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Study on the State of Methane Molecule Adsorption on Different Media in Highly Evolved Marine Shales —A Case Study on the Shales From the Lower Silurian Longmaxi Formation in the Sichuan Basin, Southern China

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The major sedimentary basins in China contain abundant shale gas resources to be explored, and the exploration of shale gas has received more attention in recent years. Shale gas exists mainly in two states, i.e., free and adsorbed. The latter mainly exists on the surface of media, including organic matter and clay minerals, etc., but its adsorption state remains unknown. In this paper, we take the Longmaxi Formation marine shale in the southern Sichuan Basin of southern China as the research object. The state of methane molecule adsorption on different media in marine shales is analyzed by conducting mineral composition analysis, TOC content analysis, isothermal adsorption experiments, FIB-SEM, and FIB-HIM experiments on the core samples referring to previous research. The conclusions are as follows: the adsorbed gas mainly exists in the organic-matter pores, which feature excellent roundness and connectivity with a large number of small pores inside like a hive. The surface of the organic matter contains many adsorption sites, featuring strong adsorption capacity and making methane molecules continuously distributed on the internal surface of the organic-matter pores. The organic matter has a large specific surface area and is lipophilic, which offers an ideal condition for the adsorption of methane molecules. Part of the adsorbed gas exists in the pores of clay minerals, which are lamellar and triangular. The surface of clay minerals contains fewer adsorption sites, featuring poorer adsorption capacity and making methane molecules discontinuously distributed on the surface of the clay minerals. The clay minerals have a smaller specific surface area than the organic matter, thus featuring a smaller space for adsorption. The clay minerals are hydrophilic. In addition to methane molecules, mixed-layer illite/smectite (I/S) and chlorite also adsorb water molecules. The illite surface adsorbs mainly water molecules and, to a lesser extent, methane molecules. Finally, the adsorption state patterns of methane molecules on organic matter and clay minerals were summarized.

Keywords: marine shales, methane molecules, organic matter, clay minerals, adsorption state

1 INTRODUCTION

In recent years, with the improvement in geological ideas as well as the advancement of good horizontal drilling and hydraulic fracturing technologies, the target of oil and gas exploration has changed from conventional clastic and carbonate reservoirs to unconventional shale ones, and great success in shale gas exploration has been achieved in North America (Curtis, 2002). Similar to North America, China contains abundant shale gas resources, and shale formations are widely distributed in the major basins of China (Guo, 2016; Zou et al., 2017; Guo, 2021). In the Sichuan Basin and its periphery in southern China, based on the exploration for marine shales of the Lower Silurian Longmaxi Formation, a series of shale gas fields such as Weiyuan, Changning, Weirong, Fushun, Yongchuan, Zhaotong, and Jiushiba have been successively established by CNPC and Sinopec and provided high production of shale gas (Zou et al., 2015; Guo et al., 2016; Guo et al., 2017; Guo et al., 2020). Based on the existence state, shale gas can be categorized into free and adsorbed types: the former exists in the shale reservoir space, while the latter on the surface of organic matter and clay minerals. The adsorbed gas can be converted into free gas under certain temperature and pressure conditions (Zou et al., 2017; Zou et al., 2019; Zou et al., 2020).

A series of studies have been conducted previously on the factors controlling the adsorption capacity of marine shales. Li et al. (2017) concluded from a study on the shales of the Lower Cambrian Niutitang Formation in the Fenggang Block, northern Guizhou, southern China, that the methane adsorption quantity of shales is related to organic carbon content, clay minerals, organic matter pore morphology, pressure, temperature and water saturation (Li et al., 2017). Gao et al. (2018) concluded that adsorbed gas is an important state of shale gas, mainly existing on the surface or inside the pores of clay minerals and organic matter particles and that the adsorption characteristics of shale play a crucial role in gas content. The adsorption capacity is controlled by a combination of factors such as organic matter content, degree of thermal evolution, pore structures, and clay mineral content (Gao et al., 2018). Ma et al. (2018) concluded that organic matter characteristics, nanopore structures, inorganic mineral composition, temperature, pressure, and water content, etc., all influence the methane adsorption capacity of shales to some extent (Ma et al., 2018).

In recent years, the large-scale exploration of shales has provided a lot more data for the analysis on the state of methane molecule adsorption in different media of highly-evolved marine shales. In this study, the adsorption states of methane on different media of highly-evolved marine shales were identified *via* mineral composition analysis, TOC content analysis experiments, isothermal adsorption experiments, and direct observation with an SEM by using the shale gas well XNY-1 lately drilled in the southern Sichuan Basin, of which the location is shown in **Figure 1**.

2 GEOLOGICAL SETTINGS

2.1 Sedimentary and Stratum Characteristics

According to previous studies (Li et al., 1995; Li et al., 2002; Mei et al., 2012; Wang et al., 2015; Mou et al., 2016; Zhang et al., 2019a; Zhang et al., 2020a), in the Upper Ordovician-Lower Silurian, the Upper Yangtze area became the interior Cratonic sagging basin after the Cathaysian Plate extruded it. In the Upper Yangtze region, the sedimentary strata of the Upper Ordovician

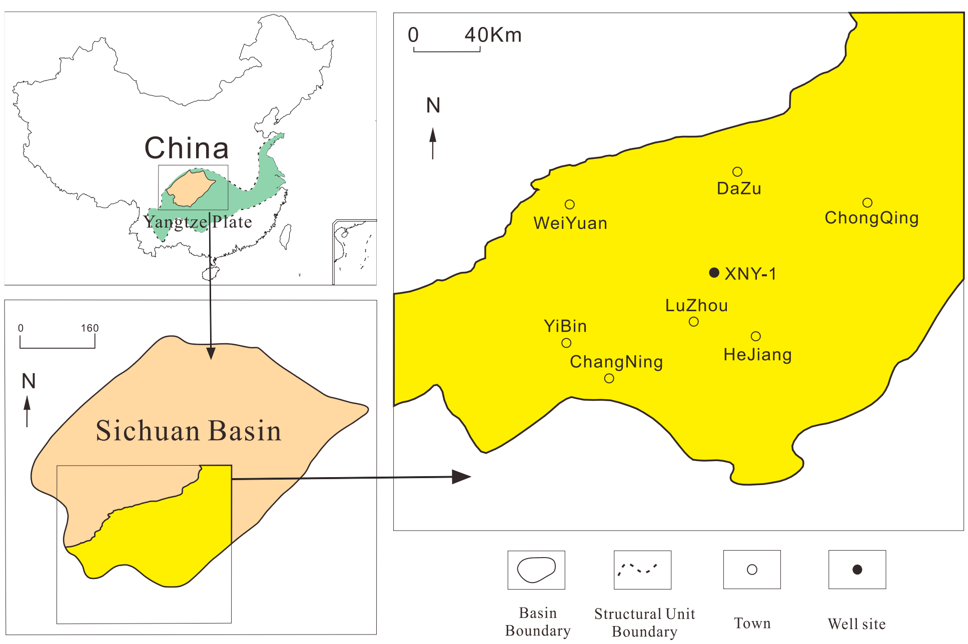

FIGURE 1 | Location of the southern Sichuan Basin in southern China and the distribution of XNY-1 well. Modified from references (Mei et al., 2012; Wang et al., 2015; Mou et al., 2016; Zhang et al., 2019a; Zhang et al., 2020a).

TABLE 1 | Core sampling layer and depth.

No.	Well	Fm.	Depth (m)	No.	Well	Fm.	Depth (m)
1	XNY-1	Longmaxi	4,024.29	7	XNY-1	Longmaxi	4,085.46
2	XNY-1	Longmaxi	4,041.66	8	XNY-1	Longmaxi	4,087.84
3	XNY-1	Longmaxi	4,053.95	9	XNY-1	Longmaxi	4,089.63
4	XNY-1	Longmaxi	4,062.35	10	XNY-1	Longmaxi	4,091.7
5	XNY-1	Longmaxi	4,072.92	11	XNY-1	Longmaxi	4,092.91
6	XNY-1	Longmaxi	4,080.01	12	XNY-1	Longmaxi	4,094.68

TABLE 2 | Results of mineral composition analysis of core samples.

Depth (m)	Quartz (%)	Potash feldspar (%)	Plagioclase (%)	Calcite (%)	Dolomite (%)	Pyrite (%)	Clay minerals (%)
4,024.29	31.5	0.7	7.6	0	0	3.2	57
4,041.66	33.3	0	6.2	0	0	2.7	57.8
4,053.95	35.9	0	7.1	2.9	6.4	5.1	42.6
4,062.35	29.9	0.9	3.6	5.5	12.4	3.7	44
4,072.92	42.1	0	8.4	2.5	5.2	3.1	38.7
4,080.01	44.3	0.9	6.9	3.5	3.4	4.7	36.3
4,085.46	57.5	0	3.4	6.9	9	2.6	20.6
4,087.84	64.4	1.1	3.3	3.7	4.7	3.8	19
4,089.63	42.2	0.9	4.4	4.5	9.7	7.3	31
4,091.7	51	0	1.4	4.2	21.9	3.6	17.9
4,092.91	31.7	1.1	5.1	2.9	5.7	3.8	49.7
4,094.68	18.4	1	3.8	0	2.8	8.2	65.8

TABLE 3 | Results of core sample analysis for TOC content, Langmuir Volume, Langmuir Pressure and clay mineral composition.

Depth (m)	TOC (%)	Langmuir volume (m^3/t)	Langmuir pressure (MPa)	I/S mixed layer (%)	Illite (%)	Chlorite (%)
4,024.29	0.77	1.07	3.84	23.94	19.38	13.68
4,041.66	0.2	0.31	0.37	24.854	21.964	10.982
4,053.95	1.82	1.22	0.51	20.448	17.892	4.26
4,062.35	2.28	1.58	2.36	20.24	18.92	4.84
4,072.92	1.72	0.73	0.39	19.737	15.867	3.096
4,080.01	2.74	1.88	2.99	16.698	16.698	2.904
4,085.46	3.2	1.28	0.54	13.39	5.974	1.236
4,087.84	4.05	1.41	0.7	11.21	7.03	0.76
4,089.63	4.79	1.72	1.19	18.29	11.78	0.93
4,091.7	2.02	1.25	2.2	8.771	8.592	0.537
4,092.91	1.26	0.5	1.09	30.317	15.904	3.479
4,094.68	2.22	0.77	0.97	38.164	23.03	4.606

are called the Wufeng Formation, while the sedimentary strata of the Lower Silurian are called the Longmaxi formation. The Longmaxi Formation can be divided as Member I, II, and III from the bottom to the top. The target layer of this study is Member I, of which the shale lithology is bipartite: the lower part of Member I of the Longmaxi Formation is mainly black siliceous organic-rich shales, while the upper part is a combination of dark grey shales, siltstone shales, and siltstone.

2.2 Tectonic Characteristics

Previous studies (Wang and Li, 2003; Chalmers and Bustin, 2008; Ji et al., 2014; Ji et al., 2015; Ji et al., 2016; Zhang et al., 2017) show that, large-scale sea erosion of the Yangtse plate in the Early Cambrian.resulted in the sedimentation of a set of organic-rich shales that almost covered the entire plate. Thereafter, the water body became shallower, while fine and silty shales gradually changed into coarse clasolite, such as siltstone and sandstone, etc. Due to the extrusion and collision from the Cathysian plate in the Ordovician, the water body continuously became even shallower, and the sedimentary system of clasolite changed into the sedimentary system of carbonite. A large-scale transgression that occurred in the Upper Ordovician-Lower Silurian changed it back to the sedimentary system of clasolite, leaving the sedimentation of a set of organic-rich shales in the deep-water shelf surrounded by the ancient land.

3 SAMPLES, EXPERIMENTS, AND SOURCE OF DATA

In this study, 12 pieces of core samples were taken from the shales of Member I of the Longmaxi Formation in the XNY-1 well at the depths shown in **Table 1**. For the six pieces of the core samples taken from the same depth, whole-rock mineral analysis and clay

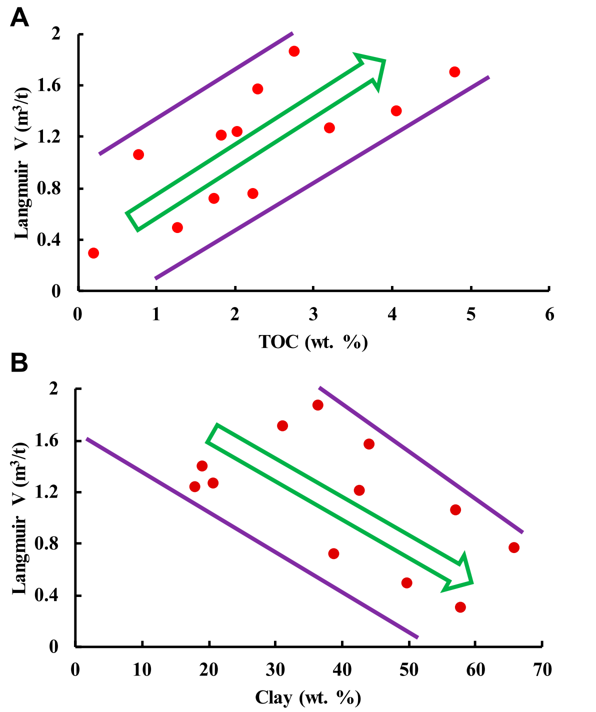

FIGURE 2 | Analysis of Langmuir Volume's relation with TOC content and clay mineral content. The graph shows Langmuir Volume has a positive correlation with TOC content and a negative correlation with clay mineral content.

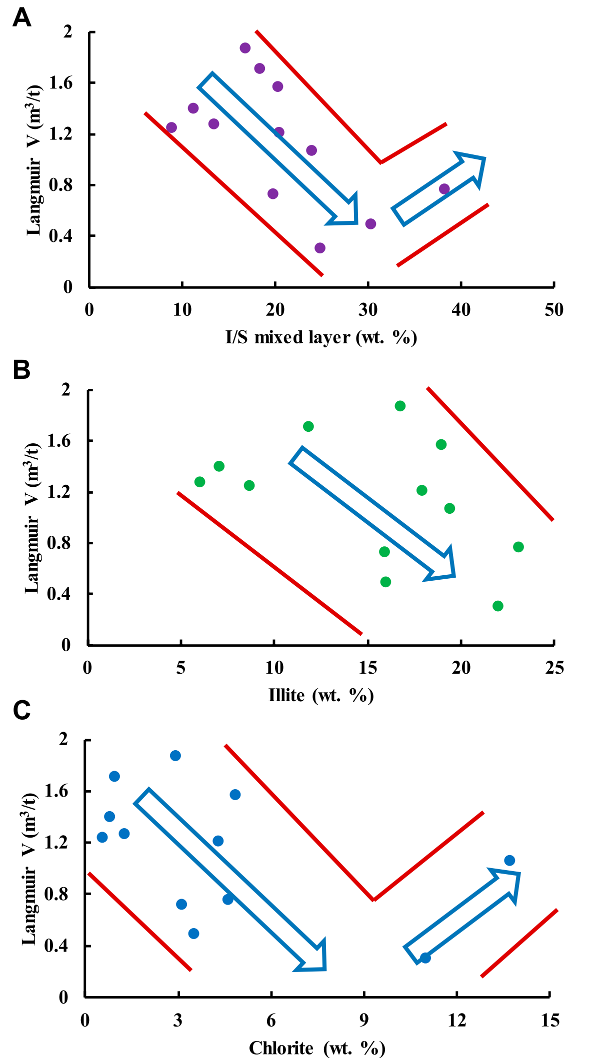

FIGURE 3 | Analysis of Langmuir Volume's relation with I/S mixed layer, illite, and chlorite. It can be seen that Langmuir Volume is first negatively and then positively correlated with I/S mixed layer content and with chlorite content. It is negatively correlated with illite content.

mineral analysis were conducted with a YST-I mineral analyzer, and isothermal adsorption experiments were conducted at 110°C with an HPVA-200-4 isothermal adsorber. Besides, TOC analysis was conducted with a Sievers 860 TOC content analyzer, and FIB-SEM (Focused ion beam—FIB-SEM) was conducted with a Helios NanoLab 660. At the same time, FIB-HIM (Focused ion beam—Helium ion microscopy) was conducted with a Zeiss Orion NanoFab, and part of the samples was used for organism maturity analysis with a ZEISS Imager A2m, J&M MSP200 polarized fluorescence microscope.

4 RESULTS AND DISCUSSION

4.1 Analysis of the Ability of Organic Matter to Adsorb Methane Molecules

The results of mineral composition analysis, TOC content analysis, and isothermal adsorption experiments carried out on the 12 shale core samples are shown in **Tables 2**, **3**. Langmuir Volume represents the maximum adsorption capacity, and its physical meaning is the adsorbed gas content at a given temperature when the methane adsorption of shales reaches saturation with m3/t as the unit. Langmuir Pressure is the pressure corresponding to half of the Langmuir volume with MPa as the unit. According to the maturity test of the shale cores, the average organic matter maturity of the shales of the Longmaxi Formation from the XNY-1 well is 2.1%. Therefore, the study target is highly evolved marine shales. This work analyzed TOC content and Langmuir Volum, and the results are shown in **Figure 2A**, according to which, the TOC content has a good positive correlation with Langmuir Volume. This implies that the organic matter provides the main adsorption space for methane molecules, which are adsorbed on the inner surface of the organic matter.

4.2 Analysis of Clay Minerals' Ability to Adsorb Methane Molecules

Figure 2B shows the analyses results of the clay mineral content and the Langmuir Volume. As can be observed, the clay mineral content has a significant negative correlation with Langmuir Volume. However, previous studies have shown that clay minerals have a certain adsorption capacity for methane molecules, which means that more works need to be carried

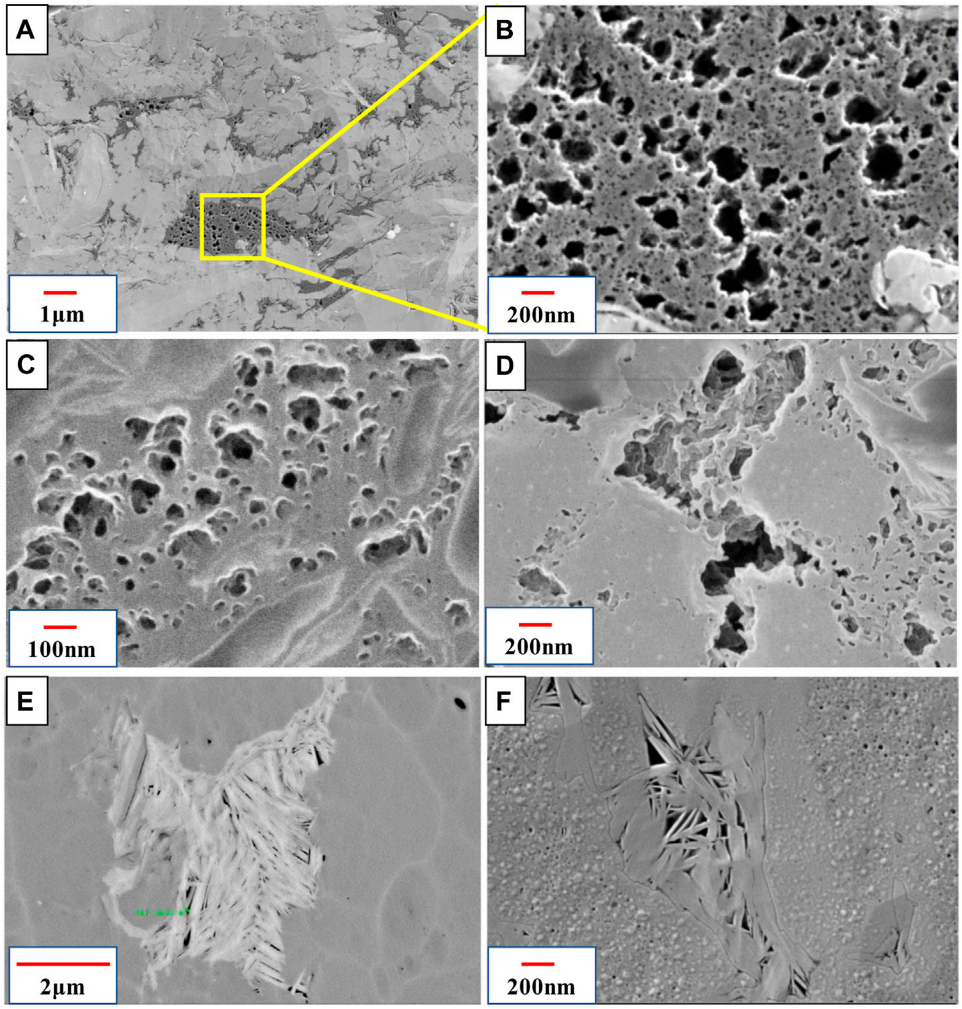

FIGURE 4 | FIB-SEM images **(A,B,E,F)**, FIB-HIM images **(C,D)**. **(A,B)**: 4,085.46 m, XNY-1 Well, Member I of the Longmaxi Formation; **(C)** 4,087.84 m, XNY-1 Well, Member I of Longmaxi Formation; **(D)** 4,089.63 m, XNY-1 Well, Member I of Longmaxi Formation; **(E)** 4,062.35 m, XNY-1 Well, Member I of Longmaxi Formation; **(F)** 4,072.92 m, XNY-1 Well, Member I of Longmaxi Formation.

out to study the negative correlation. The clay minerals in the highly evolved marine shales of the Langmuir Formation consist of I/S mixed layer, illite, and chlorite. This work analyzed the relationship between the Langmuir Volume and the contents of I/S mixed layer, illite, and Chlorite content, as shown in **Figures 3A–C**. It can be seen that the illite content is negatively correlated with Langmuir Volume, while the I/S mixed layer content and the chlorite content are respectively first negatively and then positively correlated with Langmuir Volume.

4.3 Analysis of the Adsorption State of Methane Molecules in Organic Matter and Clay Minerals

4.3.1 Distribution of "Organic Matter Surface," "Clay Mineral Surface," and "Adsorption Sites"

The adsorption site distributions of methane molecules adsorbed on organic matter surfaces and clay mineral surfaces have been studied previously. It is believed that organic matter has a denser distribution of adsorption sites on the surface and thus features a higher adsorption capacity than clay minerals (Chen et al., 2017a; Chen et al., 2018a; Wang et al., 2020a; Gao et al., 2020). The distribution of Gas molecules is discontinuous on the surface of clay minerals, whereas it's continuous on the surface of organic matter (Chen et al., 2016a; Chen et al., 2017b; Chen et al., 2019a; Chen et al., 2019b; Gao, 2021).

4.3.2 Difference in Specific Surface Area of Organic Matter and Clay Minerals

The specific surface area of each component in the shales influences the adsorption capacity significantly, with their influence ranked in descending order as organic matter > montmorillonite > I/S mixed layer > kaolinite > chlorite > illite > calcite > feldspar > quartz. As the specific surface area of organic matter is obviously higher than that of clay minerals, the adsorption capacity of organic matter is higher than that of

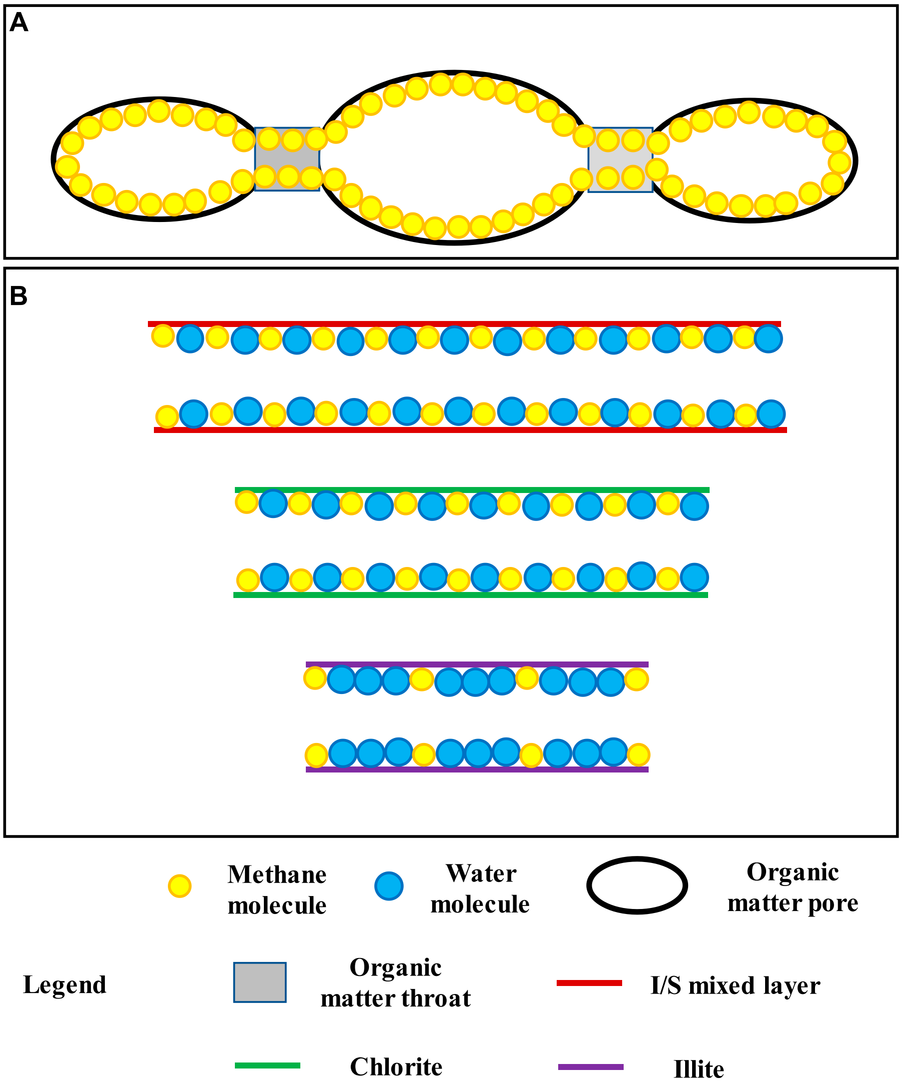

FIGURE 5 | Pattern of methane molecule adsorption on different media in highly evolved marine shales. **(A)** Methane molecules adsorbed on the internal surface of organic matter pores; **(B)** methane molecules adsorbed on the internal surface of clay minerals, which are mainly montmorillonite, I/S mixed layer, and chlorite.

clay minerals under the same unit mass (Chen et al., 2018b; Yu et al., 2022).

4.3.3 Differences in the Lipophilicity and Hydrophilicity of Organic Matter and Clay Minerals

The water content of the formation has a strong influence on the methane adsorption capacity of shales as adsorption sites for methane molecules can be occupied by water molecules. The clay minerals are hydrophilic, while the organic matter is lipophilic. Therefore, it's easier for methane molecules to be adsorbed on the surface of organic matter (Chen et al., 2016b; Wang et al., 2019). Generally, there is water in subsurface shale reservoirs, and the methane adsorption capacity of clay minerals is inhibited under actual subsurface reservoir conditions since clay minerals are hydrophilic. They will show their adsorption capacity only when their content reaches a sufficient level. As shown in **Figure 3**, in addition to adsorbing water molecules, I/S mixed layer and chlorite can also provide a certain amount of adsorption space for methane molecules to adsorb a considerable amount of these molecules. By contrast, illite mainly absorbs water molecules and cannot provide space for methane molecules adsorption.

4.4 Spatial Characteristics of Methane Molecules Adsorption in Organic Matter and Clay Minerals

4.4.1 FIB-SEM Observation

The pore characteristics of the shales can be directly observed with an SEM. As is shown in the FIB-SEM images, the largest greyscale is the pore, while the greyscale of every material composition in the shales becomes lower as its molecular weight is smaller (Zhang et al., 2019b; Zhang et al., 2019c; Wang et al., 2020b; Xia et al., 2020; Zhu et al., 2020; Huang et al., 2021; Wang et al., 2021), indicating that the grey scale of organic matter in the FIB-SEM images is larger than the one of inorganic minerals. It can be seen from the FIB-SEM images A and B of **Figure 4** that a large number of organic matter pores are developed in the shales with excellent roundness. Additionally, it can be seen from the FIB-SEM images **Figures 4E,F** that clay mineral pores are also developed in the shale and feature-poor roundness as they are lamellar and triangular.

4.4.2 FIB-HIM Observations

The situation inside the pores can be observed from the FIB-HIM images, making the two-dimensional images demonstrate three-dimensional effects. The greyscale of the FIB-HIM image is opposite to the one of the FIB-SEM images as the greyscale of every material composition in the shales becomes higher as its molecular weight is larger, indicating that the greyscale of the organic matter in the FIB-HIM image is lower than the one of the inorganic minerals (Zuo et al., 2019; Huang et al., 2020a; Huang et al., 2020b; Zhang et al., 2020b; Zhang et al., 2020c; Liu et al., 2021a; Liu et al., 2021b). It can be seen from C and D of **Figure 4** that the organic matter pores contain a large number of small pores like a hive with good connectivity.

5 ADSORPTION PATTERNS OF METHANE MOLECULES ON DIFFERENT MEDIA IN HIGHLY MATURE MARINE SHALES

The patterns of methane molecule adsorption states on different media in highly evolved marine shales are summarized based on the studies hereinabove. As shown in **Figure 5A**, the organic matter pores are sub-circular in shape with good connectivity between the organic-matter pores, and their internal surface can densely and continuously adsorb methane molecules. Besides, the specific surface area of the organic-matter pores is large and provides larger space for methane molecule adsorption. As shown in **Figure 5B**, the clay mineral pores are lamellar in shape. The surface of the clay mineral pores is loose, with adsorbed methane molecules discontinuously distributed on it. Besides, the clay mineral pores are more likely to contain water. Among the three clay minerals present in the Longmaxi Formation shale in the study area, I/S mixed layer has the largest specific surface area and can provide more adsorption space for methane molecules, followed by chlorite and finally illite. Due to the hydrophilic nature of the clay minerals, I/S mixed layer and chlorite adsorb water molecules in addition to methane molecules. Illite mainly adsorbs water molecules, and a small number of methane molecules.

6 CONCLUSION

In this paper, the shales from Member I of the Longmaxi Formation in the southern Sichuan Basin were used as the research object, and the state of methane molecule adsorption on different media in marine shales was analyzed *via* mineral composition analysis, TOC content analysis, isothermal adsorption experiments, FIB-SEM experiments, and FIB-HIM experiments by adopting core samples from the newly drilled shale gas exploration wells. The conclusions are as follows:

1) The adsorbed gas mainly exists in organic-matter pores, which feature excellent roundness and connectivity with a large number of small pores inside like a hive. The surface of organic matter contains many adsorption sites, featuring high adsorption capacity and continuous distribution of methane molecules on the internal surface of organic-matter pores. The specific surface area of organic matter is large, providing large space for methane molecule adsorption. Organic matter exhibits lipophilic properties, which makes it easier to adsorb methane.
2) Part of the adsorbed gas exists in clay mineral pores. I/S mixed layer and chlorite can not only adsorp water molocule but also provide certain space for the adsoption of methane molecule. Illite mainly adsorbs water molecules, and it is basically unable to provide adsorption space for methane molecules. Clay mineral pores are lamellar and triangular with poor roundness. Compared with organic matter, the surface of clay minerals contains fewer adsorption sites, featuring lower adsorption capacity and discontinuous distribution of methane molecules on the surface of clay minerals. Clay minerals have a smaller specific surface area, providing smaller space for adsorption. Clay mineral pores are more likely to contain water as clay minerals are hydrophilic.

DATA AVAILABILITY STATEMENT

The raw data supporting the conclusions of this article will be made available by the authors, without undue reservation.

AUTHOR CONTRIBUTIONS

LJ, YS, and LW contributed to conception and design of the study. LJ organized the database. YS performed the statistical analysis. LJ, YS, and LW wrote the first draft of the manuscript. ZC, HZ and FH wrote sections of the manuscript. All authors contributed to manuscript revision, read, and approved the submitted version.

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Conflict of Interest: Authors LJ, YS, WL, HZ, and FH are employed by PetroChina. Author ZC is employed by China National Petroleum Corporation.

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