Characteristics of Shale Oil Potential in the Late Cretaceous Qingshankou Formation in the Gulong Sag, Northern Songliao Basin, Northeast China

Thandar Aung[1], Jiang Zai Xing[2]

Abstract

The Songliao Basin is a large-scale petroliferous basin in China with resources, in place and recoverable resources of 82.9 BBQ, respectively (Ministry of Land and resources of China). The lacustrine shale of the Late Cretaceous Qingshankou Formation is the principal prospective unconventional target lithology, acting as source, reservoir, and seal. The Qingshankou Formation consists of gray, dark-gray and black mudstone interbedded with oil shale, gray sandstone and siltstone. Previous studies have shown that Cretaceous Qingshankou Formation (K_2qn) has favorable geological conditions for the formation of shale oil. Thus, shale oil in the Qingshankou Formation represents a promising and practical replacement resource for conventional oil. In the present study, core observation, sample tests, and the analysis of well logs were applied to study the geochemical and reservoir characteristic of shale identify shale oil beds and classify favorable exploration areas of shale oil from the Cretaceous Qingshankou Formation. Shale oil includes high organic matter content from the first member of the Cretaceous Qingshankou Formation (K_2qn^1) with average total organic carbon (TOC) content exceeding 2%. The organic matter is derived mainly from lower aquatic organisms in a reducing brankish to fresh water environment, resulting in mostly type I kerogen. This formation consists of highly variable near shore shallow lacustrine and semi deep to deep lacustrine facies. Among three of the Formations, the first member is the most promising Formation to get the hydrocarbon generation potential. Also Oil shale potentials have been identified in the Pliocene–Upper Miocene of the Onshore Thanintharyi, Myeik and Dawei region in Myanmar.

Keywords: Songliao Basin; Gulong Sag; Cretaceous Qingshankou Formation; Shale oil

Introduction

The Songliao Basin, covering an area of 260,000km^2(100,000 mi^2), is one of the largest continental Cretaceous Basins in the world and hosts the largest oil field in China, abundant conventional hydrocarbon resources were also found in this basin (Xu *et al*., 2015). The Upper Cretaceous rock units Qingshankou and Nenjing shale are the most prolific source rock in the Songliao Basin (Bechtel *et al*., 2012). Although commonly called "shale," these organic mudstone (Aplin and Macquaker,2011) are specifically defined as fine-grained sedimentary rocks with elevated total organic carbon (TOC) that contain clay- sized particles (<4mm), with variable amounts of silt- sized floating grains (up to 62.5mm) of biogenic and detrital origin. It is subdivided into three members (K_2qn^1, K_2qn^2 and K_2qn^3) based on lithology. The first member of the Qingshankou Formation (K_2qn^1), one of the favorable source rocks in the Songliao Basin, is widely distributed across the basin and is only disrupted on the western edge of the basin. It is up to 500 m (1600 ft) thick, it developed during a rapid, large-scale lake transgression. The second member of the Qingshankou Formation (K_2qn^2) was deposited during a period of lake regression. The Gulong sag is located in the western part of the central depression, deposition and subsidence during the deposition of the Qingshankou Fomation. This formation consists of highly variable near shore shallow lacustrine and semi-deep to deep lacustrine facies. Contributing to the worldwide growth in production of unconventional oil and gas, tight oil has been discovered

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in the sandstones of the second and third member of the Qingshankou Formation (K_2qn^{2+3}) in this Basin (Shi, 2015). The Qingshankou Formation contains shale oil layers with high TOC and high oil yield near the base of its first member (K_2qn^1; Feng *et al* 2011). For this reason, over the past two decades, high resolution studies of the potential for hydrocarbon generation and the reconstruction of paleoenvironmental changes have been undertaken (Zhou and Littke, 1999; Jia *et al*., 2013b; Xu *et al*., 2015). In recent years, excellent examples of shale oil potential from organic matter-rich shale of the first member of the Qingshankou Formation have caused researchers to focus on the reservoir characteristics of this lacustrine deep- water sedimentary sequence (Liu *et al*., 2014a; Shi *et al*., 2015). The transformation belt lies in the depocentre of the Songliao Basin where there was a deep-water environment and good source rock deposition. The tectonic transformation aided hydrocarbon migration from the belt to the up-dip reservoirs (decreasing belt and increasing belt). The basement rock consists of Paleozoic Metamorphic, Volcanic, and magmatic rocks. The Cretaceous Qingshankou Formation (K_2qn) was formed during the subsidence period when the lacustrine facies were developed in the Central Depression of the basin.

Regional Geologic Setting

The Gulong sag is located in the west of the Central Depression of the Songliao Basin in Heilongjiang and Jiling Provinces, NE China, a large north- northeast trending Mesozoic-Cenozoic Continental Lacustrine Basin (Fig. 1). Based on the sedimentary history, fault block movement, volcanic activities, and thermal evolution can be divided into four main stages: extensional rift with thermal uplift, syn- rift, subsidence, and structural inversion (Gao, 1994).

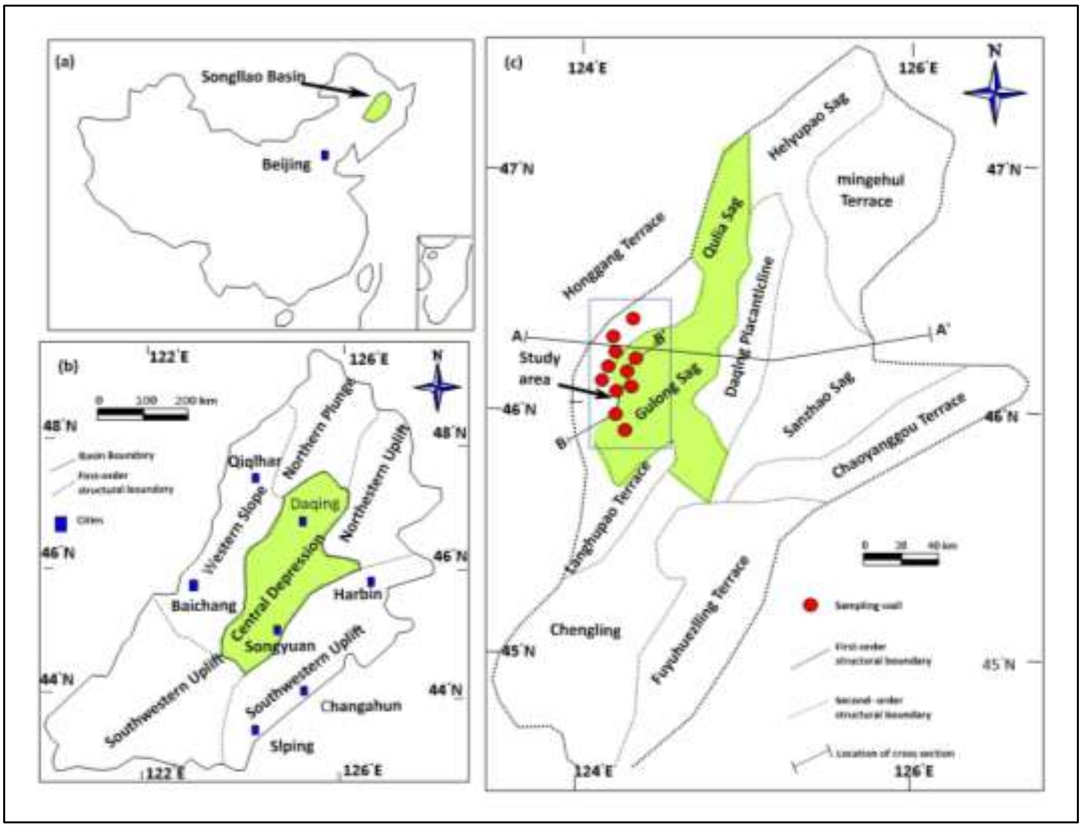


Figure (1) Generalized map showing the location of the Songliao Basin. (A) Central depression, (B) Study area (Gulong Sag), and (C) Location of core Section (after Feng *et al*., 2010).

During the Formation of the second (K_2qn^2) and third (K_2qn^3) units of the K_2qn, the entire basin experienced regression and the lake area decreased. The lake basin gradually reduces in size, and the Songliao Basin was inverted by folding and uplift (Wei *et al*., 2010). The Cretaceous Qingshankou Formation (K_2qn) was formed during the subsidence period when the lacustrine facies were developed in the Central depression of the basin. The Qingshankou Formation is dominated by lacustrine sediments formed under warm and humid conditions. Large scale water- transgression occur in the sedimentary period of the first member of the Qingshankou Formation (K_2qn^1), so the depositional environmental was semi-deep to deep lake anoxic and during this period. The first member of the Qingshankou Formation (K_2qn^1) deep water sediment in the Gulong Sag provide a material basis for establishment of fine-grained sedimentary cycles. The Qingshankou Formation (K_2qn) was deposited in a moderately deep lake setting influenced by periodic marine incursion events during a period of global sea-level rise (Liu *et al*., 1993; Huang *et al*., 2013).

Material and Methods

Thin-section analysis

The Cretaceous Qingshankou shale based on the samples were collect from 8 wells to prepare about thin sections. Thin section analysis was done using the CARL ZEISS AXIO SCOPE A1 and Fluorescence microscope to found mineral, sorting, grain size and distribution, and the presence of fossil and organic matter.

Whole-rock mineralogical analysis

Whole- rock mineralogical analysis was takeover by the Research Institute of Petroleum Exploration and Development of the Petro China Daqing Oil Field. The X-ray diffraction (XRD) analysis was carried out to obtained semi quantitative data using a Bruker D8 goniometer. Samples were dried at a temperature of than 60 °C for 2 days and then ground to <40 µm using an agate pestle and mortar. The sample powders were scanned from 3° to 70° with a counting step of 0.02° and a rate of 2° per minute. Computer analyses of X-ray diffractograms, which confirmed the relationship between the mineral content and the intensity of the diffraction peak, provided semi-quantitative relative abundances (in weight percent) of the various minerals.

Total organic carbon (TOC) determination is a generally relevant method for characterizing fine-grained source rocks. The method of determining TOC in sediments involves the combustion (oxidation) of the rock sample to convert the total organic carbon into CO_2 and CO at high temperatures (Charles and Simmons, 1986). Samples were grounded to <200 µm using an agate pestle and mortar. Sample powders of less than 1 g were weighed and treated with excessive diluted hydrochloric acid (HCl). The residue samples washed with distilled water and dried in a drying oven were measured by a LECO CS-200 carbon analyzer at the Research Institute of Petroleum Exploration and Development of the Petro China Daqing Oilfield.

Scanning Electron Microscope (FESEM) and Energy Dispersive X-ray Spectroscopy (EDS) Analysis

FESEM and EDS were carried out using the CARL ZEISS SUPRA 55HKL FESEM. 1cm cube samples were cut from both core slabs and analyzed by FESEM to investigate their molecular surface structures between 2Pa and 133Pa pressure ranges, and probe currents ranging 4pA to 10nA. The samples were additional analyzed by EDS for their elemental and chemical characterization at specific points. The SU8010 Field Emission Scanning Electronic

Microscope (made by Hitachi, Japan) at the Center of Analysis and Testing, China Petroleum of University, and Quanta 200F Field Emission Scanning Electronic Microscope (made by FEI, Hillsboro, USA) at the Microstructure Laboratory for Energy Materials, China University of Petroleum (Beijing) were used in this study to obtain high resolution images .

Pyrolysis Analysis

Pyrolysis was performed using a Rock- Eval 6 analyzer to get the amount of free hydrocarbon in the sample (S_1), the amount of pyrolyzate released from kerogen (S_2), and the temperature of maximum hydrocarbon generation (T max).The S_2 was normalized by the TOC value to give the hydrogen index (HI). Serving as a maturity indicator, T_{max} is defined as the value of the S_2 peak (Landford and Blanc- Valleron, 1990). Finally, the production index (PI) and oil saturation index (OSI) parameters were determined for each sample: PI= S_1/ (S_1+ S_2); OSI =S_1/TOC× 100 (Jarvie, 2012).

Result and Discussion

Petrological charateristics

According to sedimentary genesis of shale oil, petrological characteristics are fundamental method to identify organic matter content, mineral composition and sedimentary structure.

Geochemical characteristics

The Qingshankou Formation mudstone in the Gulong Sag has TOC of up to 3.6%, on average 1.7% and mainly between 1.2% and 2.5%, and the hydrocarbon generation potential (S_1+S_2) of 4.7- 11.2 mg/g, on average 7.9 mg/g. The mudstone with organic matter dominated by Type I and Type II_1 kerogen, is typical oil-prone lacustrine organic-rich mudstone. With R_o value mainly ranging between 0.70% and 1.13%, 0.89% on average, the mudstone is in the mature stage overall. The relationship between the original hydrogen index (HI_o) and TOC of immature-low mature shale in the study area indicates that when TOC <1%, HI_o is very low, when TOC is 1%- 2%, HI_o has a positive correlation with TOC, when TOC >2%, HI_o is stablized and does not increase with the increase of TOC (Fig. 2). Thus, for the terrestrial organic-rich shale, TOC <1% can be defined as low organic matter content, TOC between 1% and 2% is defined as the medium organic matter content, and TOC >2% is defined as high organic matter content.

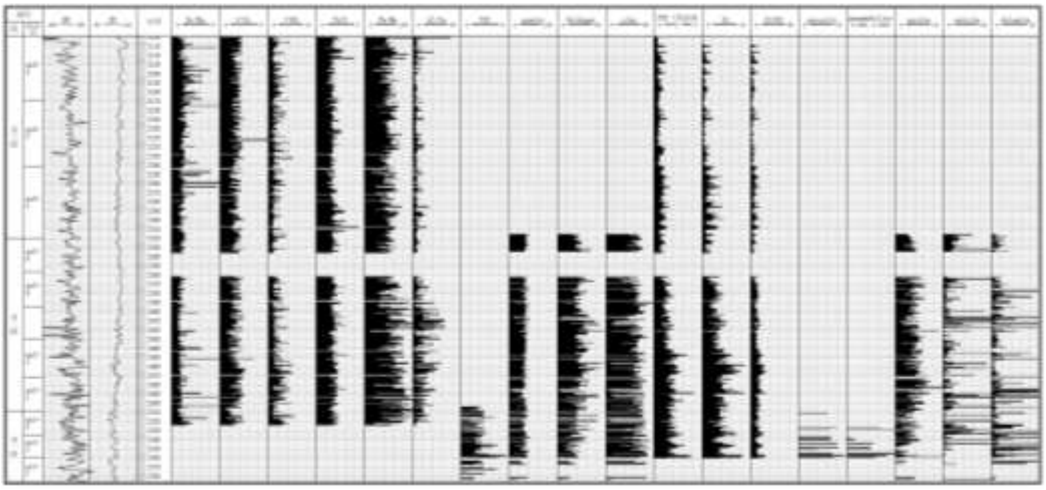


Figure (2) Geochemical variations in the Gulong Sag of the Songliao Basin.

Mineral compositions

The mineral compositions of the Qingshankou Member shale are mainly quartz (14%-71%, on average 37%), plagioclase (7%-41%, on average 21%) and clay minerals (4%-46%, on average of 35%), followed by a small amount of K-feldspar, calcite, dolomite and pyrite (Figs. 2 and 3). Triangle plot whole rock mineral composition of fine-grained rock in the Qingshankou Formation shows the relative proportions of quartz, feldspar, clay and total carbonate minerals (Fig. 4). The clay minerals are dominated by illite, followed by the illite-montmorillonite mixed-layer and kaolinite. The carbonate minerals are concentrated in the Ostracod limestone.

(a) (b) (c)

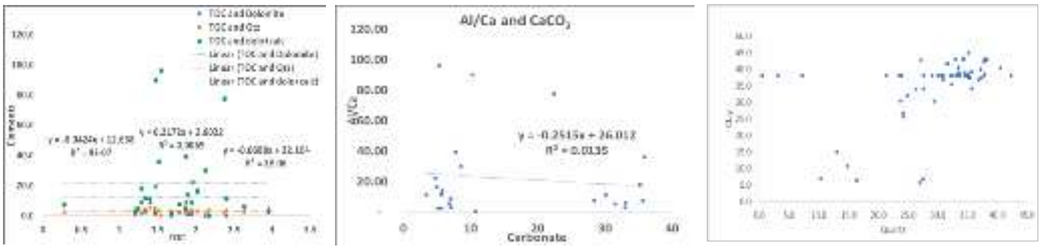


Figure (3) Comparison of (a) TOC and Dolomite, Quartz, CaCO3 (b) Al/Ca and $CaCO_3$ (c) Quartz and Clay mineral.

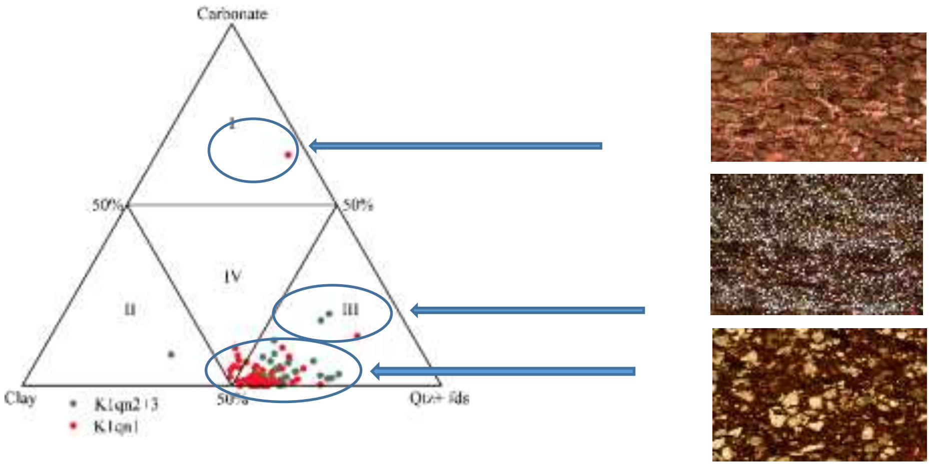
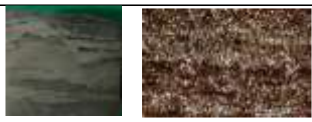
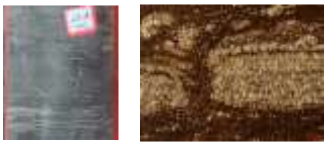
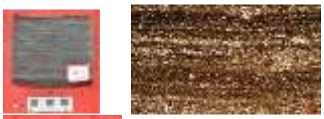
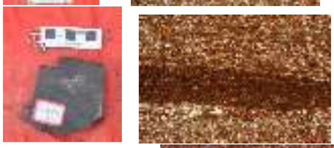
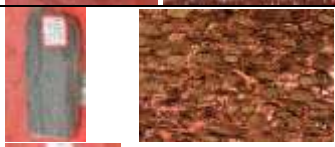
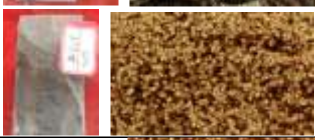
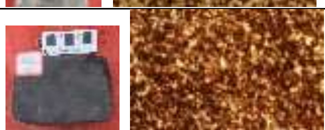


Figure (4) Triangle plot whole rock mineral composition of fine- grained rock in the Qingshankou Formation.

Lithofacies

Most of these studies used the organic matter content, bedding, and mineral composition as the main components in the definition of shale lithofacies. Combinations of these three factors are used to describe the main petrological characteristics of the shale lithofacies, and this then is indicative of a certain sedimentary depositional environment. A combination of detailed core descriptions, microscopic observations, and mineralogical and geochemical analyses allowed the determination of the TOC, fabric, and mineral composition of many samples from 8 wells, the recognition of a total of 10 lithofacies types in the Qingshankou Formationin in the Gulong sag, Songliao Basin (Table 1). It divided into 10 types of lithofacies (1) turbidite siltstone facies; (2) Turbitide shale facies; (3) Semi deep lake shale facies; (4) Deep lake shale facies; (5) Deep lake Clay shale facies; (6) Semi deep lacustrine bioclastic shoal facies; (7) Shallow lake oolitic shoal facies; (8) Deep lake microbial dolomite facies; (9) Deep lake diatom shale facies; (10) Shelly shelly-siltstone Turbidite facies.

Table (1) Characteristics of Lithofacies types in the Qingshankou Formationin in the Gulong sag, Songliao Basin

Materials Source	No	Lithofacies		Core and Slice Characteristic	Sedimenta-ry Structure	TOC	Formation Mechanism
		Facies name	Full name				
Terrigeneous Origin	1	Turbidite Siltstone Facies	Siltstone		Block	1%	Sandy turbidity current
	2	Turbitide Shale Facies	Low TOC massive muddy Shale		Block	<2%	Muddy turbidity current
	3	Semi deep lake Shale Facies	Medium TOC laminated Shale		Laminated	2-3%	Lamina-strata
	4	Deep lake Shale Facies	Low TOC laminated Shale		Laminated	1-2%	Annual release Semi - lake
	5	Deep lake Clay Shale Facies	Medium – high TOC layered Clay rock		Laminated	3-5%	Deep lake
Endogenous Origin	6	Semi deep lacustrine bioclastic shoal Facies	Shelly Limestone		Block	<1%	Semi deep lake with storm
	7	Shallow lake oolitic shoal facies	Grainstone		Block	1%	Shallow lake shoal
	8	Deep lake microbial dolomite Facies	Medium to high TOC Microcrystalline Dolomite		Massive	2-4%	Microbial induation
Siliceous genetic Facies	9	Deep lake diatom Shale Facies	High TOC lamellar Biosiliceous Shell rock		Laminated	4-5%	Diatom Bloom
Mixed source Genetic Facies	10	Shelly-Siltstone Turbidite Facies	Low TOC Shelly - Siltstone mixed fined-grained rock		Block	1%	Under turibidity current condition

Shale Oil Reservoir

The porosity and permeability of organic-rich shale are keys to the evaluation of the potential for economic production of shale oil and gas. The pores within mudstones of the study succession are of various types and sizes (Fig. 5), mainly ranging in size between 150 and 620 nm. In this study, pores are classified into three major types (Loucks *et al*., 2012): inter-particle pores between detrital or authigenic particles, intra-particle pores within detrital grains or crystals, and organic matter pores. Inter-particle pores (Fig. 5.a) are found between particles and crystals such as quartz, feldspar, clay, and carbonate minerals. Intra-particle pores are observed inside particles such as clays (Fig. 5.b), pyrite (Fig. 5.c) and are caused by diagenesis and dissolution. Clay mineral occur with shrinkage joint (Fig. 5.d) due to micro-fracture formed by sedimentation and micro stress. Calcite commonly appears as a cement with rare dissolution pores. Illite minerals always flocculate and behave as aggregates rather than as individual particles, forming an abundance of inter-particle pores between clay platelets and intra-platelet pores within clay aggregates (Fig. 5.e). Inter-particle act as high-permeability pathways through the stratified structure of clay minerals (Slatt and O'Brien, 2011). Organic matter pores are considered to be influenced by organic matter type and thermal maturation (Fig. 5.f) (Ko *et al*., 2016).

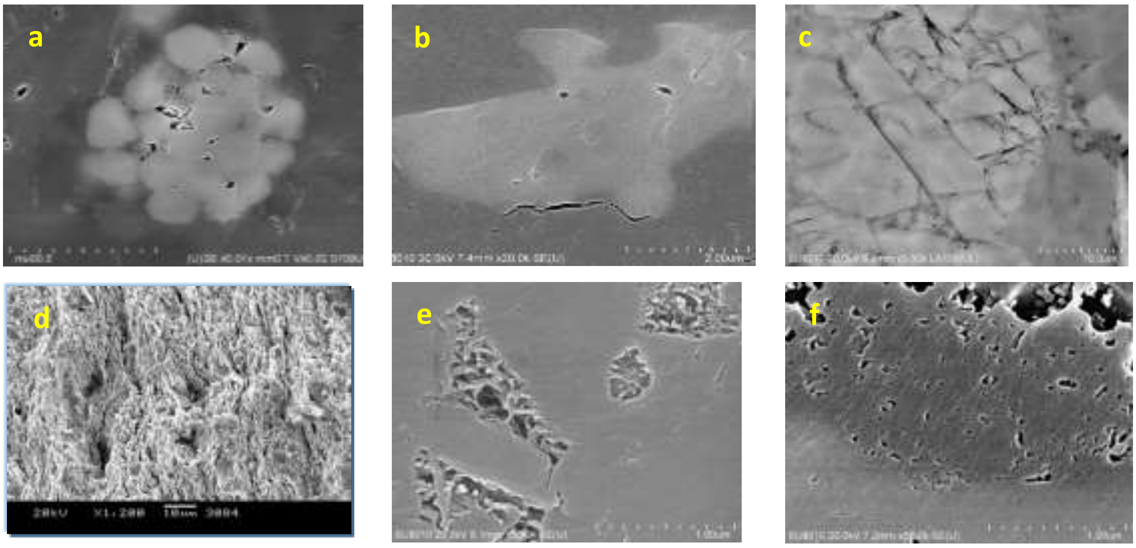


Figure (5) Field emission scanning electron microscopy images of the Qingshankou Formation. (a) Inter-particle pores around pyrite mineral grains; (b) Intra-particle dissolution pore.; (c) micro-fissure; (d) clay mineral with shrinkage joint; (e) inter-particle pore around clay mineral; (f) organic matter pore. BSED=backscattered electron detector; HFW=horizontal frame width; HV=high voltage; det=detector; mag=magnification; WD=working distance.

Shale Oil Potential

Petroleum potential of Songliao basin is substantial. Principal producing oil and gas fields are concentrated in the central and south-eastern parts of the basin. Of these, Daqing oil field of the Daqing central basin high in the Central Depression ranks as one of the world's giant oil fields and has produced an average of more than 1 million barrels of oil per day since 1976, about half of China's daily oil production. Of the ten shale lithofacies discussed above, deep lake clay shale facies and deep lake microbial dolomite facies are proposed as being the most prospective lithofacies for shale oil because of its high generative source-rock potential, high TOC, high content of indigenous hydrocarbons, high mineral brittleness index (percentage content of brittle minerals in the shales, e.g., quartz, feldspar, and carbonate; Lin *et al*., 2016), and high probability of a connecting pore network. Spatial and temporal changes of lithofacies controlled by the stacking pattern within a sequence stratigraphic framework determine the distribution of prospective shale lithofacies in a sedimentary basin. Systemic investigation of the characteristics of lithofacies are shown to be an effective method to allow recognition of prospective regional targets within unconventional petroleum reservoirs. Original oil in-place at Daqing is estimated to be about 21.9 billion barrels, of which 30 percent is likely to be recovered by existing production methods; enhanced recovery processes are currently being considered for use in Daqing Oil Field.

Shale Oil Occurrence in Myanmar

In Myanmar also Oil shales potential have been identified in the Pliocene–Upper Miocene of the Onshore Thanintharyi (Mepale and Theingun-Lahnya areas). Exploration of Shale oil has started in the Mepale Basin (also known as the Htichara Basin) where a 609 m-thick sequence of shale or oil shale is recognized (Gregory, 1923; Cotter, 1924). In the Thein Lenya area at Myeik region, (Rao, 1922) reported the presence of bituminous shales within a shale-sandstone sequence at Maungbwa near to Myeik and similar shales from the Tanintharyi valley in the bordering Dawei region (Racry *et al*., 2015). There is little overburden on these shales and only minor faulting which makes extraction by opencast mining feasible. According to pre-war reports, around 3 billion barrels of oil was estimated to be extractable from the Mepale Basin (Lynn Myint, 2014).Oil-shale exposures were make extensive also reported from the Late Tertiary intramontane Kawkareik (Phalu) basin on the border with Thailand.

Conclusion

The Cretaceous Qingshankou Formation is situated the Songliao Basin of North eastern China. This member was deposited in a semi-deep to deep lacustrine environment during a period when the lake was most extensively developed, forming the principal hydrocarbon source rocks of the Songliao Basin. The Qingshankou Formation mudstone in the Gulong Sag has TOC of up to 3.6%, on average 1.7% and mainly between 1.2% and 2.5%, and the hydrocarbon generation potential (S_1+S_2) of 4.7- 11.2 mg/g, on average 7.9 mg/g. The mudstone with organic matter dominated by Type I and Type II_1 kerogen, is typical oil-prone lacustrine organic-rich mudstone. With R_o value mainly ranging between 0.70% and 1.13%, 0.89% on average, the mudstone is in the mature stage overall. The relationship between the original hydrogen index (HI_o) and TOC of immature-low mature shale in the study area indicates that when TOC <1%, HI_o is very low, when TOC is 1%- 2%, HI_o has a positive correlation with TOC, when TOC >2%, HI_o is stablized and does not increase with the increase of TOC. Thus, for the terrestrial organic-rich shale, TOC <1% can be defined as low organic matter content, TOC between 1% and 2% is defined as the medium organic matter content, and TOC >2% is defined as high organic matter content. The mineral composition is mainly composed of quartz, plagioclase and clay minerals, with little content of K-feldspar, calcite, dolomite and pyrite. Systemic investigation of the characteristics of lithofacies are shown to be an effective method to allow recognition of prospective regional targets within unconventional petroleum reservoirs. In Myanmar also Oil shale potential have been identified in the Pliocene–Upper Miocene of the Onshore Thanintharyi (Mepale and Theingun-Lahnya areas).

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