregates are
abundant in both the deeply buried formation and the clay-rich formation. These pores have a high potential to capture and store hydrocarbon. Moreover, the typical elongated microfractures and microchannels also provide the storage sites and the permeability pathways for the generated gas.

Finally, FESEM micrograph viewings are useful for evaluation of complicated pore networks of storage or reservoir rocks.

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