Research Article

The Evolution of Diagenetic Fluids and Accumulation Characteristics of Tight Sandstone Reservoir in Upper Paleozoic, Southwestern Ordos Basin

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Received 19 January 2021; Accepted 4 May 2021; Published 15 June 2021

Academic Editor: Basim Abu-Jdayil

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The Upper Paleozoic in the southwestern Ordos Basin has significant potential for natural gas exploration. This study investigated the diagenetic fluid evolution and hydrocarbon accumulation characteristics of He 8 section from Permian Lower Shihezi formation and Shan 1 section from Shanxi formation tight sandstone reservoirs by petrographic observation, scanning electron microscope imaging, fluid inclusion study, and laser Raman spectrum analysis. The results show that He 8 section and Shan 1 section reservoirs are mainly composed of quartz sandstone, subordinate arkose quartz sandstone, and lithic quartz sandstone, with minor lithic sandstone and lithic arkose sandstone. The major pores are intergranular dissolved pores. The main diagenetic minerals include quartz overgrowth, siliceous cement, carbonate cement, illite, montmorillonite, and mixed-layer clay minerals. The overall diagenetic features show strong compaction, multistage siliceous and calcareous cements, and abundant clay minerals, strong dissolution, and well-developed fractures. Two stages of fluid inclusions developed in the He 8 and Shan 1 sections recorded the migration and accumulation of the early-stage and late-stage natural gas, respectively. The reservoir in the study area experienced early and late diagenetic stages, and its formation was simultaneous with or after its densification. The diagenetic environment changed from alkaline to acidic and again into alkaline. There are two stages of fluid activities in the study area, namely, the early diagenetic stage corresponding to hydrocarbon generation and migration and the late diagenetic stage corresponding to hydrocarbon accumulation. This study suggests that Upper Paleozoic natural gas migrated into the reservoir in Weibei Uplift, Yishan Slope, and Tianhuan Depression tectonic units during 220–197 Ma, and the large-scale migration and accumulation occurred in these tectonic units at different times. No natural gas was generated in the west margin of the basin because the temperatures of the hydrocarbon source rocks in the Upper Paleozoic were below the gas window.

1. Introduction

Fluid-rock interaction, throughout the basin formation and evolution, generally controls the diagenesis and mineralization in the basin and is closely related to hydrocarbon accumulation and occurrence [1]. Fluid inclusions are independent closed systems formed by fluids sealed in crystallographic defects of minerals [2], and they commonly record plenty of information on temperature, salinity, pressure, and composition related to the oil and gas migration processes and geological events [3–9]. Therefore, the study of fluid inclusions can be used to reverse the formation and evolution history of oil and gas reservoirs [10–13], and it has become an important and mature method for hydrocarbon accumulation research.

Exploration practice has revealed that the Upper Paleozoic of the Ordos Basin gas field is rich in oil and gas resources, with several large and medium-sized gas fields discovered successively in Yulin, Sulige, Wushenqi, Shenmu, Daniudi, and Zizhou have been proved successively in Upper Paleozoic [14–23]. Previous studies have been mainly focused in the central, western, northern, eastern, and southeastern parts of the basin. Recently, in the southwest of the basin, some exploration wells have obtained industrial gas

FIGURE 1: Locations of wells and regional structural location map in the study area.

flow [24], such as the open airflow of $6.62 \times 10^4 \text{ m}^3/\text{d}$ from Qingtan 1 well in the Shanxi formation, indicating a good prospect for natural gas exploration in the Upper Paleozoic in the southwestern basin. However, the study on the Upper Paleozoic in the southwestern basin is limited with controversial opinions. Zhang et al. [24] believed that the late Cretaceous oil and gas filling in this area was around the paleostructure of the Shan 1 section in the late Jurassic, and it is a near-source or in situ accumulation, whereas Cao et al. [25] suggested that the migration and accumulation of natural gas in the Upper Paleozoic in the southwest part of the basin was a relatively long and continuous process, with the main filling period during the Early Cretaceous (155–100 Ma). Liao et al. [26] argued that a primary natural gas charging occurred in the northern part of this area and the filling and accumulation lasted from late Jurassic to early Cretaceous (J_3 – K_1). Hu et al. [27] believed that the hydrocarbon generation started from the Late Triassic (T_3) and ended at the end of the Early Cretaceous (K_1). Moreover, the properties of the gas reservoirs in the southwestern basin are strongly disputed, such as the timing of natural gas accumulation, the types and stages of diagenesis, the processes of gas migration and accumulation, and the diagenetic fluids.

This study investigated core samples from the exploration wells by petrographic examinations, scanning electron microscope imaging, measurement of fluid inclusion homogenization temperature, and laser Raman spectroscopy analy-

sis. The findings will not only better characterize the reservoirs and the fluid inclusions, but also help reveal the diagenetic setting and evolution and the timing of gas accumulation and reservoir densification.

2. Geological Setting

The Ordos Basin, the second-largest petroliferous basin in China, is one of the most important basins abundant in oil and gases in China [28, 29]. It consists of six secondary structural units, namely, the Yimeng Uplift, the Yishan Slope, the Tianhuan Depression, the Weibei Uplift, the Jin-West Knit-belt, and the Xiyuan Thrust belt (Figure 1). The study area, with an area of $3.8 \times 10^4 \text{ km}^2$, situates in the southwestern part of the basin (Figure 1) involving four tectonic units [30], i.e., the Yishan Slope, Weibei Uplift, Tianhuan Depression, and Western Thrust belt. The strata are generally flat with small-scale faults developed in the Xiyuan Thrust belt.

As a large sedimentary basin in the west of the North China block, The Ordos Basin has experienced multistage tectonic evolution, including the Middle-Late Proterozoic Craton valley stage, the Early Paleozoic submarine platform, Late Paleozoic coastal plain, Mesozoic foreland basin, and Cenozoic peripheral fault-depression [31–33]. The main target strata of this study are the He 8 section of Xiashihezi Formation and Shan 1 section of Shanxi Group. The basin was an inland lake setting during the sedimentation of the He 8

FIGURE 2: Lithological histogram of Shihezi Group and Shanxi Group in the study area [24].

section, while it changed to offshore plain swamp and delta setting during the sedimentation of the Shan 1 sections (Figure 2).

The strata in the Ordos Basin, from bottom to top, include the Proterozoic, Paleozoic, Mesozoic, and Cenozoic sandstone, mudstone, limestone, and siltstone, with the absence of the Silurian and Devonian [32]. The basement is composed of Archean and Early Proterozoic metamorphic rocks. Due to the Caledonian Orogeny, the Upper Paleozoic strata in the studied area were subjected to uplift and erosion and then began to sink as a whole during the Middle-Late Carboniferous. These strata include the Carboniferous Benxi Group, the Permian Taiyuan, Shanxi, Upper Shihezi, Lower Shihezi, and Shiqianfeng formations [34]. The coal and dark mudstone of the Lower Shihezi formation in the He 8 section and the Shanxi formation in the Shan 1 section are the main source rocks for the Upper Paleozoic gas deposits. The sandstone in these two formations can be reservoirs, and the overlying marine limestone of the Taiyuan Formation, the argillaceous rocks of the Shiqianfeng, and the Upper Shihezi formations can be stable cap layers [17, 35]. These strata form a favorable source-reservoir-cap system in the Upper

Paleozoic. The He 8 section distributes as north-south strips with multiphase superposition in the longitudinal direction [36–38]. The sand body of the He 8 section has a thickness of 10–30 m, up to 60 m locally, and width of 10–30 km and extends up to more than 200 km. Sand and mudstone are interbedded in the He 8 section and Shan 1 section. Various sedimentary structures are developed in sandstone sections, such as parallel bedding and cross-bedding. The strong inter-layer heterogeneity and the above characteristics are favorable conditions for the formation of tight sandstone with low porosity and low permeability.

3. Samples and Experiments

In this study, more than 50 sandstone core samples of the He 8 section and Shan 1 section were collected from 12 exploratory wells in the southwest of the Ordos Basin. Among the samples, 30 chip samples were selected for petrographic observation from different depths of the Xiang 1 well, Qing-shen 2 well, He 3 well, Zitan 1 well, Ling 1 well, and Lian 1 well. Some thin sections were impregnated with red-dyed resin to identify diagenetic minerals and analyze their

FIGURE 3: Continued.

FIGURE 3: The typical diagenetic characteristics of sandstone reservoirs under microscope in the southwestern Ordos Basin. (a) Oriented mica and clastic particles and primary pores, Qingshen 2 well, Shan 1 section, 4733 m, (-); (b) bending deformation of clay minerals, Zitan 1 well, He 8 section, 3740.51 m, (+); (c) linear contact and concave-convex contacts between particles, He 3 well, Shan 1 section, 4146.16 m, (+); (d) concave-convex contacts of particles, He 3 well, He 8 section, 4079.88 m, (+); (e) quartz overgrowths and siliceous cements, Qingtan 2 well, Shan 1 section, 4705.68 m, (+); (f) authigenic quartz microcrystal, Zhentan 1 well, Shan 1 section, 4381.13 m, (-); (g) scaly illite aggregates filling intergranular pores, Qingshen 2 well, Shan 1 section, microscope; (h) flaky illite filling intergranular pores, preserving morphology of feldspar and other clastic particle. Zhentan 1 well, Shan 1 section, microscope; (i) vermicular kaolinite filling intergranular pores, Zhentan 1 well, Shan 1 section, SEM; (j) calcite cement, Ling 1 well, Shan 1 section, 3698.60 m, (+); (l) micrite calcite cement, He 3 well, Xiashihezi section, 4081.68 m, (+); (m) feldspar metasomatized by micrite with remnant twinning, Zhentan 1 well, Shan 1 section, 4379.07 m, (+); (n) metasomatic micrite and clay minerals with the morphology of lithic particles, Lian 1 well, Shan 1 section, 3469.69 m, (+); (o) quartz metasomatized by sparry calcite cement, He 3 well, Shan 1 section, 4146.62 m (+); (p) lithic particles metasomatized by flaky illite, He 3 well, Shan 1 section, 4146.62 m (+); (q) kaolinite altered from volcanic materials (K) and illite (Y), Lian 1 well, Shan 1 section, 3470.03 m, (-); (r) kaolinization altered from volcanic materials, He 3 well, Shan 1 section, 4146.23 m, (-); (s) dissolution pores of the clay and calcareous cements, Zhentan 1 well, Shan 1 section, 4387.7 m; (t) dissolution pores of cement, Chuntan 1 well, He 8 section, 1723.73 m. All photomicrographs were under microscope unless otherwise specified. Mineral abbreviations: Y: foliated illite; Yi: scaly illite; Ca: calcite cement; K: volcanic material.

distribution under the optical microscope. Ten samples were selected for scanning electron microscopy (SEM) analyses, six samples for X-ray diffraction (XRD) analyses, and seven samples for electron probe microanalysis (EPMA).

A total of 20 samples that are mainly quartz sandstone and lithic sandstone from 7 representative wells (Lian 1 well, Ling 1 well, He 3 well, Zitan 1 Well, Qingtan 2 well, Qingtan 1 well, and Zhentan 2 well) were selected for microscope petrographic observation. We observed the color, size, and occurrence of inclusions under transmitted light and fluorescence and identified different types and stages of inclusions. All analyses were performed at the Xi'an Institute of Geology and Mineral Resources, China. Compositional analyses were conducted on 15 samples by ramnor-U1000 laser Raman molecular microprobe (Jobin-Yvon Instrumentation, France) under the conditions of 22°C and 65% humidity. The laser was operated at a wavelength of 514 nm with a laser exposure time of 15 s, a laser-beam spot size of approximately 1 μm , a spectral resolution of 0.14 cm^{-1} , and output power of 22 mW. The fluid inclusion homogenization temperatures of these 15 samples were determined by THMS600 heating and cooling stage (Linkam Scientific Instruments, UK), calibrated by standards to obtain an error of 0.1°C for temperatures below 100°C and no higher than 0.5°C for temperatures above 100°C [39].

4. Results

4.1. Reservoir Characteristics

4.1.1. Reservoir Rock Types. The Upper Paleozoic clastic rocks of the Xiashihezi Formation in the study area mainly com-

prise quartz sandstone, arkose quartz sandstone, lithic quartz sandstone, and lithic arkose sandstone, with minor lithic sandstone and arkose sandstone. The debris grains show close contact dominated by linear and concave-convex boundaries. These sandstones are subangular to subrounded and moderately to poorly sorted (Figure 3(d)), suggesting a moderate textural maturity of the Xiashihezi Formation. The Shan 1 section is mainly composed of quartz sandstone, with minor lithic quartz sandstone, lithic sandstone, and arkose sandstone (Figure 4), and most of the arkoses have been kaolinized. The clastic particles in tight sandstone are mainly subangular to subrounded and moderately to poorly sorted. Their contacts are dominated by linear boundaries with a few points or suture contacts (Figure 3(c)). These features suggest grain-supported cementation. Petrographic observations on all thin sections show the pore space in the studied area/samples includes primary intergranular pore, intergranular dissolution pore, intragranular dissolution pore, and autogenous mineral intergranular pore (Figure 5).

4.1.2. Diagenesis. Diagenesis has been regarded as the controlling factor of reservoir densification [40–49]. Based on comprehensive analyses through petrographic observation, SEM analysis, cathodoluminescence imaging, XRD analysis, and EPMA, the Upper Paleozoic in the southwestern Ordos Basin has undergone compaction, cementation, metasomatism, dissolution, and fracturing [50], of which compaction and cementation are important factors to form a dense reservoir in the study area [51–55]. Under a microscope, compaction is represented by bent plastic grains such as mica, broken rigid grains, linear or concave-convex interparticle contacts, and “pressure cracks” in quartz (Figures 3(a)–3(d)). Cements

FIGURE 4: Lithologic triangle map of Upper Paleozoic in the study area.

FIGURE 5: Characteristics of pore types in the Upper Paleozoic Erathem sandstone of the study area. (a) Intergranular pores and fractures, Zitan 1 well, He 8 section, 3736.19 m; (b) intergranular pores and intragranular pores, Zhentan 2 well, Shan 1 section, 2893.10 m; (c) residual intergranular pores and intergranular dissolution pores, Ling 1 well, Shan 1 section, 3469.69 m; (d) intergranular pores and intragranular pores, Qingtan 2 well, Shan 1 section, 4723.68 m.

include multistage siliceous and calcareous cements, illite, kaolinite, and mixed-clay minerals (Figures 3(e)–3(o)). Metasomatism and dissolution can improve the physical properties of reservoirs [56–59]. The metasomatism of quartz, feldspar, and lithics by carbonate and clay minerals and the dissolution of volcanic materials, clastic particles, and cements (Figures 3(p)–3(t)) formed illite, kaolinite, and secondary overgrowth of quartz [60]. In summary, the tight reservoir in the study area is strongly compacted with multistage siliceous and calcareous cements, a large number of clay minerals, strong dissolution, and well-developed fractures and fissures (Figures 3(a)–3(d)).

4.1.3. Diagenetic Mineral Characteristics. Diagenetic minerals mainly include siliceous cements, carbonate cements, authigenic illite, kaolinite, and mixed-layer clay minerals. The siliceous cements include mainly different forms of siliceous cements among the particles, secondary quartz overgrowth around particles, and authigenic microcrystalline quartz (Figures 3(e) and 3(f)). Under a microscope, the secondary quartz overgrowths are clean and transparent with few inclusions, and it can be divided into early and late diagenetic stages. Clay films/rings are common at the rims of the overgrowths.

Carbonate cementation is another main cementation in the reservoir, and it can block the pores of sandstone. Early carbonate cementation weakens or prevents compaction, providing favorable conditions for later dissolution to form secondary pores. Later, after carbonate corrosion, the reservoir's physical properties become better. In this study, two stages of calcareous cementation have been observed in this study under the microscope (Figures 3(k) and 3(l)): The early stage was dominated by micrite and iron-bearing calcite, most of which are irregular fine grains and micrite. Due to dissolution, they are in patchy shapes and filled the pores. The calcite content is relatively low and less than 3%. The late stage was dominated by sparry calcite, with a relatively high calcite content of 8% on average, which distributed in intergranular pores in forms of granular or poikilitic cementation. Based on EPMA and XRD data (Tables 1 and 2), iron-bearing dolomite is also a calcareous cement in this study area.

Illite is the most abundant clay mineral in the study area. Under the microscope, illite is mainly an altered product or cement in various forms such as fish scales, hairs, fibers, and flakes (Figure 3(g)). Under SEM, most illite appears as pore liners and fillings forms of network or bridge, while honeycomb aggregates occur as flakes or filamentous growth

TABLE 1: Whole-rock X-ray diffraction analysis of sandstone and clay in the Upper Paleozoic of the study area.

Well number	Depth (m)	Stratum	Sample number	Whole-rock X-ray diffraction analysis of mineral relative content (%)							Clay mineral relative content (%)	
				Amount of clay	Quartz	Ankerite	Graphite	Siderite	Anatase	Not detected	Illite	Kaolinite
Xiang 1 well	2554.77	Zhifang Formation	1	31	52	11		5	1	0	61.06	38.94
Qingtian 2 well	4731.5	Shan 1	2	26	65	5		2	1	1	67.00	33.00
He 3 well	4146.62	Shan 2	3	15	61	18	1	2	2	1	87.89	12.11
Zitan 1 well	3742.50	He 8	4	21	49	23		5	1	1	58.41	41.59
Ling 1 well	3697.14	Shan 1	5	37	44	14		2	1	2	66.55	33.45
Lian 1 well	3470.03	Shan 1	6	39	54	2		2	2	1	56.08	43.92
Average				28.3	54.2	12.2		3.0	1.3	1.0	66.17	33.83

TABLE 2: Electron probe microanalysis of the compositions of sandstone cements in the Upper Paleozoic Erathem in the southwest of study area.

Well number	Depth (m)	Stratum	Sample number	Na ₂ O	P ₂ O ₅	MnO	MgO	K ₂ O	Cr ₂ O ₃	Al ₂ O ₃	CaO	TiO ₂	SiO ₂	FeO	NiO	Mineral type
Qingtian 2 well	4731.5	Shan 1	1		0.023	0.535	20.66	0.02		0.088	49.84	0.071		28.75	0.002	Ankerite
He 3 well	4146.62	Shan 2	2		0.004		0.032	0.024	0.002				99.89	0.044		Quartz
Zitan 1 well	3742.50	He 8	3		0.014			0.012				0.026	99.87	0.012	0.058	Quartz
Ling 1 well	3697.14	Shan 1	4	0.026	0.018				0.038		0.011		99.87		0.028	Quartz
Lian 1 well	3470.03	Shan 1	5			0.11	3.14	3.3		33.6	0.19		45.7	11.4		Mixed-layer clay
Qingtian 2 well	4731.5	Shan 1	6				0.015	0.42		37.4	0.066	0.142	48.46			Kaolinite
He 3 well	4146.62	Shan 2	7				0.057	3.98		33.74		0.062	50.29	1.163		Kaolinite

on particle surface or in the pores (Figure 3(h)). Illite in this area is either authigenic or altered products formed by the dissolution of feldspar and volcanic materials. Generally, the transition of diagenetic fluid from acid to neutral is favorable for the formation of illite.

Kaolinite is a common clay mineral in the study area. It occurs in vermicular and leave-like shapes under SEM (Figure 3(j)), and some appear as microcrystalline aggregates under a microscope (Figure 3(i)). Kaolinite is usually formed by feldspar corrosion during water-rock interaction in an acid environment, and it also has a positive correlation with the secondary porosity. Thus kaolinite with well-developed intracrystalline pores as important reservoir space can indicate the formation of favorable reservoirs [61–64].

The mixed-layer/clay minerals in the study area are mainly illite-smectite, which is the middle product of the transformation of montmorillonite to illite. This transformation could only occur when the temperature reaches 60–100°C in an alkaline fluid environment with a large number of K⁺ and Al³⁺. Mixed-layer clay mineral contents in this area are not high, second to illite and kaolinite.

4.2. Fluid Inclusions

4.2.1. Inclusion Types. Through the microscopic observation of fluid inclusion distribution and occurrence, combined with diagenetic characteristics of the research area, fluid inclusions can be divided into two phases. The early phase inclusions are distributed in the pores and fissures and were produced by dissolution and fracturing, consisting mainly of gas-liquid organic inclusions and brine inclusions (Figures 6(a)–6(c)). The early cracks, where organic inclusions distributed along the fissures as a string of beads, did not cut the overgrowths of detrital particles. Single inclusions are nearly circular or elliptic. Inclusions in the fissures are generally larger, and their long axes are consistent with the orientation of the fissures (Figures 6(b)–6(d)). Under the fluorescence microscope, liquid hydrocarbon in the early organic inclusions emitted weak bright yellow fluorescence (Figures 6(e)–6(h)) but gaseous hydrocarbon did not fluoresce, suggesting liquid and gaseous hydrocarbon have occurred in the early diagenetic stage, and the gas/liquid ratios of early organic inclusions were 10% or so. Oil and

FIGURE 6: Microscopic characteristics of fluid inclusions in the sandstone reservoir of the study area. (a) Gas-liquid two-phase inclusions in dissolution pores, He 3 well, Shan 1 section, 4146.16 m; (b) oriented gas-liquid two-phase inclusions in fissures in early stage, Lian 1 well, Shan 1 section, 3467.72 m; (c) gas-liquid two-phase inclusions in dissolution pores, Lian 1 well, Shan 1 section, 3424.26 m; (d) oriented gas-liquid two-phase inclusions in fissures in early stage, Qingshen 2 well, Shan 1 section, 4732.6 m; (e) dark-colored hydrocarbon gas-liquid two-phase inclusions in the dissolution pores, He 3 well, Shan 1 section, 4146.62 m; (f) same view field of (e) under the fluorescence microscope; (g) dark hydrocarbon gas-liquid two-phase inclusions in early fissures, Lian 1 well, Shan 1 section, 3424.26 m; (h) same view field of G under the fluorescence microscope; (i) gas-liquid two-phase inclusions along late fissures, He 3 well, He 8 section, 4081.68 m; (j) gas-liquid two-phase inclusions with beaded fractures and directional distribution, Qingshen 2 well, Shan 1 section, 4722.8 m; (k) late dark gas-rich inclusions, Lian 1 well, Shan 1 section, 3424.26 m; (l) gas-rich inclusions with fracture distribution in late stage, Qingshen 2 well, 4730.14 m.

gas filling strength was weak and distributed and migrated along the dissolution pores and fissures.

The late inclusions are distributed in dissolution pores and fissures and formed in the late diagenetic stage. Most late inclusions are gas-liquid inclusions with a few pure gas-phase inclusions. They are elongated and distributed along the crack extension or as a string of beads along the fissure (Figures 6(i)–6(l)). The gas/liquid ratios of gas-liquid organic inclusions were generally greater than 20%, suggesting that

the strength of the gas filling was high and reached the hydrocarbon generation peak. A large amount of natural gas has migrated and accumulated in favorable locations.

4.2.2. Inclusions Composition. Laser Raman microprobe analyses of inclusions from the Xiashihezi and Shanxi Groups in the southwestern Ordos Basin have revealed both organic and inorganic components (Figure 7). In the different phases of inclusions (Tables 3 and 4), gas-phase inclusions have

FIGURE 7: Continued.

FIGURE 7: Laser Raman spectrogram of fluid inclusions in sandstone reservoir in the study area. (a) Qingtan 2 well, Shanxi Group, 4722.80 m; (b) Qingtan 2 well, Shanxi Group, 4722.80 m; (c) Qingtan 2 well, Shanxi Group, 4728.08 m; (d) Lian 1 well, Shihezi Group, 3424.26 m; (e) Lian1 well, Shihezi Group, 3424.26 m; (f) He 3 well, Shihezi Group, 4073.09 m; (g) He 3 well, He 8 section, 4081.68 m; (h) Zitan 1 well, Shihezi Group, 3736.19 m; (i) Zitan 1 well, Shihezi Group, 3740.51 m.

major organic ingredients of CH_4 and inorganic components of CO_2 in addition to H_2S and N_2 , with H_2O contents varying in a wide range. In liquid phase inclusions, H_2O content is greater than 95% but CO_2 content is less than 2%, whereas CH_4 is the only organic component. However, in this area, no hydrocarbon gas components such as C_2H_4 , C_2H_6 , H_2S , C_2H_2 , and C_4H_6 were detected in the organic inclusions, indicating that the natural gas had a high degree of thermal evolution in the hydrocarbon generation, reflecting the feature of “dry gas” [65].

Comparing the early and late organic inclusions of sandstone reservoirs in Upper Paleozoic in the study area (Figure 8), the CO_2 and CH_4 contents of gas-phase organic inclusions were higher than those of liquid-phase inclusions, whereas the individual contents of CO_2 and CH_4 were a trade-off. Thus, the early inclusions were rich in CO_2 and low in CH_4 , but the late inclusions were rich in CH_4 and low in CO_2 . Different stages of inclusions captured in the same sample have different CO_2 and CH_4 contents, indicating that the degree of thermal evolution of the hydrocarbon gas was relatively low in the early diagenetic stage but higher in the late diagenetic stage.

The homogenization temperatures of inclusions in the early stage of the He 8 and Shan 1 sections vary from 80°C to 200°C in distinct ranges (Figure 9). In the He 8 section, the homogenization temperatures of the early inclusions range from 80°C to 140°C with the peak from 120°C to 130°C. In the Shan 1 section, the homogenization temperatures of late inclusions range from 110°C to 200°C with the peak from 150°C to 160°C. The uniform continuous distribution of the homogenization temperature of the two stages of fluid inclusions in the He 8 and Shan 1 sections indicates that the oil and gas generation, migration, and accumulation was a continuous filling process. The early inclusions recorded the natural gas generation and migration into the reservoir, while the late inclusions witnessed the accumulation in the reservoir. Based on the freezing temperature, the salinity of the fluid inclusions was calculated. The results show consistent salinity distribution of fluid inclusions in the He 8 and

Shan 1 sections between 0 and 17 wt%. The peaks of 4 wt% NaCl and 8 wt% NaCl in the two major peak ranges correspond to the homogenization temperature analysis, confirming the existence of two stages of fluid inclusions. Besides, the salinity of the early inclusions is relatively higher than that of the late inclusions, indicating that the reservoir had better sealing during the formation of the early inclusions as opposed to the formation of late inclusions.

5. Discussion

5.1. Diagenetic Events and Characteristics. On the basis of authigenic minerals, the relationship of metasomatism and dissolution-filling of different cements, features of fluid inclusions, and uniform homogenization temperature distribution, the He 8 section and Shan 1 section experienced early and late diagenetic stages and are currently in the late diagenetic stage. Combined with burial history from the study area and its adjacent regions [22, 66–68], the diagenetic evolution of reservoirs in the He 8 and Shan 1 sections has been further studied. During the burial process, the diagenesis in the study area was dominated by mechanical compaction due to the increased burial depth and overlying sediments. Clastic particle contacts are close, and plastic minerals were squeezed due to deformation while rigid minerals were dissolved and ruptured (Figures 3(b) and 3(c)). The compression of mudstone released Ca^{2+} and HCO_3^- in the early stage, forming alkaline diagenetic fluids that were favorable for the formation of early siliceous and calcareous cements [67]. With the increase of burial depth, temperature, and pressure increased, when mica reacted with pore water [69], potassium ions were released and entered the pore solution. Hydrolysis of volcanic tuffaceous resulted in the release of alkali metal ions such as Na^+ and K^+ . All of those led to increased pH values of the pore fluids, promoting a more alkaline diagenetic environment [41], in which authigenic illite gradually precipitated (Figures 3(g) and 3(h)). At this time, organic matter was immature and the temperature

TABLE 4: Results of laser Raman analysis of liquid phase composition of Paleozoic inclusions in southwestern Ordos Basin (molar relative content %).

Well number	Sample number	Description	Depth(m)	Stratum/lithology	CO ₂	H ₂ S	CH ₄	SO ₂	H ₂ O	Cl ₂	O ₂	CO	F ₂	Gas phases%											Total
														N ₂	H ₂	C ₂ H ₂	C ₂ H ₄	C ₂ H ₆	C ₃ H ₆	C ₃ H ₈	C ₄ H ₆	C ₆ H ₆			
Qingtian 2 well	5-1	Late stage	4722.8	Shanxi formation/magenta medium-coarse quartz sandstone				0.04	99.96																100.0
	5-2	Early stage	4722.8						100.00																100.0
	29	Early stage	4728.1	Shanxi formation/gray-white medium-coarse quartz sandstone	0.07				99.93																100.0
	35-1	Early stage	4729.4	Shanxi formation/gray-white medium-coarse quartz sandstone	0.12				99.88																100.0
	35-2	Late stage	4729.4						100.00																100.0
Lian 1 well	47	Late stage	4732.0	Shanxi formation/grayish-white gravel medium-coarse quartz sandstone			0.04		99.96																100.0
	Li98-1	Early stage		Shanxi formation/gray-black fine sandstone					100.00																100.0
	Li98-2	Late stage	3467.7		0.07				99.93																100.0
	Li93-1	Late stage		Shanxi formation/gray-white medium-fine-grained sandstone	2.03		1.37		96.60																100.0
Zitan 1 well	Li93-2	Late stage	3423.3																						100.0
	TZ1,116-2	Early stage	3736.2	Shihezi Group/gray-white fine sandstone					100.00																100.0
	132-1	Late stage	4073.1		0.17				99.80																100.0
He 3 well	132-2	Early stage	4073.1	Shihezi Group/gray-white medium-fine lithic sandstone			0.02																		100.0
	138-1	Early stage	4081.7				0.06		99.94																100.0

Dark single gas phase

Dark single gas phase

FIGURE 8: Comparison of gas-phase CO₂ and CH₄ contents compositions of early and late organic inclusions between He 8 section and Shan 1 section sandstone reservoir in the study area.

FIGURE 9: Homogenization temperature and salinity distribution profiles of sandstone inclusions in the Upper Paleozoic Erathem in the study area.

was less than 80°C, further confirming that the diagenetic fluid was alkaline [67].

In the late period of the early diagenetic stage, organic matter matured and gas production began, resulting the fluid environment gradually became less alkaline but more acidic. The acidic fluids led to metasomatism of feldspar, lithic, volcanic, and calcareous cements as well as the dissolution of feldspar (Figure 3(n)) and volcanic materials, forming kaolinite and quartz secondary overgrowth. The

dissolution pores of feldspar were filled with organic inclusions (Figures 6(a) and 6(b)), indicating that the acid dissolution was ahead of early oil and gas filling. Under appropriate conditions, SiO₂ dissolved from feldspar in an acidic environment formed quartz secondary overgrowth around debris particles. The pure gas phase of the second phase fluid inclusions (Figures 6(k) and 6(l)) and the laser Raman spectroscopy analysis results, as well as the vitrinite reflectance (Ro) greater than 1.0% in Permian mudstone, all together

suggest a high degree of thermal evolution (Figure 7). In the study area, micritic calcite and sparry calcite replaced/metamorphosed quartz, feldspar, and other debris particles (Figures 3(n)–3(p)), some of which were early phase quartz overgrowth and dissolved feldspar. This relationship suggests that calcite cementation was after feldspar dissolution and quartz overgrowth and also later than oil and gas filling. The fissures with late inclusions cut quartz edge but not the late cements that formed at the late diagenetic stage without inclusions, suggesting that large-scale migration and accumulation of gas happened before reservoir cementation. In Upper Paleozoic, gas accumulation time was not later than the time of reservoir densification, meaning that the gas was accumulated simultaneously with or after the reservoir densification.

In conclusion, the reservoir diagenetic evolution sequence can be summarized in the following order: (1) compaction/early siliceous and calcareous cement; (2) formation of authigenic clay minerals and organic acid; (3) start of natural gas filling; (4) early dissolution and metasomatism including dissolution of carbonate, particle, feldspar, and volcanic debris and formation of illite and kaolinite and quartz secondary overgrowth; (5) a large amount of natural gas filling; (6) dissolution of late-stage feldspar and calcareous cement; (7) late-stage calcite metasomatizing clay minerals, quartz, and feldspar (Figure 10).

5.2. Evolutionary Sequence of Diagenetic Fluids

5.2.1. Fluid Evolution of Early Diagenetic Stage.

Based on Petromod 1D and related simulation parameters of The Ordos Basin [70], the tectonic burial history of the Qingtan 1 well was established (Figure 10). The early Permian strata underwent early burial diagenesis until the late Jurassic period (145 Ma) which belonged to the early diagenetic stage (288 Ma–211 Ma). During this period, compaction was predominant and the intergranular contacts vary from linear to concave-convex. Some early siliceous and calcareous cements began, reducing the pore space greatly. The organics of mudstone were immature in the alkaline diagenetic fluid environment. As the strata continued subsiding with the rise of temperature and pressure, the pore fluid gradually changed from alkaline to acidic. When buried depth was over 2500 m, the strata entered the late diagenesis stage (211 Ma–145 Ma). At this time, the thermal evolution of strata reached a mature stage and began to produce gas. Oil and gas began filling and migrating, and the organic acids and methane gas made the pore fluid acidic. During this stage, diagenesis was given priority to dissolution and metasomatism. The dissolution of feldspar and rock debris, volcanic materials, and early carbonate cements produced secondary porosity such as dissolution pores, which consequently improved the reservoir's physical properties. The formation of illite and kaolinite and quartz overgrowth at particle edges reduced the porosity of the reservoir, leading to the reservoir densification.

5.2.2. Fluid Evolution of Late Diagenetic Stage.

The early Cretaceous late diagenetic stage (145 Ma–48 Ma) can be further divided into two phases, 145 Ma–95 Ma and 95 Ma–48 Ma.

From Late Jurassic to Early Cretaceous, a regional thermal anomaly event occurred in the Ordos Basin [71–73], resulting the Upper Paleozoic source rocks reached the highest degree of thermal evolution. The source rocks reached the peak of gas generation while oil and gas filling continued and accumulated in the reservoir. The concurrent diagenetic environment was acidic, and the diagenesis was characterized by the dissolution of feldspar and calcareous cements, which increased the secondary porosity. The second phase from 95 Ma to 48 Ma was dominated by strata uplift and erosion caused by the Yanshanian movement, during which the geothermal gradient and strata temperature decreased. The burial depth of gas source rocks became shallow and the organic became less acidic. The release of internal pressure produced fractures and cracks, which may become the channels of gas migration and places of accumulation. The diagenetic environment during this phase was weak alkaline. The main diagenesis included cementation and metasomatism, which severely lowered the reservoir's physical properties, and thus, the reservoir was further densified.

In Paleogene from 48 Ma to 30 Ma, the strata were uplifted due to the Himalayan movement, and the hydrocarbon generation gradually weakened. The concurrent diagenetic environment was alkaline. During this period, quartz and feldspar particles were metasomatized by late calcite, leading to the reduction of reservoir pore flow channels and worse physical properties, and hence further densified reservoir.

Based on the diagenesis and accumulation characteristics of different periods in the study area, the diagenetic environment was transitional from alkaline to acidic and then to alkaline. This corresponds well with the diagenetic evolution sequence concluded above.

5.3. Investigation of Fluid Characteristics.

Fluid plays an important role in the formation of sedimentary basins and the formation, migration, and accumulation of hydrocarbon gases. It participates in diagenesis and improves reservoir performance. Through the analyses of fluid inclusions, diagenetic fluid evolution, and diagenesis, we can further understand the fluid properties and the characteristics of active stage. The results indicate the two generations of paleo-fluid flows corresponding to the early and late diagenetic stages. The first generation of paleo-fluid flows is represented by fluid inclusions filling in the pores and fractures of the early diagenetic sandstone, recording the generation and migration of early oil and gas. The second generation of paleo-fluid flows filled in the late diagenetic sandstone pores and fractures, recording the late hydrocarbon accumulation.

Fluid inclusion analysis shows their homogenization temperatures (80–200°C) and salinities (0–17 wt%) are continuously distributed over wide ranges. The homogenization temperature peak of the early fluid is 120–130°C, and the salinity peak is 4 wt% NaCl. The homogenization temperature peak of the late fluid is 150–160°C, and the salinity peak is 8 wt% NaCl. These data suggest the diagenetic fluid is in the range of medium-low temperature and moderate-low salinity [39], which is dominated by strata water. According to the initial melting temperature of the inclusions, the

FIGURE 10: The diagenetic evolution sequence and accumulation history of the sandstone reservoir in the Upper Paleozoic Erathem of the Qingtan 1 drilling well.

paleo-fluid properties recorded by the Upper Paleozoic fluid inclusions can be determined. There are four types of brine systems in the southwestern part of the basin: (1) $\text{CaCl}_2\text{-H}_2\text{O}$ system, (2) $\text{CaCl}_2\text{-NaCl-H}_2\text{O}$ system, (3) $\text{CaCl}_2\text{-H}_2\text{O}$ system, (4) $\text{MgCl}_2\text{-H}_2\text{O}$ system, and (5) $\text{MgCl}_2\text{-NaCl-H}_2\text{O}$ system, of which the $\text{CaCl}_2\text{-H}_2\text{O}$ and $\text{MgCl}_2\text{-NaCl-H}_2\text{O}$ systems are dominant. It is considered that the formation environment and migration and storage conditions of Upper Paleozoic

$\text{MgCl}_2\text{-H}_2\text{O}$ system, and (4) $\text{MgCl}_2\text{-NaCl-H}_2\text{O}$ system, of which the $\text{CaCl}_2\text{-H}_2\text{O}$ and $\text{MgCl}_2\text{-NaCl-H}_2\text{O}$ systems are dominant. It is considered that the formation environment and migration and storage conditions of Upper Paleozoic

FIGURE 11: Natural gas accumulation time of the Upper Paleozoic in the study area recorded by fluid inclusions.

natural gas in the study area are medium. Based on the analysis of diagenesis and diagenetic fluid evolution in different periods of the study area, the diagenetic environment of the study area has experienced the transition from alkaline through acidic to alkaline.

5.4. Accumulation Time. Accumulation stages can be determined through the study of natural gas inclusions, which are the key evidence for the process of oil and gas migration or accumulation [74]. In this study, based on the diagenesis and inclusion petrography, the natural gas accumulation in the study area is divided into natural gas formation and migration stage and massive accumulation stage.

The specific time of natural gas migration and accumulation represented by inclusions can be determined by comparing the inclusion homogenization temperatures (T_h) against the burial history curves of exploratory wells in different secondary tectonic units across the Ordos basin. In detail, the Xuntan 1 well represents the Weibei Uplift, the Ningtan 1 well and Hetan 1 well represent the Yishan Slope, the Huan 14 well represents the Xiyuan Thrust belt, and the Zhentan 2 well represents the Tianhuan Depression (Figure 11).

According to the results of inclusion homogenization temperatures, the low temperature (120°C – 130°C) inclusions are the first-stage inclusions, which are distributed in the early fissures or dissolution pores. They belong to hydrocarbon inclusions in the initial gas migration to the reservoir, and some inclusions have fluorescence. The high-temperature inclusions (150°C – 160°C) are the second-stage inclusions, belonging to the nonfluorescent natural gas inclusions formed during the massive gas migration and accumulation in the tectonic uplift stage. Considering the gas “window” is $R_o = 1.30\%$ for the Upper Paleozoic source rocks in the southwest of Ordos Basin, large-scale gas generation could only occur when the paleogeothermal temperature was higher than 170°C . However, the paleogeothermal temperature of the Upper Paleozoic in the Huan 14 well did not reach this temperature (Figure 11(a)), so the thermal evolution of the Upper Paleozoic in the Huan 14 well did not enter the gas production stage. In other tectonic units, the paleogeothermal temperatures all reached the hydrocarbon generation windows, and hence, the intersections of early inclusion homogenization temperatures and the burial curves represent the formation time of hydrocarbon source rocks in the Upper Paleozoic.

The intersection of the late inclusion homogenization temperature curve and the uplift section of the well burial history curve (rather than the descending stage) represent the formation time of the inclusion/hydrocarbon source rocks. Following the same principle, natural gas in the Weibei uplift migrated into the reservoir at 205 Ma (late Triassic) and accumulated during 170–136 Ma as represented by the Xuntan 1 well (Figure 11(c)). The gas migration time within the Yishan Slope (Hetan 2 well and Ningtan 1 well) was 220–210 Ma (Figure 11(d)), and the accumulation time was during 170–142 Ma. Natural gas in the Tianhuan Depression (Zhentan 2 well) migrated into the reservoir at 197 Ma and accumulated during 164–152 Ma (Figure 11(b)). In conclusion, the gas migration time of the Upper Paleozoic reservoir in Weibei Uplift, Yishan Slope, and Tianhuan Depression tectonic units were 220–197 Ma, but the large-scale migration and accumulation time in these tectonic units varied.

6. Conclusion

- (1) In the southwest of the Ordos Basin, the He 8 section and Shan 1 section of the Upper Paleozoic reservoir consist mainly of quartz sandstone, arkose quartz sandstone, and lithic quartz sandstone. The diagenesis includes compaction, cementation, metasomatism and dissolution, and fissure, among which compaction and cementation are important factors for reservoir densification. The diagenetic process of the reservoir is featured by strong compaction, development of multistage siliceous and calcareous cements and abundant clay minerals, strong dissolution, and relatively well-developed fractures and fissures
- (2) The He 8 and Shan 1 sections of reservoirs both have two stages of inclusions. Both early and late inclusions of the He 8 section and Shan 1 section show similar peak temperatures that are between 120–130°C and 150–160°C, respectively. The early inclusions recorded the natural gas generation and migration into the reservoir, while the late inclusions witnessed the accumulation in the reservoir. The salinity of fluid inclusions in He 8 and Shan 1 sections ranged from 0 to 17 wt%, with the two main peak values of 4 wt% NaCl and 8 wt% NaCl
- (3) Diagenetic evolution of the He 8 and Shan 1 sections of the reservoir has gone through early and late diagenetic stages and is currently in the late diagenetic stage. The reservoir diagenetic evolution sequence can be summarized in order as follows: (1) compaction/early siliceous and calcareous cement; (2) formation of authigenic clay minerals and organic acid; (3) start of natural gas filling; (4) early dissolution and metasomatism including dissolution of carbonate, particle, feldspar, and volcanic debris and formation of illite and kaolinite and quartz secondary overgrowth; (5) a large amount of natural gas filling; (6) dissolution of late-stage feldspar and calcareous cement; (7) late-stage calcite metasomatizing clay minerals, quartz, and feldspar. The diagenetic envi-

ronment was transitional from alkaline through acidic and then to alkaline

- (4) Two periods of oil and gas filling occurred in the reservoir in the study area. The densification of the reservoir was simultaneous with or after the reservoir densification. There are two stages of fluid activity in the study area, namely, early diagenetic stage corresponding to hydrocarbon generation and migration and late diagenetic stage corresponding to hydrocarbon accumulation
- (5) The natural gas accumulation in the study area is divided into natural gas formation and migration stage and massive accumulation stage. The gas migration time of the Upper Paleozoic reservoir in Yishan Slope, Weibei Uplift, and Tianhuan Depression tectonic units was during 220–197 Ma, but the gas accumulation time in different tectonic units varied. However, in the western margin of the basin, no natural gas was generated because the hydrocarbon source rocks of the Upper Paleozoic in the western margin of the basin did not reach the gas “window.”

Data Availability

The X-ray diffraction (XRD) analyses, electron probe microanalysis (EPMA), fluid inclusion homogenization temperature, etc. data, and laser Raman spectroscopy analysis data used to support the findings of this study are included within the article.

Conflicts of Interest

The authors declare that they have no conflicts of interest.

Acknowledgments

This study was funded by the National Natural Science Foundation of China (number: 41772118) and the Fundamental Research Funds for the Central Universities, Chang'an University (No. 300102279106).

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