

Research Article

Storage Properties of the Shale Reservoir of the Lower Keluke Formation in the Shihuigou Area: Implications for the Controlling Factors

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In this study, the characteristics of shale gas reservoirs of the lower part of the Keluke Formation in the Shihuigou area were investigated by using thin sections, scanning electron microscopy (SEM), and isothermal adsorption, combined with geochemical experimental analysis. Consequently, the controlling factors of the adsorption capability and gas content of the studied shale reservoir are explored. The results show that the shale reservoir space of the Keluke Formation is dominated by pores and fractures. The pores are divided into intergranular pores, intragranular pores, and dissolved pores. The pore volume of medium pores is higher than that of micropores, but the specific surface area of the medium pores is lower than that of micropores. The pore size distribution of medium pores has a feature of single peak, while the distribution of micropores is multi-peaked. The results of physical properties and field gas adsorption indicate that the reservoir quality and gas content of the lower part of the Keluke Formation are good. Meanwhile, the total organic content (TOC) has a positive relationship with the methane adsorption. However, the field gas adsorption does not have a clear relationship with TOC and methane adsorption, but a positive correlation with clay mineral content. Therefore, the present study concludes that the natural gas in the shale reservoir of the lower part of the Keluke Formation is predominantly accumulated in form of free gas, and the concentration of free gas is higher than that of the adsorbed gas.

1. Introduction

In response to the increasing depletion and diminishing production of conventional oil and gas resources, the demand for unconventional oil and gas resources is gradually increasing [1–4]. Because of the success of shale gas exploration and development in the United States, the local shale gas wells have been increasing, and the production continues rising. As a result, the shale gas becomes a new exploration target in the field of petroleum geology, including the reservoir mode, reservoir space characteristics, and controlling factors of reservoir quality. In 2007, a pilot test of shale gas exploration and development was conducted in the marine shale formation in the Sichuan basin, where the shale reservoir is developed and rich in organic matters [5]. This pilot test reveals the prologue of shale gas exploration and development in China.

The Shihuigou area of the Delingha Depression, located in the northeast of the Qaidam Basin, is rich in hydrocarbon resources with multiple high-quality hydrocarbon source rocks. The cumulative exploration area has reached 121,000 km² after sixty-year exploration and development. The Keluke Formation, which is marine-to-terrestrial deposition with clastic and carbonate rocks, has been appraised as a potential hydrocarbon source rock [6–8]. There has been significant progress in the study of marine or terrestrial shale gas [9–12]. However, the study on the shale gas of transitional deposition from marine to terrestrial is rare [13]. The present study investigates the characteristics of the shale gas reservoir of the Keluke Formation in the Shihuigou area and then discusses the controlling factors of the shale gas adsorption. Consequently, we provide a reference for future in-depth research and evaluation of the shale gas accumulated in transitional facies.

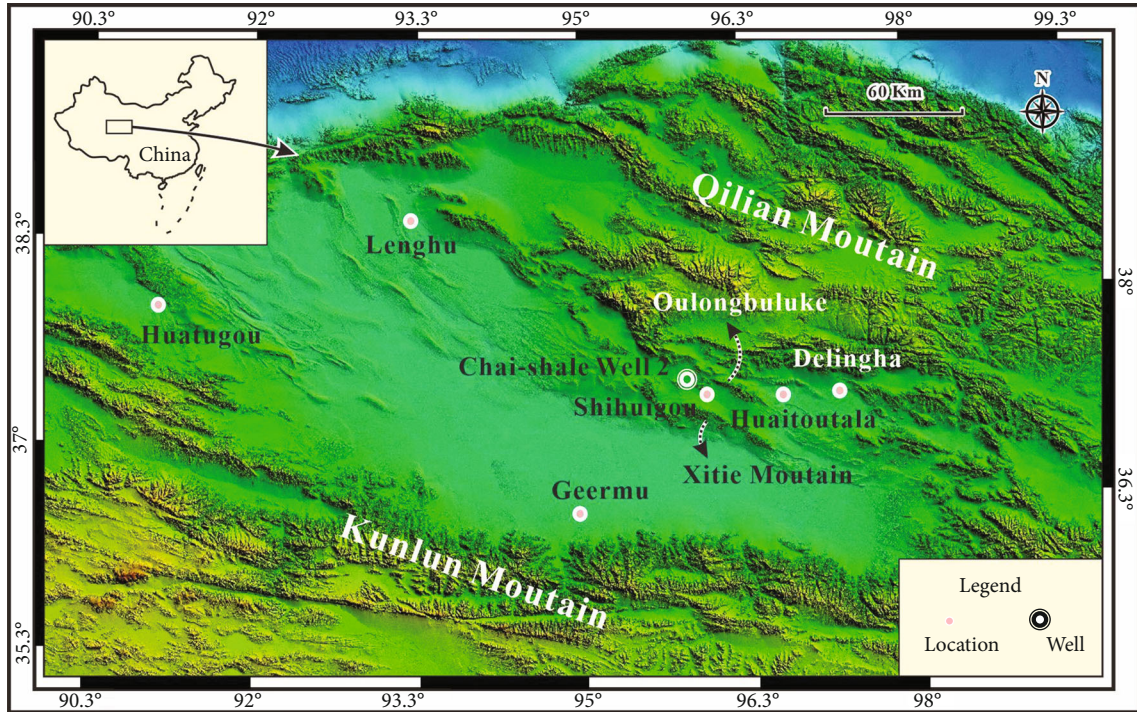


FIGURE 1: Location map of study area.

2. Geological Setting

The Shuihuigou area is located in the Qaidam Basin, west of the Delingha Depression, the southwest of the Oulongbuluke Mountain (Figure 1). The Delingha depression is a rifted basin that originated in the Early Paleozoic, which became a postarc rifted basin during the Middle Paleozoic. It became an intralund multi rift basin since the Jurassic [14–16].

The present study focuses on the Carboniferous Keluke Formation. The Carboniferous System is composed of the Amunike Formation, the Chuanshangou Formation, the Chengqianggou Formation, the Huaitoutala Formation, the Keluke Formation, and the Zabusagaxiu Formation. The lower Carboniferous System is characterized by marine deposition, while the upper Carboniferous System is dominated by marine and marine-land transitional deposition with mud shales and coal (Figure 2). The representative field section of the Keluke Formation was constructed in the Shihuigou area, of which the lithology is characterized by the sandstone, shale, tuff, and interbedded with coal layers. Fan-delta facies, confined terrace facies, open terrace facies, barrier coastal facies, and coastal tidepool facies were primarily identified [7, 17]. In general, the shale reservoir of the Keluke Formation is well developed. The maximum single layer thickness is 21.7 m, the minimum single layer thickness is approximately 6 m, and the maximum continuous thickness is approximately 100 m. The accumulated thickness is 389 m with a shale ratio of 71%. According to the evaluation criteria of “Shale Gas Resource Potential Evaluation Method and Favorable Area Preference Criteria” which

is proposed by the Strategic Research Center for Oil and Gas Resources, Ministry of Land and Resources, China, a single layer with a thickness of more than 5 m and a continuous thickness of not less than 30 m are defined as a favorable target for shale gas production, and therefore, the Keluke Formation has a well potential for shale gas accumulation.

The present work investigated the reservoir features of shale gas reservoirs in the lower Keluke Formation utilizing the continuously cored section of the Chai-Shale Well 2. The core has a length of approximately 120 m, and 31 samples were collected. The average value of TOC in this section was 4.03%, and R_o was between 1.3% and 1.9% (Figure 3). According to the evaluation method for shale gas resource potential of marine-to-terrestrial transitional facies and the preferential selection criteria for favorable area [18], the Keluke Formation is considered to be a shale gas exploration target.

3. Reservoir Types and Features

Several classification schemes exist for the shale gas reservoir types. The International Union of Pure and Applied Chemistry (IUPAC) classifies pores into micropores (2 nm), mesopores (2 to 50 nm), and macropores (>50 nm) based on pore size [19]. While in recent years, a number of scholars have classified shale gas reservoirs based on the pore size distribution and exiting position [20, 21]. Using SEM and thin section observation, the reservoir space of the studied interval was revealed, and the size and distribution of micro- and mesopore volumes were studied using nitrogen adsorption and CO_2 adsorption.

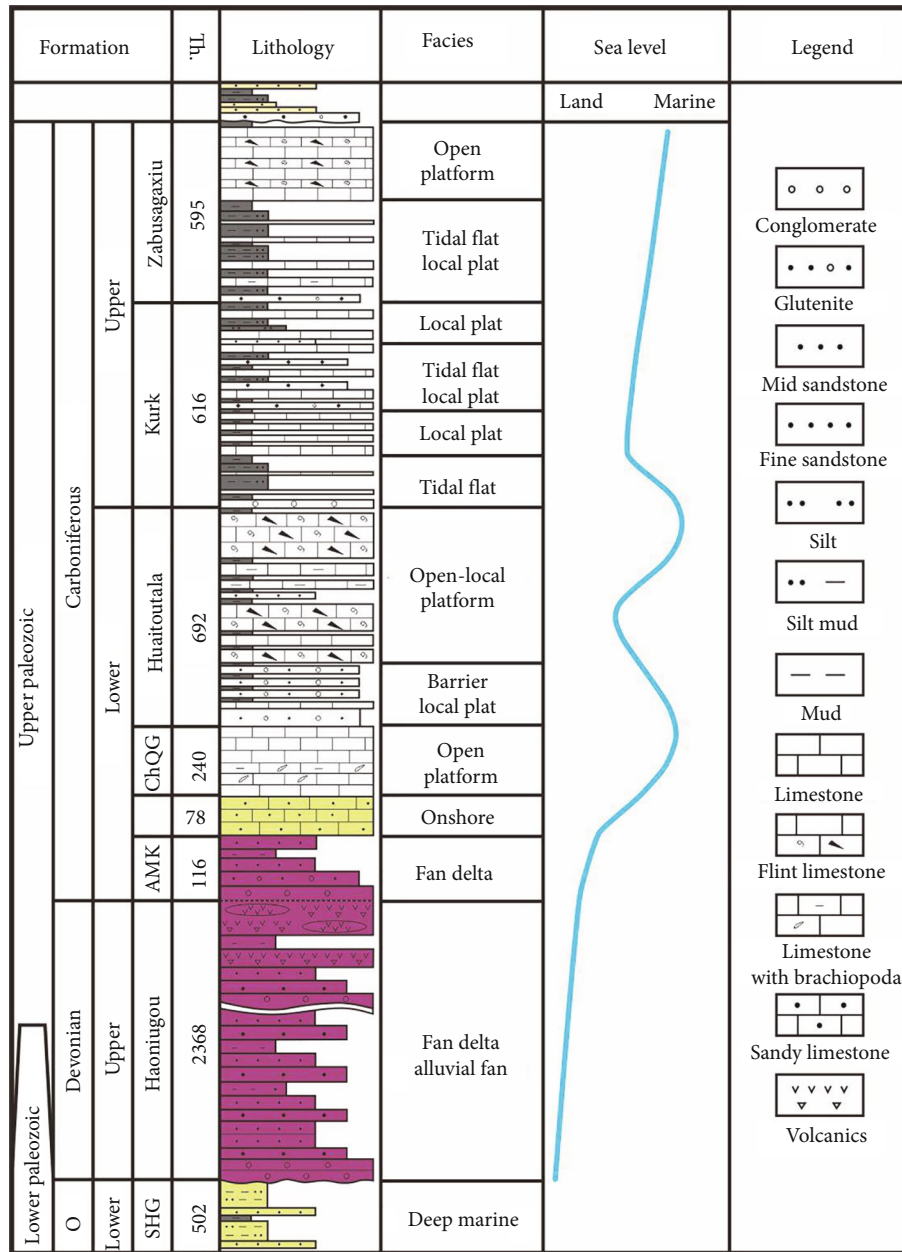


FIGURE 2: Comprehensive stratigraphic column of study area.

3.1. Reservoir Space Type. The reservoir space in the lower Keluke Formation is dominated by pores and fractures. The pores include intergranular pores, intragranular pores, and dissolution pores. The mineral joints fractures, tectonic fractures, diagenetic fractures, and anomalous pressure fractures comprise the majority of fracture-type reservoirs.

3.1.1. Pore Type. Intergranular pores exist between grains or crystals and typically at the contact mineral grains. Commonly, the intergranular pore that exists in the mud shale of the Keluke Formation is intergranular pores of clay minerals and pyrite clusters (Figures 4(a) and 4(b)). The pore size ranges between 200 nm and 2 μm and is predominantly

developed in minerals with big crystals and well crystallization.

Intragranular pores generally exist within mineral grains, organic matters, and biological skeletons. The predominant intragranular pores in the studied interval are developed in biological skeletons and organic matters. The bioclastic intragranular pore are commonly on a micron-millimeter size scale and are mostly found in form of cavity pore and skeletal pore. The common types of bioclastic intragranular pores are brachiopod rash pores, spiny pores, and sea lily stem pores (Figure 4(c)), which are primarily developed in bioclastic muddy siltstones. The organic matter intragranular pore is micron- and nanoscale-scale.

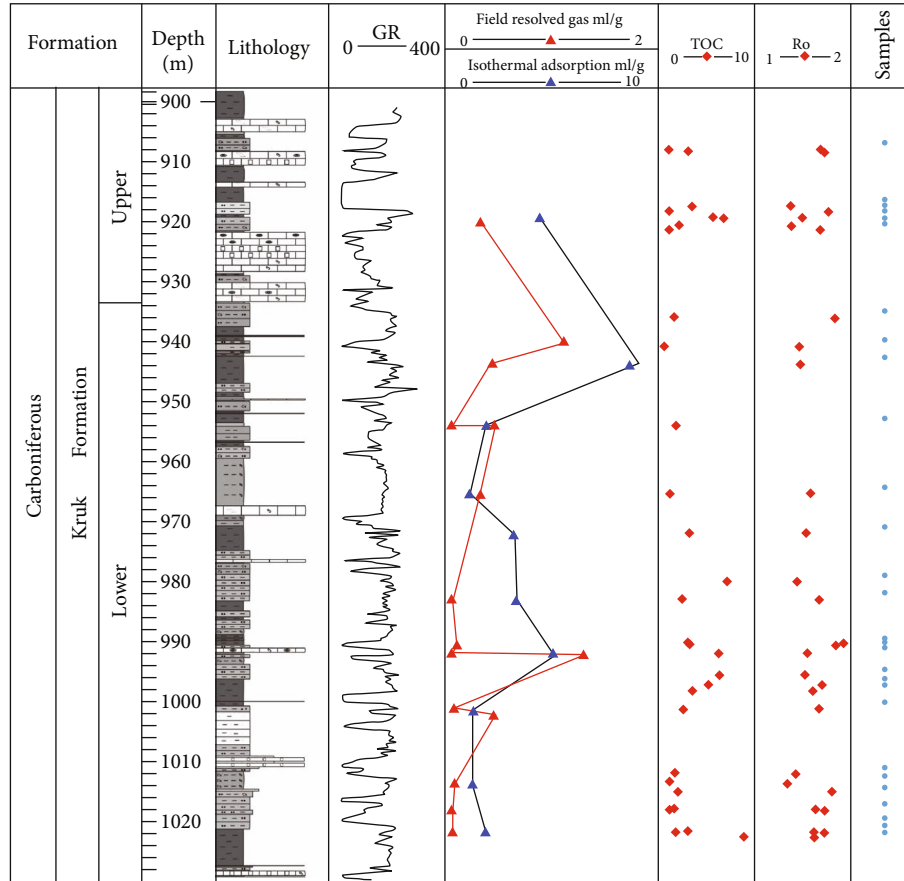


FIGURE 3: Comprehensive column of the core section of the Well Chai-Shale 2.

The TOC increases with R_o which ranges between 1.3% and 2.0%. The thermal evolution of organic matter is at a mature stage, which serves as the fluid source for the formation of dissolution pores. The dissolution pores of the shale of the Keluke Formation are primarily formed by the dissolution of clay minerals, feldspars, and rock fragments. The edges of the dissolution pores are irregular due to dissolution. Additionally, the dissolution pores are solely distributed and have a pore sizes ranging between 20 and 200 μm . (Figures 4(e) and 4(f)).

3.1.2. Fracture Type. Mineral joint fractures are resulted from the breaking of mineral joints (mica, feldspar, etc.) along the joint surface after the release of compressive stress or differential compaction [22]. Joint fractures in Mica, feldspar, and certain fibrous mineral or sheet microfractures are typically observed (Figure 5(a)).

Microfractures are primarily classified into paragenetic, dissolution, and grain margin joints. Most parallel microfractures observed under SEM are developed in the surface of shales (Figure 5(a)), and under the observation of thin sections, they are mostly developed within the interface of rock grain layer. The width of the fracture is between 0.005 and 0.052 mm, with an average width of 0.012 mm. Dissolution fractures are primarily formed by the dissolution on both sides of the fracture. The shape is extremely irregular and port-like (Figure 5(b)). The width of the fracture is

micron-scale, generally around 2 ~ 3 μm . Due to the different mechanical properties between rigid particles and plastic particles, as well as the external force, the grain edge fracture is primarily distributed at the edge of the rock skeleton mineral of the particles such as feldspar and quartz (Figures 5(c) and 5(b)).

Based on the tectonic background of the studied area, tectonic fractures can be categorized primarily as tectonic tension fractures and tectonic shear fractures. Generally, tectonic shear fractures have flat surface and small apertures, as well as extrusion, distortion, and misalignment. The fracture surface of extension fractures is more permeable and serrated (Figure 5(e)).

Anomalous pressure fractures were produced by the overpressure. This type of fracture in the Keluke mud shale is commonly accompanied by the vertical transit of hydrocarbons. The anomalous pressure fractures are predominantly perpendicular to the interval surface (Figure 5(f)) and commonly turn off after overpressure release.

3.2. Micropores and Mesopores

3.2.1. Micropores. Under low temperature, the kinetic energy of N_2 molecules is relatively low, and they cannot enter the micropores. However, CO_2 molecules can enter the 0.35 nm pores. Therefore, the accurate measurement of the pore size ranges around 0.35~2 nm, which is more suitable

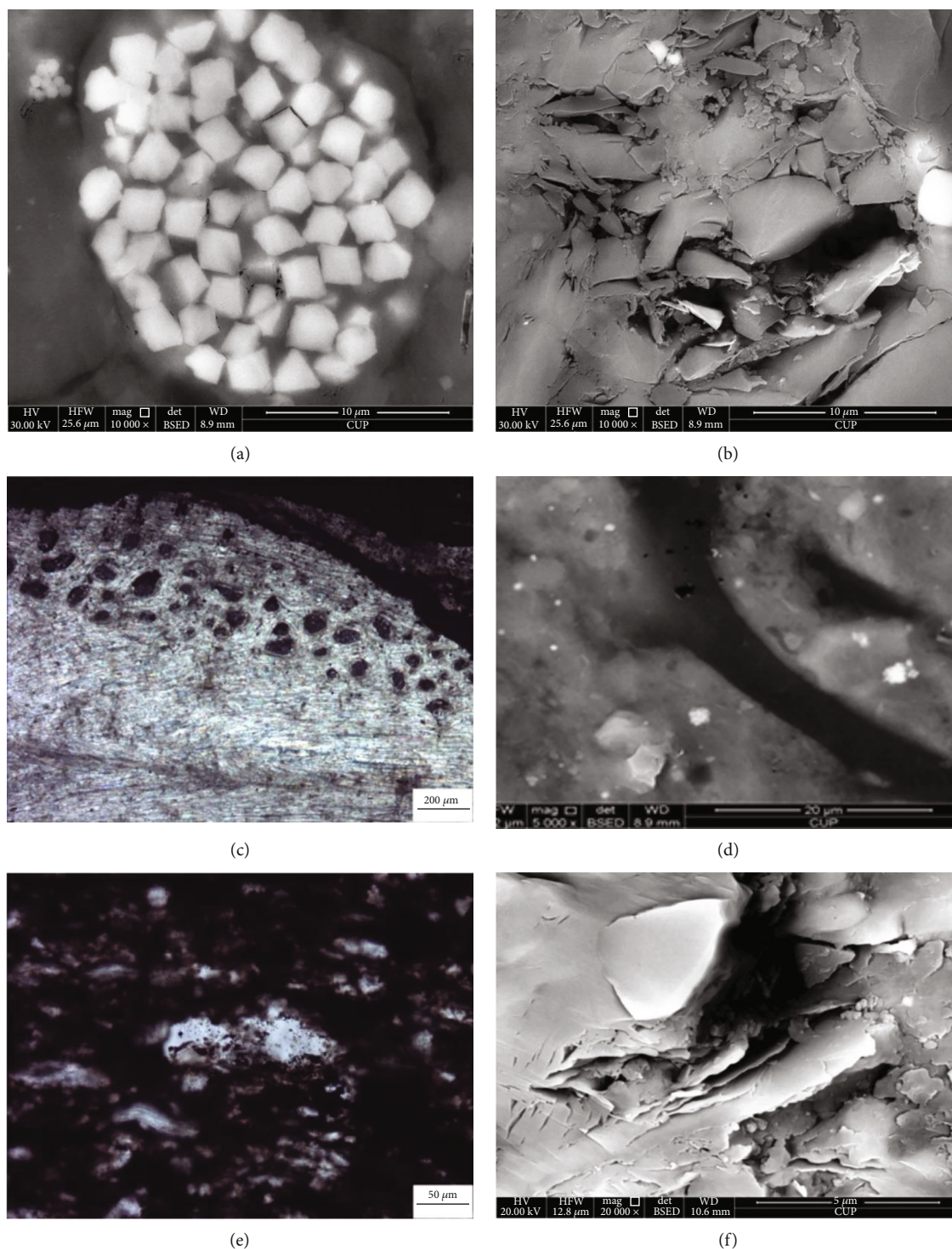


FIGURE 4: Types and microcharacters of the pores. (a) Intercrystalline pore of pyrite, SEM, 990.90 m. (b) Intercrystalline pores of clay minerals, SEM, 997.15 m. (c) Bioclastic grain endopore, (+)50×, 1033.10 m. (d) Organic matter grain inner pore, SEM, 990.90 m. (e) Clay mineral dissolution pores, (-)200×. (f) Inner particle dissolution pores, SEM, 1020.74 m.

for the study of micropores. The results of CO₂ adsorption illustrate the variation of pore volume and specific surface area of micropores with pore diameter. The absolute volume of adsorption of individual samples varies strongly but show a similar variation pattern. Both the pore volume and specific surface area exhibit a multippeak characteristic when

the pore size is ranging between 0.4 and 0.7 nm, which indicates that the micropores of the shale samples are dominated by pores in the size range of 0.4 to 0.7 nm, and that the pore size distribution is not uniform (Figure 6). The CO₂ adsorption experiments indicate that the micropore volumes of the shale samples varies between $0.5 \cdot 10^{-4}$ and $5.8 \cdot 10^{-4}$ ml/g,

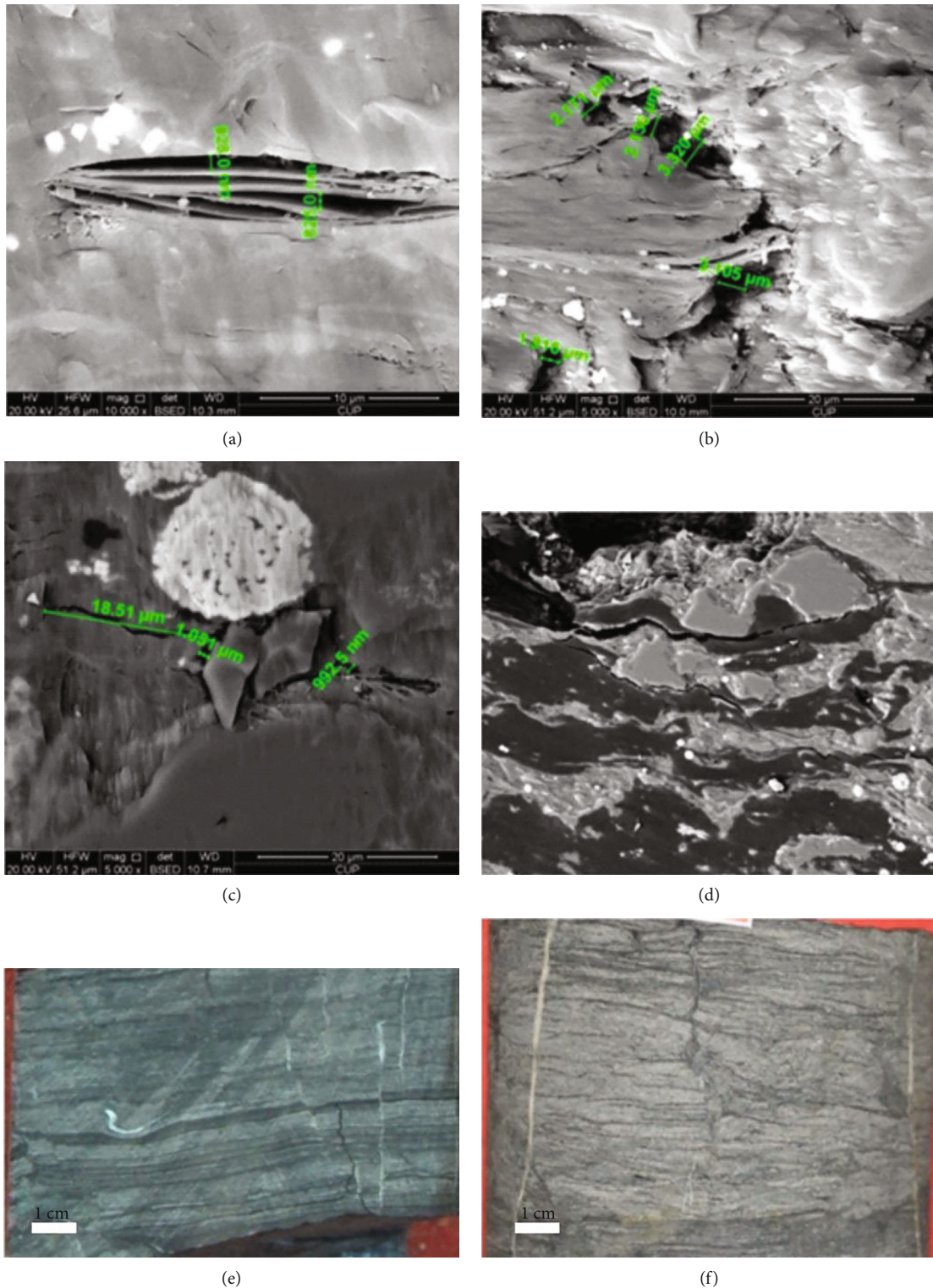


FIGURE 5: Types and characters of the fractures. (a) Mineral joint fracture, SEM, 911.77 m. (b) Dissolved fracture, SEM, 916 m. (c) Microcracks between pyrite and mineral skeleton, SEM, 907 m. (d) Microcracks between bands of organic matter, SEM, 1021.6 m. (e) Tectonic fracture, 1047.90 m. (f) Abnormal pressure fracture, 1039.10 m.

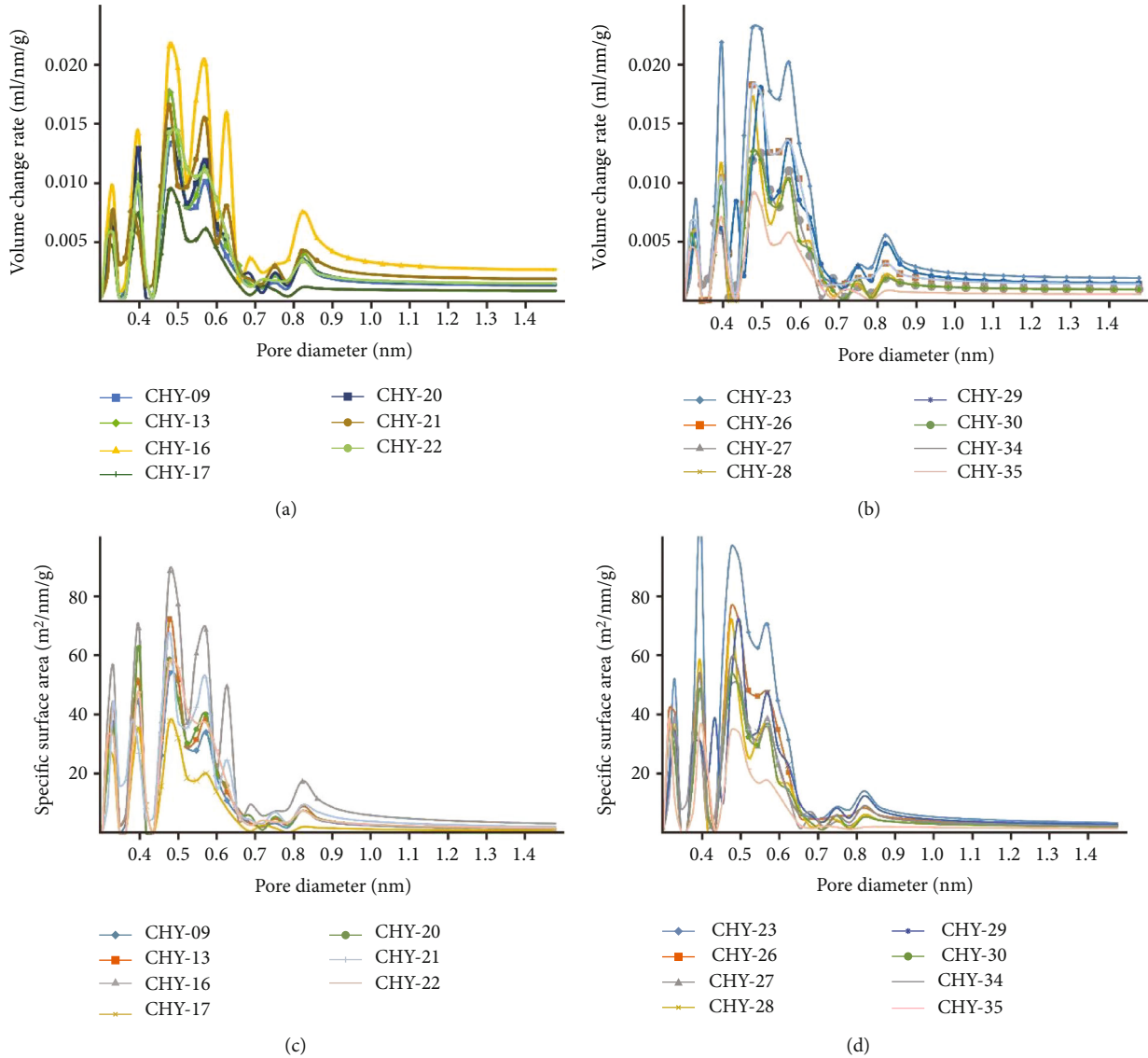


FIGURE 6: Pore volume and surface area variation of micropores with the pore diameter.

while the micropore specific surface area ranges from 0.18 to 1.83 m²/g, with an average of 0.43 m²/g.

3.2.2. Mesopore. The nitrogen adsorption-desorption isothermal curves have an inverse-S shape, with minor variations in the overall curve morphology (Figure 7). According to the international BET categorization for isothermal adsorption curves, the curves are defined as the type II isothermal adsorption morphology. When the ratio of P/P_0 is over 0.4, the desorption curve of the sample no longer coincides with the adsorption curve, and a hysteresis loop and establishing an adsorption return line generate. The morphology of adsorption return line indicates different pore types. Based on De Boer’s classification of adsorption return lines, IUPAC divides the adsorption return lines into four groups. The sample of the Keluke Formation shows that at $P/P_0 = 1.0$, the curve is steep. The desorption curve is steep at moderate pressure of $P/P_0 = 0.5$, which is more sim-

ilar to the H3-type adsorption return line in the IUPAC classification. The adsorption return line of minor samples has the characteristics of H2-type, which indicates that the pores of the studied shale reservoir are mainly a mixture of H2 and H3. It suggests a complex pore morphology and irregular pore structure constituted by ink bottle-like pores and slit-like pores.

The nitrogen adsorption results demonstrate that the pore volume distribution of the mesopores is featured by single peak, as well as the pore size distribution with a peak at approximately 3 nm (Figures 8(a) and 8(b)). While the specific surface area also exhibits a single peak characteristic at 3 nm and increased gradually from 6 nm (Figures 8(c) and 8(d)). Only two samples deviated from the main pattern among them (CHY-09 and CHY-35).

The CO₂ and N₂ adsorption results suggest that the pore volume distribution of the studied shale reservoir does not have an overall uniform tendency but similarities to some

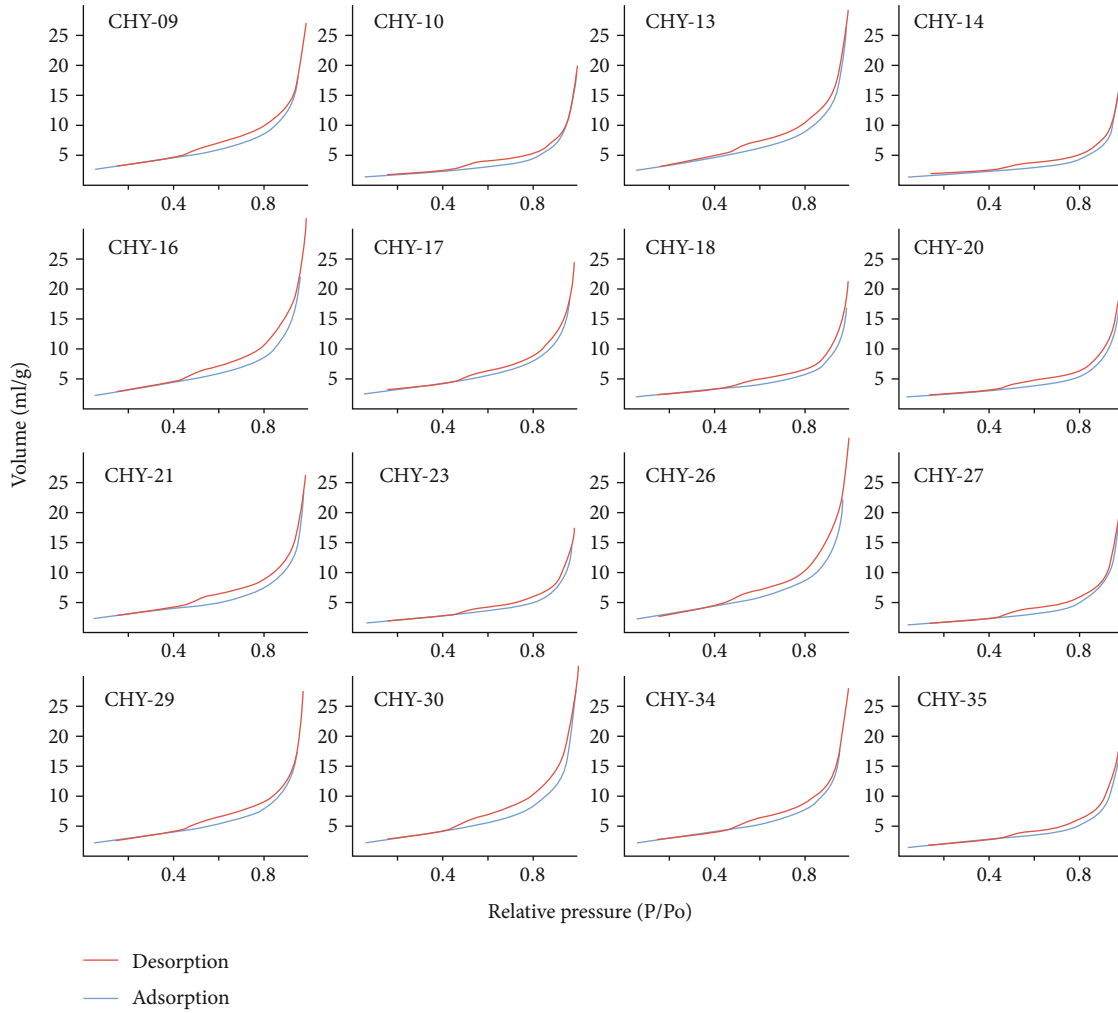


FIGURE 7: N_2 adsorption-desorption isotherms of shale of the Keluke Fm.

extent. The micropores are mainly developed at the diameter ranging between 0.3 and 0.6 nm, and the pore volume decreases significantly when the micropore pore diameter is larger than 0.6 nm. The diameter of mesopores is smaller than 10 nm. The specific surface area of micropores is significantly greater than that of mesopores, which indicates that the specific surface area is primarily provided by micropores. Meanwhile, the pores with diameter between 0.3 nm and 0.6 nm account for the substantial part of specific surface area, which indicates that the adsorbed gas is primarily stored in mesopores pores.

4. The Storage Property of Shale Reservoirs

4.1. Porosity and Permeability. The porosity and permeability of 51 shale samples from the lower Keluke Formation in the study area were tested. The porosity of the studied reservoir ranges between 0.69% and 5.55%, with an average value of 2.1%. The permeability primarily ranges between 0.01 and 2.21 mD, with an average value of 0.39 mD.

4.2. Storage Property. The Oil and Gas Resources Investigation Center drilled the second shale gas well in the Qaidam Basin, Chai-Shale Well 2, in the Shihuigou area. The target formation is the shale deposition of the Keluke Formation, and 17 shale samples were desorbed. The evaluated gas concentration in the field ranges between 0.01 and 1.23 ml/g with an average of 0.26 ml/g, and only two coal samples exceeded 1 ml/g.

Ten samples of shale from the lower the Keluke Formation in the research area were subjected to isothermal adsorption modeling experiments at 30°C and pressures between 0 and 12 MPa. The adsorption data were used to plot the adsorption isotherms (Figure 9), and the measured methane adsorption isotherms conformed with type I. For type I, the maximum theoretical adsorption volume (PL) of the samples was obtained by using the Langmuir equation. The maximum adsorbed gas concentration of the studied shale reservoirs ranges between 0.88 and 8.65 m^3/t , with an average of 2.52 m^3/t . The fitted methane isotherm adsorption line is defined as type I isotherm, which mostly demonstrates the adsorption of the microporous surface. The

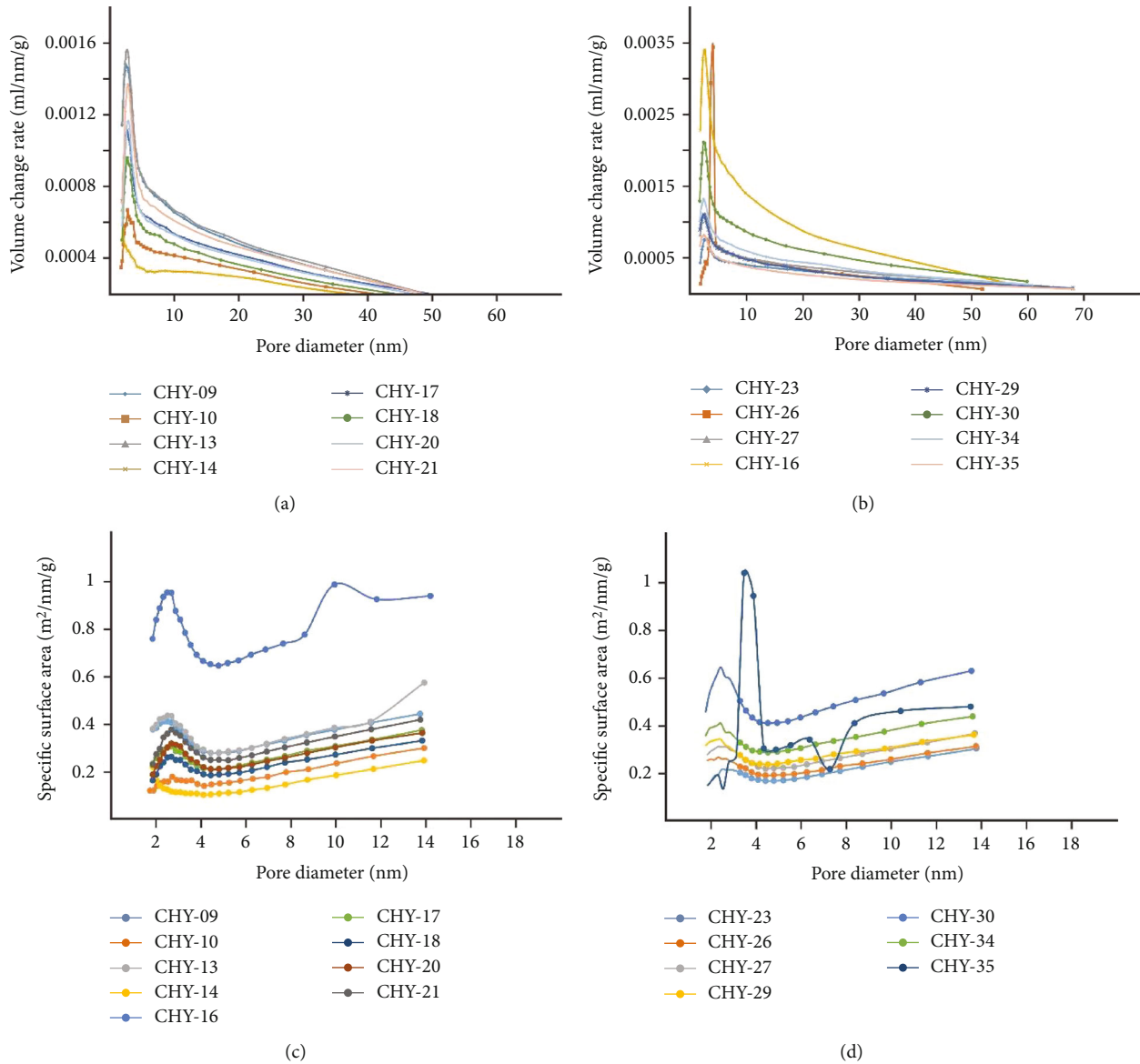


FIGURE 8: Nitrogen adsorption curve with the pore diameter.

adsorption of methane in the study shale increases with pressure between 0 and 12 MPa at a constant temperature and approaches saturation at a pressure close to 12 MPa.

5. Controlling Factors on Gas Content

The controlling factors of shale gas absorption and its mechanism challenge the theoretical study on shale gas development. The shale gas adsorption is primarily influenced by temperature, pressure, mineral composition, TOC, organic matter type, R_o , and other variables. Furthermore, the effect of clay minerals on adsorption is more complex. Using experimental data, the present work analyzes the adsorption-influencing factors of the shale reservoir of the Keluke Formation.

5.1. TOC. For shales with high maturity, the parameters of chloroform asphalt A and hydrocarbon generation potential

can only reflect the content of residual hydrocarbons, and only the TOC can accurately reflect the organic matter abundance. Therefore, TOC is one of the important controlling factors of gas-bearing properties of shale gas. The present study results agree that the higher the TOC content, the higher the adsorbed gas content of the shale, which shows a good linear relationship with a fitting coefficient (R^2) of 0.8507 (Figure 10(a)). This indicates that the TOC is the main controlling factor of shale gas adsorption. When TOC is zero, methane adsorption is not zero, indicating that although the adsorbed gas is mostly adsorbed by organic matter pores, other form of micropores and mesopores still provide space for methane gas adsorption.

5.2. Clay Minerals. Most investigations have shown that clay mineral conduces to shale adsorption. The higher the percentage of clay minerals is, the larger the volume of adsorbed gas. Nonetheless, other academics suggest that

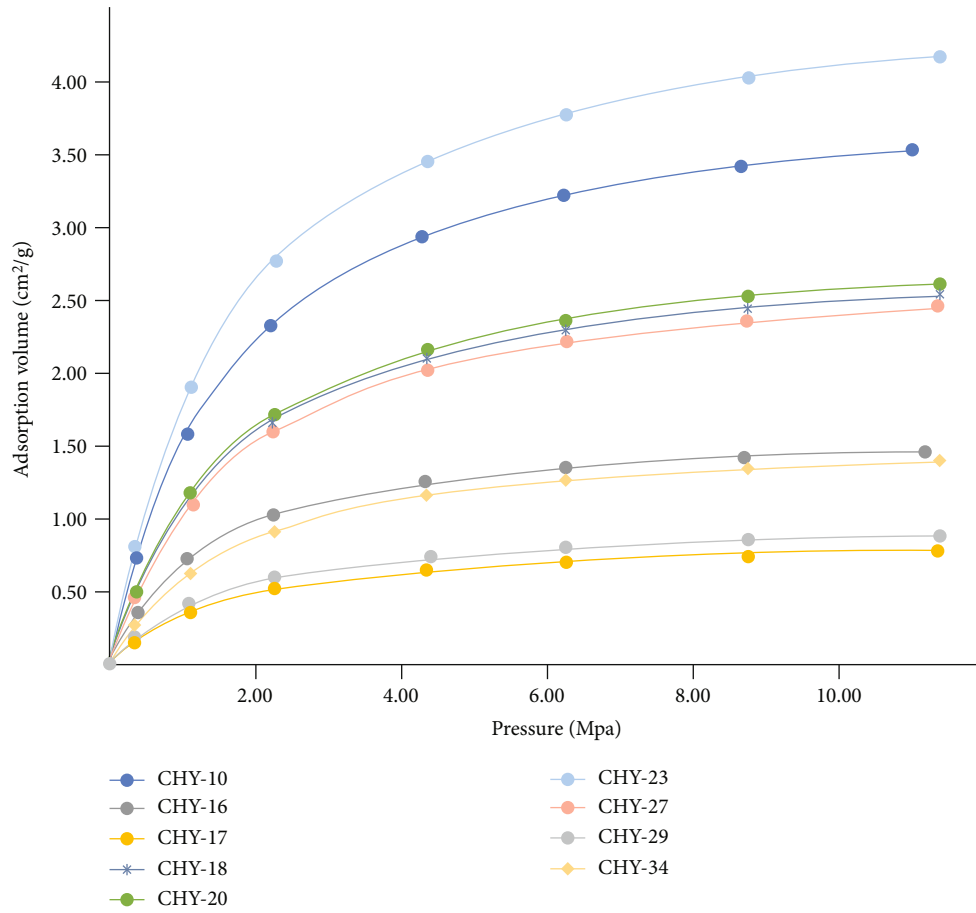


FIGURE 9: Methane isothermal adsorption curve of shale of Keluke Fm.

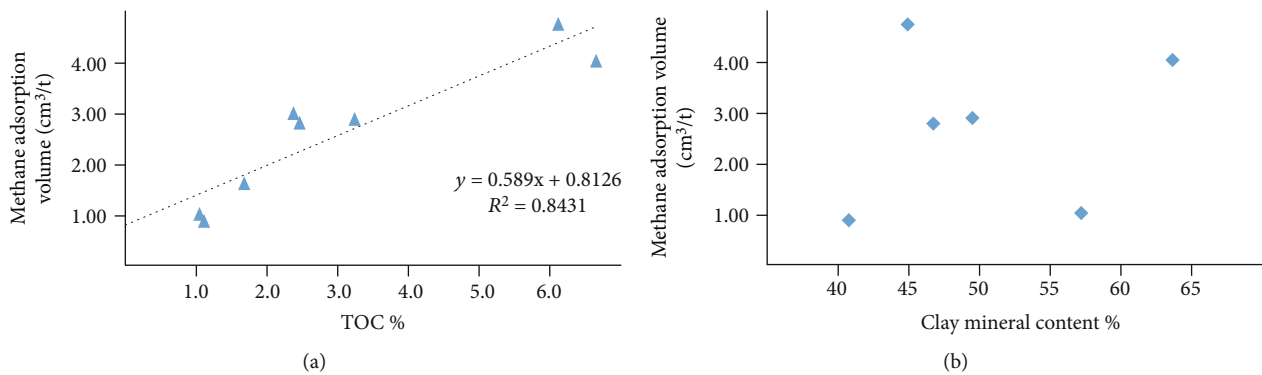


FIGURE 10: Relationship between methane adsorption and TOC, clay mineral content.

clay minerals are adversely connected with adsorption, which indicates that the influence of clay minerals on sorption is not well understood. Figure 10(b) demonstrates that the correlation between clay mineral and methane adsorption is weak, demonstrating that the effect of clay mineral content on the adsorption of shale gas in this study is unclear.

5.3. *Content of Field Resolved Gas.* The amount of field resolved gas can reflect the real volume of gas absorbed in

the shale reservoir, including adsorbed and free gas. According to above discussion, it is inferred that TOC was the primary factor influencing the absorbable gas content. However, it does not imply that TOC affects the real gas content. The present study evaluated the link between TOC, clay mineral content, and the volume of field resolved gas to determine the main factor that affect the actual gas content (Figure 11). The result indicates a positive association between field resolved gas and clay mineral concentration, and the relationship between TOC and field resolved

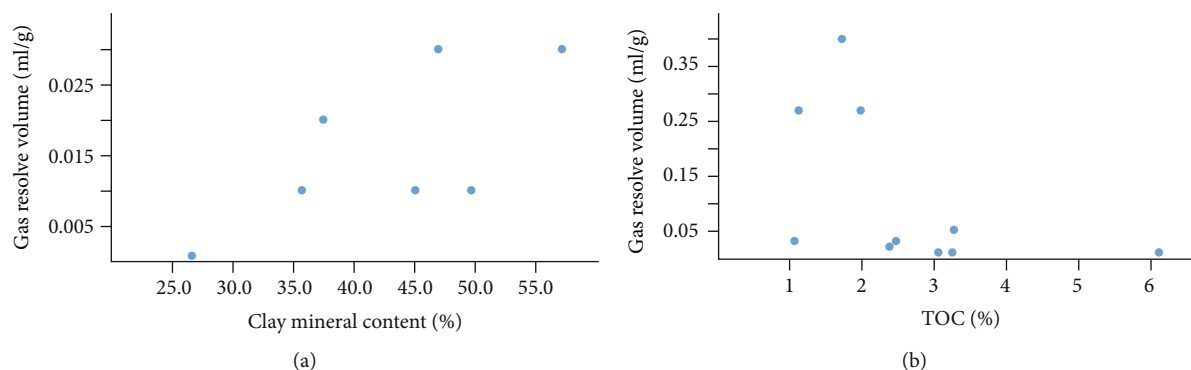


FIGURE 11: Relationship between field resolved gas and TOC, clay mineral content.

gas is weak. This does not adequately explain the link between field resolved gas and TOC. However, it can be presumed that the majority of the natural gas present in the lower shale Keluke Formation is free gas and gas adsorbed by clay minerals.

6. Conclusion

- (1) The shale reservoir of the lower Keluke Formation is classified into pores and fractures
- (2) Micropores are primarily formed by pores with a diameter between 0.3 and 0.6 nm; when the pore diameter of micropores exceeds 0.6 nm, the pore volume is drastically reduced. The pore diameter of medium pores is unevenly distributed and smaller than 10 nm. The pore with diameter around 3 nm accounts for the main volume of the total pore volume. The pore volume of mesopores is larger than that of micropores. However, the specific surface area of micropores is much higher than that of mesopores, which indicates that the micropores play a dominated role on the adsorption capacity
- (3) According to the experiment results, the porosity of the studied shale reservoir ranges between 0.69% and 5.55%, with an average value of 2.1%. The permeability ranges between 0.01 and 2.21 mD, with an average value of 0.39 mD. The field resolved gas content ranges between 0.01 and 1.23 ml/g, with an average of 0.26 ml/g. It demonstrates the well storage capacity and adequate gas content
- (4) TOC plays a dominant role in the adsorption capacity of the shale reservoir of the Keluke Formation, and clay minerals do not have clear influence on it. However, there is no obvious relationship between TOC and field resolved gas volume, while the clay mineral content has a positive correlation with field resolved gas volume. Therefore, it is assumed that the free gas is predominant form in the shale reservoir of the lower Keluke Formation, and adsorbed gas comes in second

Data Availability

The data that support the findings of this study are available from the corresponding author upon reasonable request.

Conflicts of Interest

The author declares that they have no conflicts of interest.

Acknowledgments

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