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Discovery of solid bitumen in the Cambrian reservoirs and its geological implications in the Ordos Basin, China

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This study reports a comprehensive geochemical analysis of several samples of the solid bitumen recently discovered in the Cambrian reservoirs in drillings and outcrops in the Ordos Basin. The results show that the solid bitumen in the reservoir features intergranular pores and micro-fractures, with mineral particle surfaces of dolomite and limestone, and relatively clear and flat boundaries. The D and G peaks in the laser Raman spectrum were prominent. According to distributions of shapes of the D and G peaks and the relevant parameters of the laser Raman spectrum, the vitrinite equivalent reflectance (*Roeq*) values were from 2.12% to 3.46% in the solid bitumen. This indicates that the solid bitumen in the reservoirs had undergone significant thermal evolution and was mainly composed of pyrobitumen. Moreover, it mainly consisted of four types of atoms, C, O, Ca, and Mg, which confirms the hypothesis of the significant thermal evolution of the samples. In addition, the analysis of the origin of the Cambrian natural gas and the correlation between the solid bitumen and the potential source rocks show that the solid bitumen in the Cambrian reservoirs of the Southern Basin had features similar to those of the Lower Cambrian source rocks, except for the samples from well T59. All evidence suggests that the high-abundance source rocks of the Lower Cambrian have made considerable contributions to the Cambrian natural gas and the solid bitumen in the Cambrian reservoirs of the Southern Basin. The work here provides a new scientific basis for exploring natural gas in the Cambrian reservoirs and deep strata of the Ordos Basin.

KEYWORDS

solid bitumen in the reservoirs, geochemical characteristics, geological implications, Cambrian, Ordos Basin

Introduction

In recent years, our extensive observations of core samples and reconnaissance of field outcrops have led to the discovery of solid bitumen in the Cambrian reservoirs of the Ordos Basin. Horizontally, they are chiefly distributed in the Southern Basin (Figure 1A); vertically, the samples of solid bitumen in the reservoirs are mainly distributed in the Middle–Upper Cambrian (Figure 1B). Most samples of solid bitumen in the reservoirs were found in dolomite or limestone, with a few in sandstones. In general, the solid bitumen cannot be easily identified in field outcrops or drilling cores (Peters et al., 2005; Yang Hua et al., 2010; Shifa, Z et al., 2016), yet some fresh samples have an "oily" smell. The solid bitumen can be better identified using experimental approaches, such as environmental scanning electron microscopy, energy-dispersive spectrometry, and laser Raman spectroscopy.

With the discovery the of low-rate natural gas stream from the Cambrian reservoirs in the Southern Basin, the Cambrian reservoir has attracted increasing attention from explorationists. In the Middle and Late Proterozoic, a series of rift troughs that were striking north and northeast direction were developed in the Ordos Basin (YANG Junjie, 2002). During the depositional period of the Early Paleozoic, due to the influence of the inherited development of rift troughs, a number of deep-water bays were developed in the Western and Southern basins, and most of the basins were uplift. Transgression occurred in the late of the early Cambrian, and littoral deposits were developed in the Western and Southern basins. The transgression continued to expand until the middle Cambrian, the platform margin, and deep-water trough deposits were developed in the Western and Southern basins. During the late Cambrian, the sea regressed, and slope and deep-water trough deposits continued to develop in the Western and Southern basins, while constrained platform deposits were in most areas of the basin (Bai et al., 2014; Dai. 2005; Du et al., 2019). The paleogeographical framework of the Cambrian makes the distribution of the Cambrian strata particularity in the Ordos Basin. The Cambrian strata are the thickest in the Western and Southern basins but thinner in the interior of the basin (Figure 1A). Moreover, due to the Caledonian orogeny, the Ordos Basin was subjected to uplifting and erosion to different degrees during the Cambrian Period (Du et al., 2019; Hu, W. et al., 2021; Zhao, T. et al., 2021) because of which the drillings and outcrops in the basin cannot fully reveal the complete Cambrian sedimentary sequence. In addition, no large-scale reservoirs of oil and gas have yet been discovered, owing to the poor exploration of the Cambrian system. The latest research on Cambrian source rocks in the Ordos Basin shows a set of high-abundance marine source rocks that developed in the Southern Basin during the Early Cambrian (ϵ_1), with a highest *TOC* of 11.18% (Du et al., 2019). In addition to that there is the lighter carbon isotopic composition of kerogen (−31.6 to −27.4‰) (Huang et al., 2020a) and relatively high

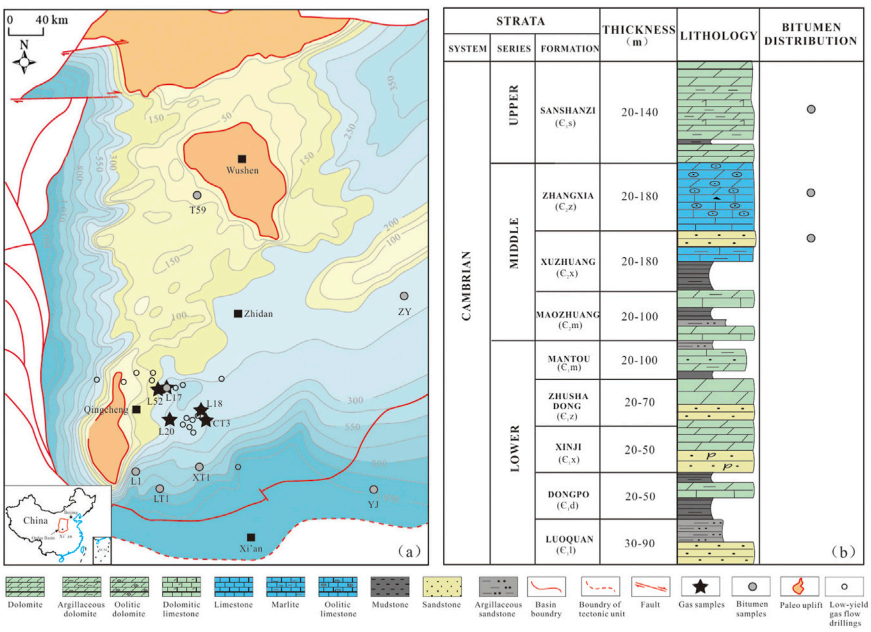

FIGURE 1
Thickness of Cambrian strata and sampling locations **(A)** and comprehensive stratigraphic column; **(B)** of the Cambrian at the southern part of the Ordos basin.

sterane/hopane ratios (S/H) (0.50–1.36) and tricyclic terpene/hopane ratios (TT/H) (0.70–1.13) (Huang et al., 2020b) in the Lower Cambrian source rocks of the Ordos Basin, which indicates that the source of the organic matter was dominated by lower algae. According to the organic matter type classification of kerogen carbon isotopic composition (Dai et al., 2005), the organic matter types of the Lower Cambrian source rocks are mainly type II, and some are type I. However, the Lower Cambrian source rocks are at high degrees of thermal evolution (Du et al., 2019; Huang et al., 2020a). Moreover, this set of source rocks has two stages of hydrocarbon generation and expulsion: the first large-scale hydrocarbon generation and expulsion period was in the Late Permian to the Middle Triassic, and then, the hydrocarbon generation stopped due to the uplift and denudation caused by the Indosinian Movement. In the Mesozoic and Cenozoic, the Ordos Basin subsides again, and there may be secondary hydrocarbon generation (Huang et al., 2020a). Thus, it has good prospects for natural gas exploration.

The solid bitumen in the Cambrian reservoirs provides direct evidence of hydrocarbon accumulation, and its presence is generally considered to indicate paleo-oil reservoirs or processes of hydrocarbon accumulation occurring through geological times. The reflectance of the Ordovician bitumen in the Ordos Basin is between 1.4% and 3.8% (Liu et al., 2009), demonstrating a highly mature stage. Generally, thermal maturity increases as burial depth increases. Also, the solid bitumen in the Cambrian reservoirs is buried deeper than the Ordovician bitumen, which indicates that the degree of thermal evolution of solid bitumen in the Cambrian reservoirs is higher than that of the Ordovician bitumen. Hence, it is critical to clarify the geochemical characteristics of the solid bitumen in the Cambrian reservoirs at a high and over-mature stage to correctly understand the Cambrian system in the Ordos Basin. This paper discusses the origin and source of the solid bitumen in the Cambrian reservoirs of the Ordos Basin based on a comprehensive analysis of its geochemical characteristics and the origin of the Cambrian natural gas.

The Ordos Basin is located in the middle and west of China, which is one of the largest petroliferous sedimentary basins in China, with an area of about 250,000 km^2 (Cai et al., 2005). During the middle and upper Proterozoic, the Ordos Basin is characterized by rifted marine deposits (Bai et al., 2014). Until the early Paleozoic, the basin was passive continental margins. The Cambrian and Ordovician are mainly carbonate platform deposits. From the end of the Ordovician to the middle of the Carboniferous, the entire basin was uplifted and denuded due to the influence of the Caledonian Movement. Transgression occurred in the middle Carboniferous, so the Carboniferous–Permian was deposited a set of alternating marine and terrestrial coal-bearing strata. The Triassic-Lower Cretaceous is characterized by lacustrine and fluvial deposits. After that, no deposition has occurred (Cai et al., 2005).

Samples and methods of analysis

The solid bitumen samples in the Cambrian reservoirs of the Ordos Basin were collected from drillings and outcrops, and the sampling location is shown in Figure 1A. The gaseous samples were mainly collected from the Qingcheng area in the Southern Basin (Figure 1A). The solid bitumen samples were subjected to environmental scanning electron microscopy (ESEM), energy-dispersive spectroscopy, laser Raman spectroscopy, and trace element analysis. In the field-emission environmental SEM analysis, the solid bitumen samples were first cut into 1 m^3 cubes or irregular blocks. A relatively flat and fresh section was selected and observed under the FEI ESEM Quanta™ 450 FEG equipped with an energy spectrometer. The energy-dispersive spectroscopic analysis of the area of interest was carried out using the Genesis Apollo XL instrument after correcting the energy spectrometer using cadmium or gold.

A LabRAM HR Evolution Confocal Raman microspectroscope was used for Raman spectroscopic measurement of samples. Main experimental conditions for Raman analysis include the following: a solid laser device of 532 nm/50 mW, confocal pinhole of 400 μm, raster slit of 100 μm, exposure time of 3–8 s, scanning wave number range 100–4000 cm^{-1}, and a silicon wafer to be used for the wave number calibration of the Raman spectroscope. The samples were ground into powder less than 200 mesh for trace element (element composition) analysis based on the ZSX Primus II X-ray fluorescence spectrometer.

Results

Geochemical characteristics of the solid bitumen in the reservoirs

Microscopic observations showed that the solid bitumen in the Cambrian reservoirs featured micro-fractures and intergranular pores in the form of strips (Figure 2A) and rings (Figures 2B,C). Moreover, most of the samples had clear boundaries, which indicates that the bitumen may mainly consist of pyrobitumen subjected to heat-induced alteration. Compared with bitumen from different origins, pyrobitumen usually has a clearer and straighter boundary (Stasiuk, 1997; Hwang et al., 1998; Hao F et al., 2011). Most of the samples were sheet-like, and some were observed on the surfaces of mineral particles in the SEM images (Figures 2D–F), which may demonstrate the process of cracking and coking of crude oil. This is because during the high-temperature cracking and coking of crude oil, small mesophase spheroids with optic anisotropy first emerge from the isotropic mother liquor; then, with progressive evolution, the mesophase spheroids continuously grow and deform and eventually change into sheet-like heterogeneous textures (Liu et al., 2009; Hao Bin et al., 2016).

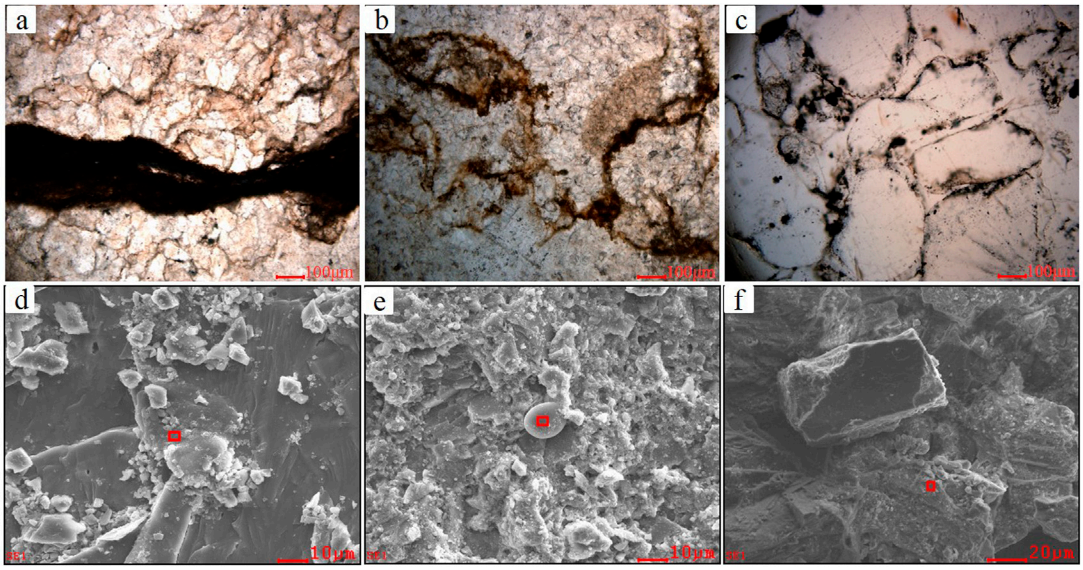

FIGURE 2
Examples of microscopic images showing the optical characteristics of solid bitumen in the Cambrian reservoirs of the Ordos Basin. **(A)** Bitumen in fractured dolomite, ϵ_2z, 4190.3 m, well L17; **(B)** bitumen in intergranular pores of limestone, ϵ_2z, ZY outcrop in Shanxi province; **(C)** bitumen in the intercrystalline pores of sandstone, ϵ_2x, 4348.0 m, well T59; **(D)** bitumen in dolomite, ϵ_2z, 4720.1m, well LT1; **(E)** bitumen in oolitic limestone, ϵ_2x, YJ outcrop in Shanxi province; **(F)** bitumen in dolomite, ϵ_1s, 3982.0 m, well L1.

The energy spectrum (Figure 3) showed that the solid bitumen in the Cambrian reservoirs of the Ordos Basin was mainly composed of C, O, Ca, and Mg atoms, where this can be attributed to the host minerals (chiefly dolomite and limestone) of the solid bitumen in the reservoirs. The mass fraction of Au atoms in the samples was high (up to 21.47% in some samples), yet it was artificially plated onto samples prior to the analysis, which means that this mass fraction was a background value. Moreover, the mass fraction of C atoms was 8.99%–45.68%, corresponding to even higher atomic fractions (15.19%–59.49%), and the mass fraction of O atoms was 19.67%–47.26%, with atomic fractions of 27.98%–59.70%. This indicates that the solid bitumen in the reservoirs had undergone carbonization and high thermal evolution.

The laser Raman spectrum presented two characteristic peaks of solid bitumen in the reservoirs (Olivier Beyssac et al., 2002, 2003; Lahfid et al., 2010), i.e., a "defect peak" (also known as the "D-peak", usually at 1320–1330 cm^{-1}) and a "graphite peak" (also known as the "G-peak," often within 1590–1650 cm^{-1}) (Kostova et al., 2012) (Figure 4). The existence of these characteristic peaks confirmed that the analyzed samples were in fact bitumen from the reservoirs. In general, with increasing maturity of bitumen, the peaks D and G of the samples become more pronounced and sharper, with peak G higher than peak D (Quirico et al., 2005).

The wavelengths of D and G peaks and the wavelength difference between D and G peaks in the laser Raman spectrum parameters are commonly used to quantitatively characterize the thermal evolution degree of solid bitumens (Wang et al., 2015). The laser Raman parameters of solid bitumen in the Cambrian reservoirs of the Ordos Basin are shown in Table 1. According to the wavelength difference between the D and G peak positions, the solid bitumen reflectance (*Rb*) in the Cambrian reservoirs can be calculated (Wang et al., 2015), which is between 1.98% and 3.39%. According to the conversion formulas in different publications (Jacob, 1989; Landis and Castano, 1995; Schoenherr et al., 2007), the bitumen reflectance (*Rb*) can be converted to the vitrinite equivalent reflectance (*Roeq*) (Table 1). However, both calibrations of the vitrinite equivalent reflectance (*Roeq*) differ from each other (Table 1). The *Rb* data on the solid bitumen in the Cambrian reservoirs of the Ordos Basin have their maximum at 3.39% (Table 1), and the regression equation of Jacob (1989) only includes the *Rb* value with a maximum of up to 2.7%, which caused the calibration small. However, the calibration of Landis and Castano (1995) is bigger than that of Jacob (1989), which may be either the high maturity of the selected samples or the fact that the formula itself has a certain scope of application. The calibration results of Schoenherr (2007) are used in this paper. Because the regression equation of Schoenherr (2007) combined the datasets of Jacob (1989) and Landis and Castano (1995), in which both samples with *Rb* values less than 2.7% and samples with *Rb* values greater than 4.0% are considered. The vitrinite equivalent reflectance (*Roeq*) of solid bitumen in the Cambrian

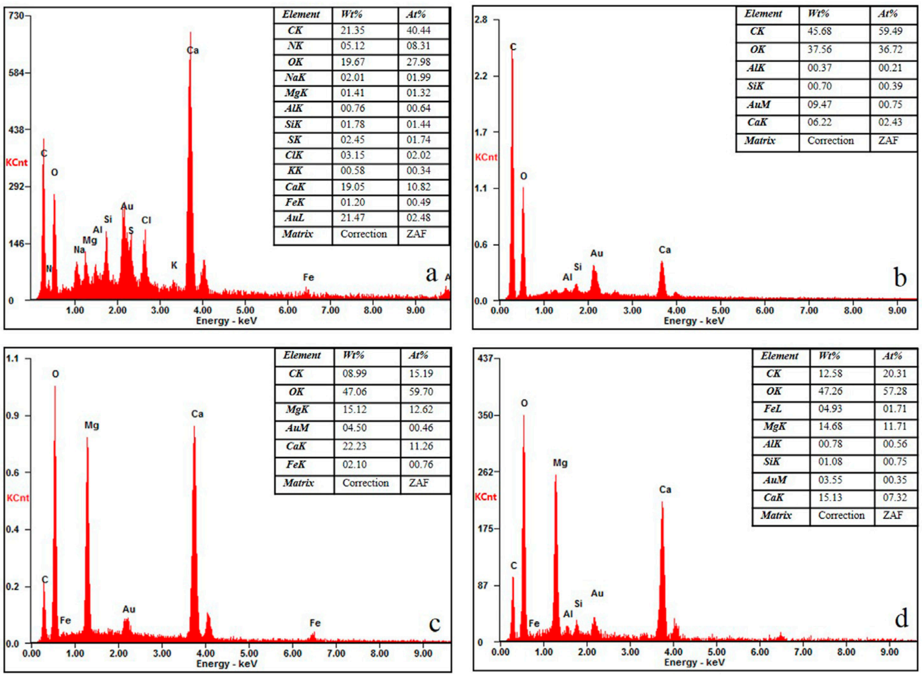

FIGURE 3
Characteristics of the energy spectrum of the solid bitumen in the Cambrian reservoirs. **(A)** ϵ_2z, 4720.1 m, well LT1. The energy spectrum shows that it mainly included C, O, Ca, and N. A higher content of C atoms occurred primarily due to the interference of the background (e.g., solid reservoir bitumen); **(B)** ϵ_2x, YJ outcrop in Shanxi province, with the energy spectrum indicating the bitumen; **(C)** ϵ_2z, ZY outcrop in Shanxi province. The energy spectrum shows that it included O, Mg, Ca, and C. Higher contents of Mg and Ca atoms indicate that the bitumen had occurred in dolomite; **(D)** ϵ_1s, 3982.0 m, well L1, with the energy spectrum indicating bitumen.

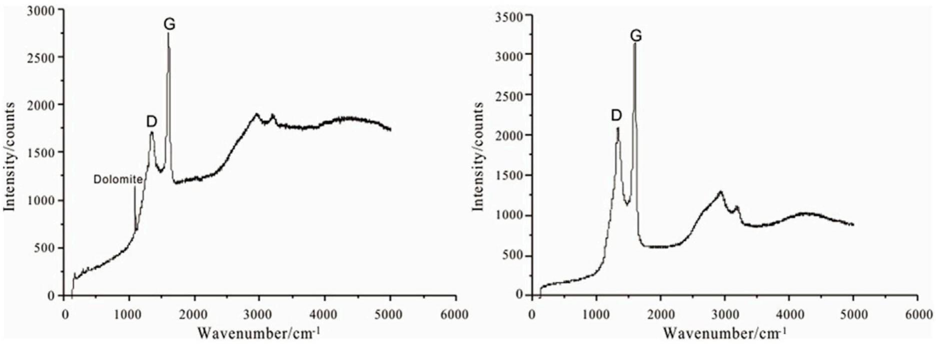

FIGURE 4
Laser Raman spectrogram of solid bitumen in the Cambrian reservoirs. Left–bitumen in dolomite, ϵ_2z, 4190.3 m, well L17; Right–bitumen in dolomite with intercrystalline pores, ϵ_2z, 3920.2 m, well XT1.

reservoirs of the Ordos Basin is calculated to be 2.12%–3.46% (Schoenherr et al., 2007), indicating that the solid bitumens in the Cambrian reservoir of the Ordos Basin had undergone a high thermal evolution process and is at the over-mature stage.

As shown in Figure 2, the solid bitumen in the Cambrian reservoirs of the Ordos Basin has a distinguishable optical microstructure. The solid bitumen in the reservoirs in Figures 2A–C is mainly filled with microfractures and intergranular pores. Also, the solid bitumen in the reservoirs in Figures 2D–F is mainly hosted on the surface of mineral particles. However, all the solid bitumen samples in the Cambrian reservoirs of the Ordos Basin do not display any fluorescence.

TABLE 1 Cambrian bitumen reflectance (Rb) data in the reservoirs of the Ordos Basin and their conversion into vitrinite equivalent reflectance (Roeq) values according to different authors.

Sample no./well name	Formation	D-band wavelength (cm^{-1})	G-band wavelength (cm^{-1})	Wavelength difference (G-D) (cm^{-1})	Wang et al. (2015) [a] bitumen reflectance (Rb%)	Jacob (1989) [b] (Roeq %)	Schoenherr et al. (2007) [c] (Roeq%)	Landis et al. (1995) [d] (Roeq %)
ZY-1	$Є_2z$	1338.0	1608.0	270.0	3.39	2.50	3.46	3.48
ZY-2	$Є_2z$	1350.0	1606.0	256.0	2.58	1.99	2.69	2.73
T59	$Є_2x$	1357.8	1603.5	245.7	1.98	1.62	2.12	2.18
L17	$Є_2z$	1360.9	1625.1	264.2	3.05	2.29	3.14	3.17
XT1	$Є_2z$	1330.3	1593.5	263.3	3.00	2.25	3.09	3.12

[a]Rb% = 0.058 wavelength difference (G-D) −12.27.
[b]Roeq% = 0.618 Rb%+0.40.
[c]Roeq% = (Rb%+0.2443)/1.0495.
[d]Roeq% = (Rb%+0.4)/1.09.

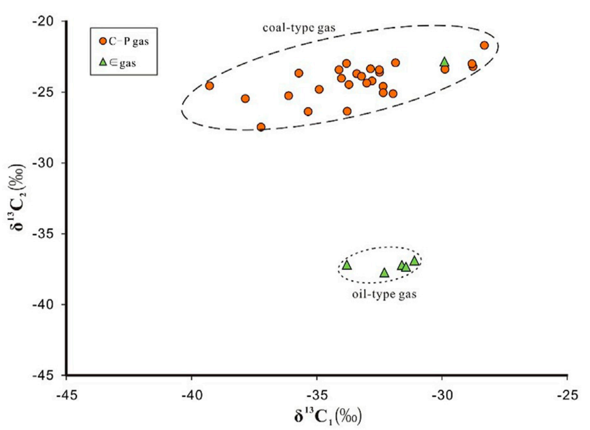

FIGURE 5
Scatter plots of $\delta^{13}C_1$ and $\delta^{13}C_2$ in the Cambrian natural gas of the Ordos Basin.

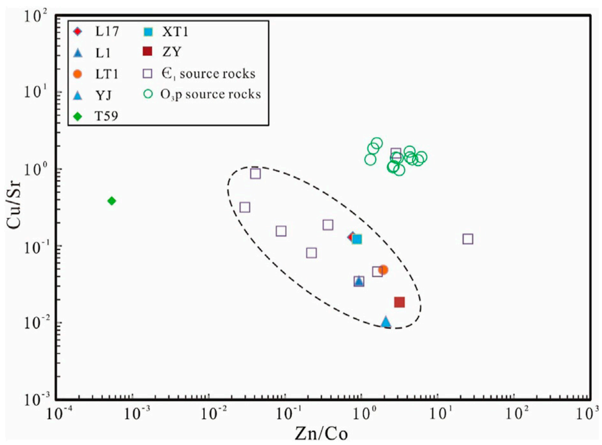

FIGURE 6
Trace element ratio scatterplot shows solid bitumen in the Cambrian reservoirs and potential source rocks of the Ordos Basin.

Correlation between solid bitumen in the reservoirs and potential source rocks

The mature bitumen had undergone a series of changes in terms of the commonly used organic geochemical indicator for bitumen–source rock correlation. This made it difficult to apply such parameters as biomarkers and stable carbon isotope ratios to the correlation to achieve the desired performance (Liang and Chen, 2005; Peters et al., 2005). Given this, the correlation between solid bitumen in the reservoirs and the source rock was investigated mainly with respect to the origin of the Cambrian natural gas and trace elements less affected by thermal maturity.

As illustrated by the cross-plot between the carbon isotope ratios of methane ($\delta^{13}C_1$) and ethane ($\delta^{13}C_2$) (Figure 5), the coal-type gas from the Upper Paleozoic Carboniferous–Permian was different from the Cambrian natural gas. Although the ranges of distribution of $\delta^{13}C_1$ for these two natural gases partially coincided, their values of $\delta^{13}C_2$ were significantly different. Overall, the content of $\delta^{13}C_2$ of the Cambrian natural gas was relatively low, mostly between −36.90% and −37.74%, exhibiting the characteristics of the oil-type gas. Only one sample contained $\delta^{13}C_2$ within the range of a coal-type gas. By comprehensively considering the planar distribution of the Cambrian natural gas samples (Figure 1A) and the source rocks (Du et al., 2019), it is inferred

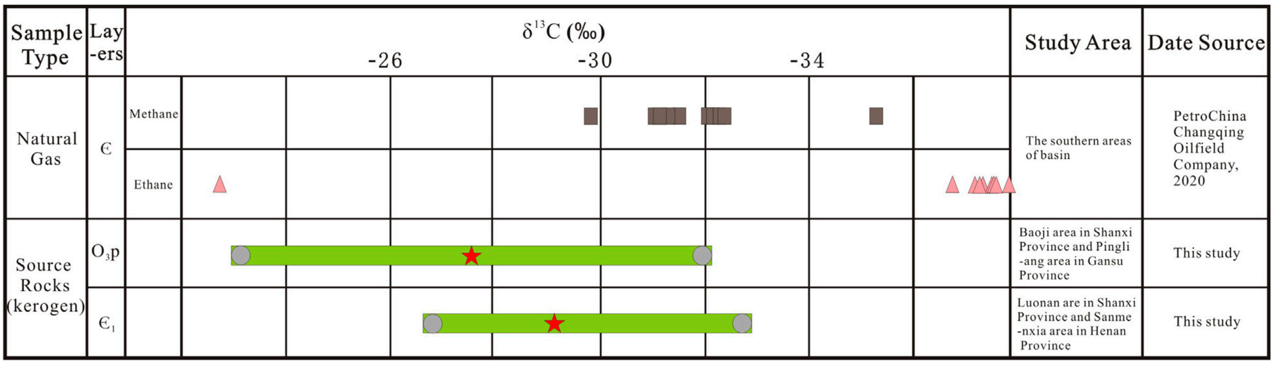

FIGURE 7
Carbon isotopic distribution of the Cambrian natural gas and the kerogen carbon isotopic distribution of the potential marine source rocks in the Ordos Basin.

that the natural gas had mainly come from the Cambrian or Ordovician marine source rocks in the Southern Basin.

The trace elements in the solid bitumen also contain genetic information on the parent rock, just like biomarkers and, more importantly, are not susceptible to hydrocarbon migration, reservoir destruction, oxidation, and biodegradation (Al-Shahristani et al., 1972; Lewan et al., 1982; Filby R H, 1994; Ramirez, 2013; Ping Gao et al., 2015; Chunhua Shi et al., 2015). Therefore, the trace elements were useful indicators to examine the correlation between bitumen in the over-mature Cambrian reservoir and the source rock of the Ordos Basin. The cross-plot of Cu/Sr versus Zn/Co for the trace elements clearly shows that most of the solid bitumen in the Cambrian reservoirs had similar characteristics of distribution to those of the Lower Cambrian source rock samples but are different from those of the Upper Ordovician Piangliang Formation source rocks (O_3p) (Figure 6). Interestingly, the trace element ratios (Cu/Sr and Zn/Co) of the solid bitumen samples in well T59 are not similar to those of the ϵ_1 and O_3p potential source rocks.

Discussion

It is worth noting that the low-yield gas flow drillings of the Cambrian are mainly distributed around the Qingyang paleo-uplift in the southwest of the Ordos Basin (Figure 1A). The ethane carbon isotope of the Cambrian in the Lower Paleozoic natural gas of the Ordos Basin is the lightest, which is lighter than the methane carbon isotope value of the Cambrian natural gas, and almost all of them are less than −36‰ (Huang et al., 2021). From the carbon isotope distribution of the Cambrian natural gas and the kerogen carbon isotopic distribution of the potential marine source rocks in the Lower Cambrian and the Upper Ordovician Pingliang Formation developed in the Western and Southern basins (Figure 7), it can be inferred that the carbon isotopic distributions of the Cambrian natural gas are similar to the kerogen carbon isotopic distributions of the Lower Cambrian source rocks. Generally, the ethane carbon isotope values of natural gas are mainly related to the type of organic matters. The ethane carbon isotope values of the oil-type natural gas are lighter, which are derived from sapropel-type organic matters. Also, the coal-type gas derived from humic-type organic matters has much heavier the ethane carbon isotope values (Huang et al., 1996).

The discovery and source of solid bitumen in the Cambrian reservoirs of the Ordos Basin provides direct evidence of hydrocarbon generation and accumulation in the Lower Cambrian source rocks. The solid bitumen in the Cambrian reservoirs is mainly located in the Western and Southern basins, and less in other places, which may be attributed to the planar distribution of the paleogeography (Dai. 2005; Bai et al., 2014; Du et al., 2019) and strata thickness (Figure 1A) of the Cambrian in the Ordos Basin. The solid bitumen in the Cambrian reservoirs was formed by over-mature pyrobitumen, which indicates that it might have been the product of cracking of crude oil. Moreover, an examination of the correlation between the solid bitumen in the reservoirs and the potential source rocks revealed similar distributions of trace elements (Cu/Sr and Zn/Co) between the solid bitumen in the Cambrian reservoirs and the Lower Cambrian source rocks in the Southern Basin, where well T59 in the basin was an exception. This shows that the solid bitumen in the Southern Basin might have primarily originated from the Lower Cambrian source rocks, while that in well T59 can be attributed to another source rock.

The structural-sedimentary evolution characteristics in the Southern Basin further show that the Caledonian Movement and Huaiyuan Movement caused the Cambrian–Ordovician strata to suffer overall uplift and denudation in the Ordos Basin, but the original structural framework was not changed (Huang et al., 2021). Also, the Cambrian–Ordovician strata of the Qingyang paleo-uplift in the southwest of the basin still keep dipping southward. Therefore, before the Late Triassic, the Qingyang paleo-uplift in the southwest of the Ordos Basin and the surrounding areas were the oil and gas migration areas, which were favorable for oil and gas migration and accumulation in the highly abundant marine source rocks from the Lower Cambrian.

Conclusion

The presence of solid bitumen in the Cambrian reservoirs of the Ordos Basin may represent a self-generating, self-storing pattern of hydrocarbon accumulation and provides direct evidence of hydrocarbon generation and expulsion in the Lower Cambrian source rocks of the Southern Basin. The highly abundant marine source rocks of the Lower Cambrian were revealed by outcrop profiles only in the Southern Basin, which is consistent with the solid bitumen being primarily distributed in the Southern Basin. This suggests that the Lower Cambrian source rocks might have contributed to the source of solid bitumen in the Cambrian reservoirs. The analysis of the microscopic characteristics, energy spectrum, and Raman spectrum of the samples showed that the solid bitumen in the Cambrian reservoirs was formed by over-mature pyrobitumen, where this is consistent with the degree of thermal evolution of the source rocks from the Lower Cambrian. Moreover, the oil-type origin of the Cambrian natural gas in the Southern Basin and the similar characteristics of distribution of certain trace elements shared between the solid bitumen in the Cambrian reservoirs and the source rocks further support the claim that the solid bitumen in the Southern Basin mainly originated from the highly abundant source rocks from the Lower Cambrian. This study on the correlation between the solid bitumen in the Cambrian reservoirs and the Lower Cambrian source rocks provides a new frontier for exploring natural gas in the Cambrian and the deep strata of the Ordos Basin.

Data availability statement

The original contributions presented in the study are included in the article/supplementary material; further inquiries can be directed to the corresponding author.

Author contributions

JH: writing—idea, original draft, formal analysis, and methodology. XL: review and editing, and supervision. KT: resources and project administration. NX and YZ: experiments and analysis.

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Conflict of interest

Author JH was employed by the China National Petroleum Corporation.

The remaining authors declare that the research was conducted in the absence of any commercial or financial relationships that could be construed as a potential conflict of interest.

Publisher's note

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