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Organic Geochemistry and Hydrocarbon Generation Characteristics of Shale of the Fourth Member of Yingcheng Formation in the South Shuangcheng Fault Depression, Songliao Basin

Lidong Shi [1], Lidong Sun [1], Liang Yang [1], Jinshuang Xu [1], Changpeng Du [1] and Fangwen Chen [2,*]

[1] Exploration & Development Research Institute, PetroChina Daqing Oilfield Co., Ltd., Daqing 163712, China
[2] School of Geosciences, China University of Petroleum (East China), Qingdao 266580, China
* Correspondence: chenfw@upc.edu.cn; Tel.: +86-532-86983191

Abstract: In order to evaluate the potential for oil and gas resources in the deep fault depression of the Northern Songliao Basin, shale from the fourth member of the Yingcheng Formation (K_1yc^4) in the South Shuangcheng Fault Depression was selected as an example. The organic geochemical characteristics such as abundance, type and maturity from experiments on low-maturity source rock samples, the hydrocarbon generation conversion rate, hydrocarbon generation amount and hydrocarbon generation period of the shale from K_1yc^4 were evaluated via the chemical kinetics method. The hydrocarbon generation threshold of shale from K_1yc^4 in the South Shuangcheng Fault Depression was analyzed by examining the organic matter (OM) in shale core samples from K_1yc^4. Based on the thermal simulations to an approximate buried depth of 750 m, the maximum oil-generation stage corresponds to an approximate buried depth of 1380 m. The amounts of generated oil and gas from the shale in K_1yc^4 are approximately 2.417×10^8 t and 0.546×10^{11} m^3, respectively. The shale in K_1yc^4 generated crude oil mainly during the sedimentary period of the Qingshankou Formation, Yaojia Formation and Nenjiang Formation, and mainly generated natural gas during the sedimentary period of the Nenjiang Formation. In the South Shuangcheng Fault Depression, the high parts of the local structure are the favorable areas for oil and gas exploration of K_1yc^4 in the sag zone, which could be used for the combined production of shale oil, tight sandstone oil and conventional oil.

Keywords: northern Songliao Basin; South Shuangcheng Fault Depression; Yingcheng Formation; deep oil and gas; hydrocarbon generation characteristics

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1. Introduction

In the past, shallow oil and gas resources with low difficulty and low development cost have been the focus of exploration and development [1–3]. With the middle and shallow oil and gas resources entering the middle and late stages of development, geologists are gradually exploring and developing oil and gas resources in deep and ultra-deep layers [4,5]. The exploration and development of deep and ultra-deep petroleum have gradually become a research hotspot in the fields of oil and gas [6–8]. There are deep and ultra-deep oil and gas reservoirs such as volcanic gas reservoirs, tight gas reservoirs and deep oil reservoirs in the northern Songliao Basin [9–11]. Among them, the most representative deep oil reservoirs are the fourth member of the Yingcheng Formation (K_1yc^4) and the third member of the Denglouku Formation (K_1d^3) in the South Shuangcheng Fault Depression (Figure 1) [12,13].

Shuangcheng Fault Depression is located in the eastern margin of the Southeastern Uplift Area of the Songliao Basin, in the southeast of Xujiaweizi Fault Depression, adjacent to Yingshan Fault Depression (Figure 1). The sedimentary strata in the fault depression are distributed in an area of about 5500 km^2. Shuangcheng Fault Depression is composed of Lower Cretaceous strata, including the Huoshiling Formation (K_1h), the Yingcheng

Formation (K_1yc), the Denglouku Formation (K_1d) and the Quantou Formation (K_1q); Upper Cretaceous strata, containing the Qingshankou Formation (K_2qn), the Yaojia Formation (K_2y) and the Nenjiang Formation (K_2n); Tertiary, including the Taikang Formation (N_2t); and the Quaternary System [14,15]. The sedimentary strata in this area are missing the Shahezi Formation (K_1sh) of the Lower Cretaceous, the Mingshui Formation (K_2m) and Sifangtai Formation (K_2s) of the Upper Cretaceous, and the Da'an Formation and Yi'an Formation of the Tertiary [16]. Exploration in the Shuangcheng Fault Depression has gone through the exploration stages of a shallow secondary gas reservoir, a volcanic gas reservoir and the Yingcheng–Denglouku reservoir. A set of high-quality source rocks has developed in K_1yc^4, which is a deep sedimentary stratum in the South Shuangcheng Fault Depression (Figure 1). The source rocks are buried relatively shallow, and their thermal evolution reached the mature stage of oil generation. The S66 well was drilled in the Denglouku Formation, and the oil-bearing layer was encountered with a daily crude oil test of 10.02 tons in 2016. The S68 well was drilled, and 100 tons of oil per day was obtained from self-injection of the Denglouku Formation in 2019, which resulted in high-yield industrial oil flow. The success of oil exploration in deep sedimentary strata in the South Shuangcheng Fault Depression has an important guiding role for oil and gas exploration in small faulted depressions in the Songliao Basin.

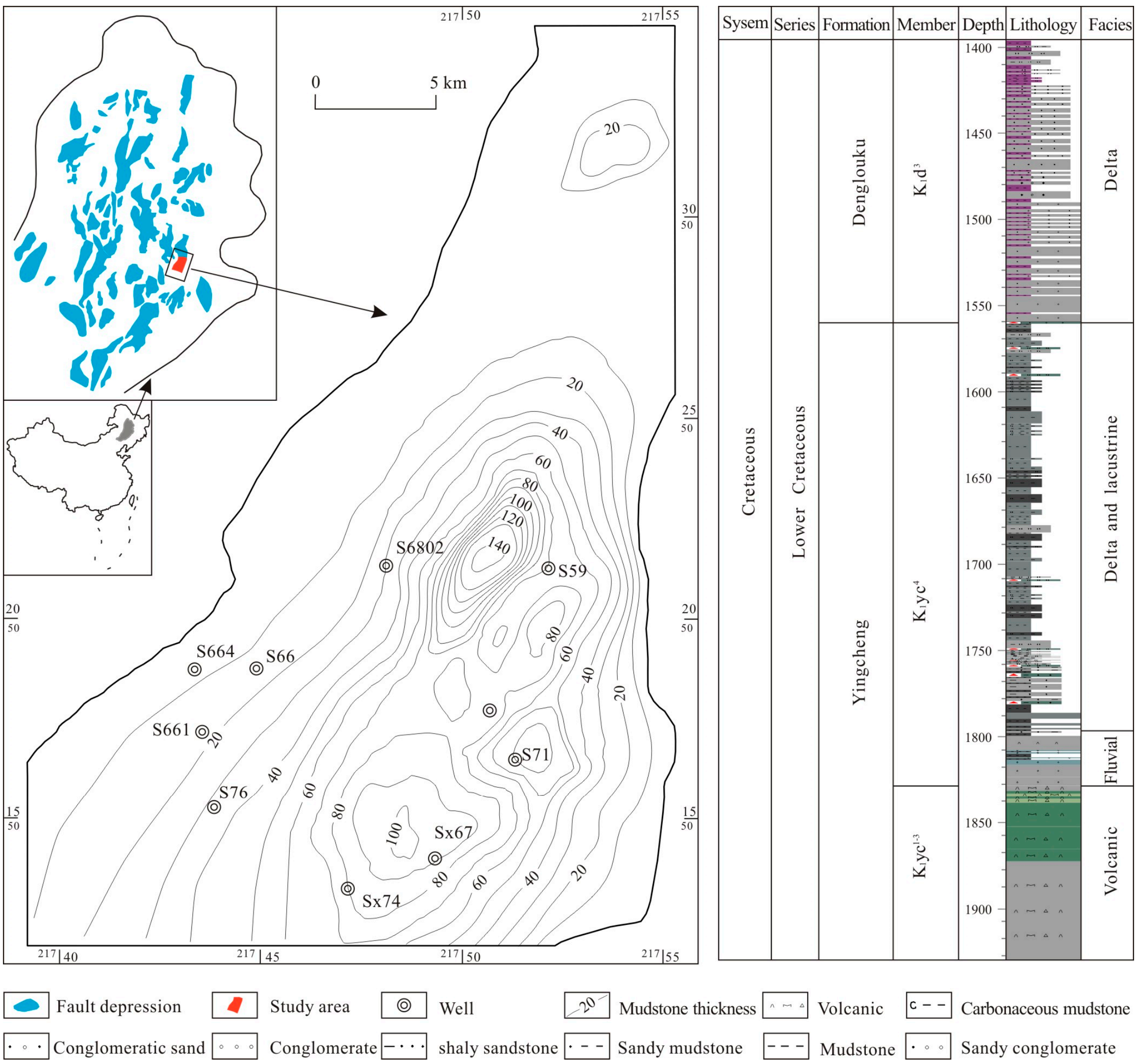

Figure 1. The mudstone thickness and lithological characteristics of K_1yc^4 in the South Shuangcheng Fault Depression.

However, evaluations of the source rock characteristics, such as the abundance of organic matter (OM), type and maturity, and hydrocarbon generation potential of the K_1yc^4 of deep sedimentary strata in the South Shuangcheng Fault Depression have been relatively weak. This seriously restricts the exploration of deep sedimentary reservoirs in the South Shuangcheng Fault Depression. In this study, the source rock characteristics and hydrocarbon generation potential of K_1yc^4 in the South Shuangcheng Fault Depression were evaluated based on the parameters of the source rocks. The amounts of oil and gas generated from the source rocks of K_1yc^4 were calculated using the method of chemical kinetics.

2. Samples and Experiments

2.1. Samples

In total, 218 core samples of the fourth member of the Yingcheng Formation were selected from 11 exploratory wells in the South Shuangcheng Fault Depression of Songliao Basin. All of them were organic-rich shale samples and were pretreated for experiments evaluating the total organic carbon (TOC), pyrolysis and chloroform bitumen "A", and vitrinite reflectance.

2.2. TOC, Pyrolysis and Chloroform Bitumen "A"

Prior to measurement, 250 g of each core sample was crushed into powder of 80–100 mesh size (sieve pore diameter 180–150 um). Each powdered sample was divided into three parts for the TOC, pyrolysis and chloroform bitumen "*A*" experiments, which were performed at the Research Institute of Exploration & Development, PetroChina Daqing Oilfield Company.

The TOC values of these core samples were determined using a Leco CS-230 carbon analyzer. Before measuring, the samples were treated with hydrochloric acid to remove carbonate [17,18]. The pyrolysis results of the samples, including the amounts of volatile gas (S_0), liquid hydrocarbons (S_1), pyrolysis hydrocarbons (S_2) and oxygen dioxide (S_3) and the temperature of maximum S_2 (T_{max}) [18], were measured using a Rock-Eval 6 instrument. The powdered samples were extracted with organic solvent ($CHCl_3$) [19]. The fraction which is recovered after solvent evaporation is called chloroform bitumen. The chloroform bitumen contents of the samples were measured using an AC210S electronic balance.

2.3. Kerogen Maceral and Vitrinite Reflectance

Kerogen maceral analyses were carried out using a transmitted light microscope. Thirty-five representative samples with different TOC contents and burial depths were selected and prepared for maceral identification. Kerogen macerals were divided into four groups: sapropelinite, liptinite, vitrinite and inertinite. The vitrinite reflectance (R_o) values of the thirty-five samples were measured on polished blocks using an MPM80 microscope under monochromatic light according to the detailed procedures recommended by the ICCP [20].

2.4. Thermal Simulation Experiments

Thermal simulation experiments on the oil and gas generated from kerogen were performed by means of pyrolysis, gas chromatography and mass spectrometry (PY-GC-MS) with an open pressure system. The temperature of the thermal simulation experiments was increased from 200 °C to 600 °C with three different heating rates (10 °C/h, 20 °C/h and 40 °C/h) using a Rock-Eval-II pyrolysis instrument. The thermal simulation experiments of gas generated from oil cracking were conducted using a gold tube simulation system under a closed pressure system. Twenty-six gold tubes containing crude oil were divided into two groups, which were heated from 350 °C to 680 °C with different heating rates (2 °C/h and 20 °C/h). When the temperature reached the set value, the corresponding gold tube was taken out, and the type and content of the gas products were measured.

3. Results

3.1. Abundance of OM

Most of the TOC values of the source rocks from K_1yc^4 in the South Shuangcheng Fault Depression were larger than 1.0% (Figure 2). The TOC contents of type I, II_1, II_2 and III OM were in the ranges of 2.25%–3.39%, 0.78%–4.65%, 1.11%–5.58% and 1.12%–4.00%, with average values of 2.82%, 2.60%, 2.93% and 2.91%, respectively (Table 1). The potential hydrocarbon generation (S_1 + S_2) values of the source rocks from K_1yc^4 in the South Shuangcheng Fault Depression were mainly distributed above 2.0 mg/g (Figure 2). The potential hydrocarbon generation values of type I, II_1, II_2 and III OM were in the ranges of 5.91–6.68 mg/g, 0.41–12.78 mg/g, 1.17–14.32 mg/g and 1.08–10.17 mg/g, with average values of 6.30 mg/g, 6.07 mg/g, 6.77 mg/g and 6.37 mg/g, respectively (Table 1). The chloroform bitumen "A" of the source rock from K_1yc^4 in the South Shuangcheng Fault Depression was mainly distributed above 0.1 mg/g (Figure 2). The chloroform bitumen "A" values of type II_1, II_2 and III OM were in the ranges of 0.03–0.25 mg/g, 0.02–0.34 mg/g and 0.04–0.36 mg/g, with average values of 0.15 mg/g, 0.17 mg/g and 0.18 mg/g (Table 1). By comprehensively evaluating the TOC content, hydrocarbon generation potential and chloroform bitumen "A", it is concluded that the source rocks of K_1yc^4 in the South Shuangcheng Fault Depression are of good quality and could provide a good material basis for hydrocarbon generation, according to the industry standard regarding OM abundance evaluation of continental source rocks.

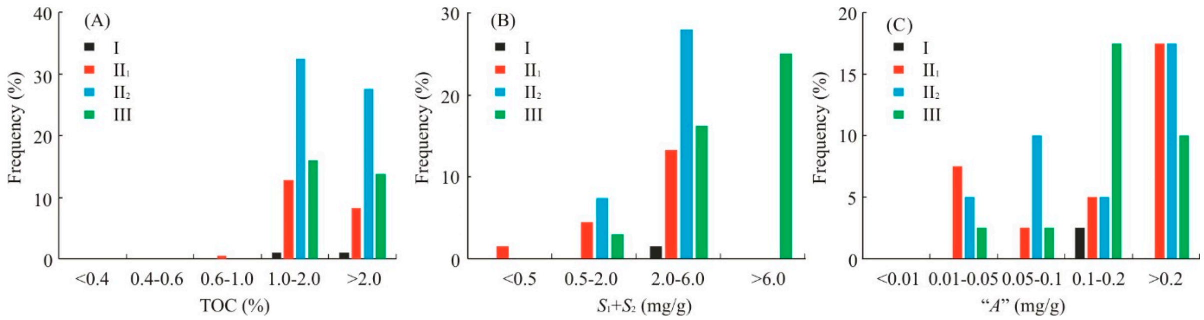

Figure 2. The characteristics of OM abundance in K_1yc^4 in the South Shuangcheng Fault Depression: (**A**) TOC content; (**B**) hydrocarbon generation potential S_1 + S_2; (**C**) chloroform bitumen "A".

Table 1. The organic geochemical characteristics of K_1yc^4 in the South Shuangcheng Fault Depression.

OM Type	TOC (%)		S_1 + S_2 (mg/g)		"A" (mg/g)		R_o (%)		Type Ratio (%)
	Range	Mean	Range	Mean	Range	Mean	Range	Mean	
I	2.25–3.39	2.82	5.91–6.68	6.30	/	0.10	/	0.89	2.27
II_1	0.78–4.65	2.82	0.41–12.78	6.07	0.03–0.25	0.15	0.84–0.93	0.88	27.27
II_2	1.11–5.58	2.93	1.17–14.32	6.77	0.02–0.34	0.17	0.76–1.08	0.93	45.45
III	1.12–4.00	2.91	1.08–10.17	6.37	0.04–0.36	0.18	0.87–1.05	0.94	25.00

3.2. Type and Maturity of OM

The OM types in K_1yc^4 in the South Shuangcheng Fault Depression are mainly type II_2 and type II_1, followed by type III and, rarely, type I. Their frequency percentages were 45.45%, 27.27%, 25.00% and 2.27%, respectively (Table 1 and Figure 3A). The intersection diagram of the hydrogen index and T_{max} analyzed using pyrolysis data showed that the OM types in K_1yc^4 in the South Shuangcheng Fault Depression are mainly II_1, II_2 and I (Figure 3B).

In terms of OM maturity, the vitrinite reflectance (*R*o) of source rocks in K_1yc^4 of the South Yingcheng Fault Depression was mainly in the range of 0.7–1.1%, and the average reflectance vitrinite values of type I, II_1, II_2 and III OM were 0.89%, 0.88%, 0.93% and 0.94%, respectively (Table 1 and Figure 3C). The thermal evolution stage of the source rocks of K_1yc^4 in the South Yingcheng Fault Depression is during the main oil-generation window.

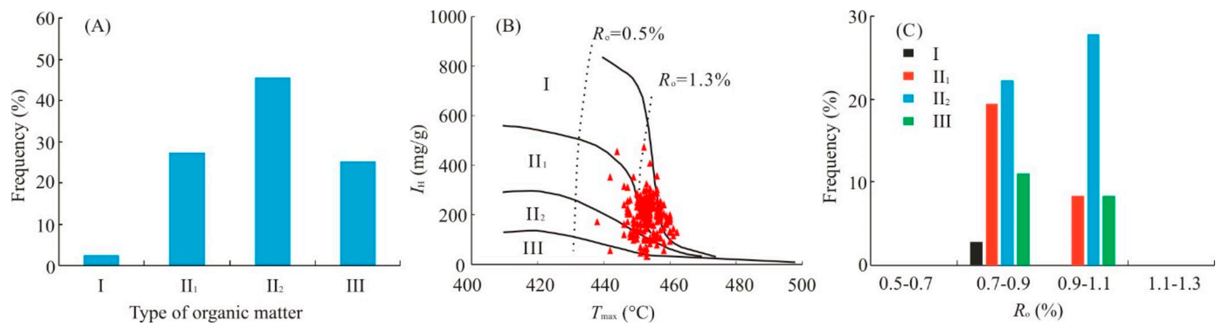

Figure 3. The type and maturity of OM in K_1yc^4 in the South Shuangcheng Fault Depression: (**A**) kerogen maceral analyses; (**B**) I_H–T_{max} intersection diagram; (**C**) vitrinite reflectance.

3.3. Thermal Simulation Experiments

Figure 4 shows the relationships between the conversion ratios of oil and gas generated from kerogen, gas generated from oil cracking, and temperature through the thermal simulation experiments and chemical kinetics calibration. The conversion ratios of oil generated from kerogen, gas generated from kerogen and gas generated from oil cracking lagged behind the increase in temperature, and all showed an "S"-type curve with three segments. This was divided into a slow increase stage (<380 °C), a fast increase stage (380–550 °C) and another slow increase stage (>550 °C). The trends of the conversion ratios of oil and gas generated from kerogen with temperature are similar. However, at high temperatures of 500–600 °C, the conversion ratios of oil generated from kerogen were basically stable, while the conversion ratios of gas generated from kerogen were still in a slow rising stage, and kerogen continued to generate hydrocarbon gases such as methane.

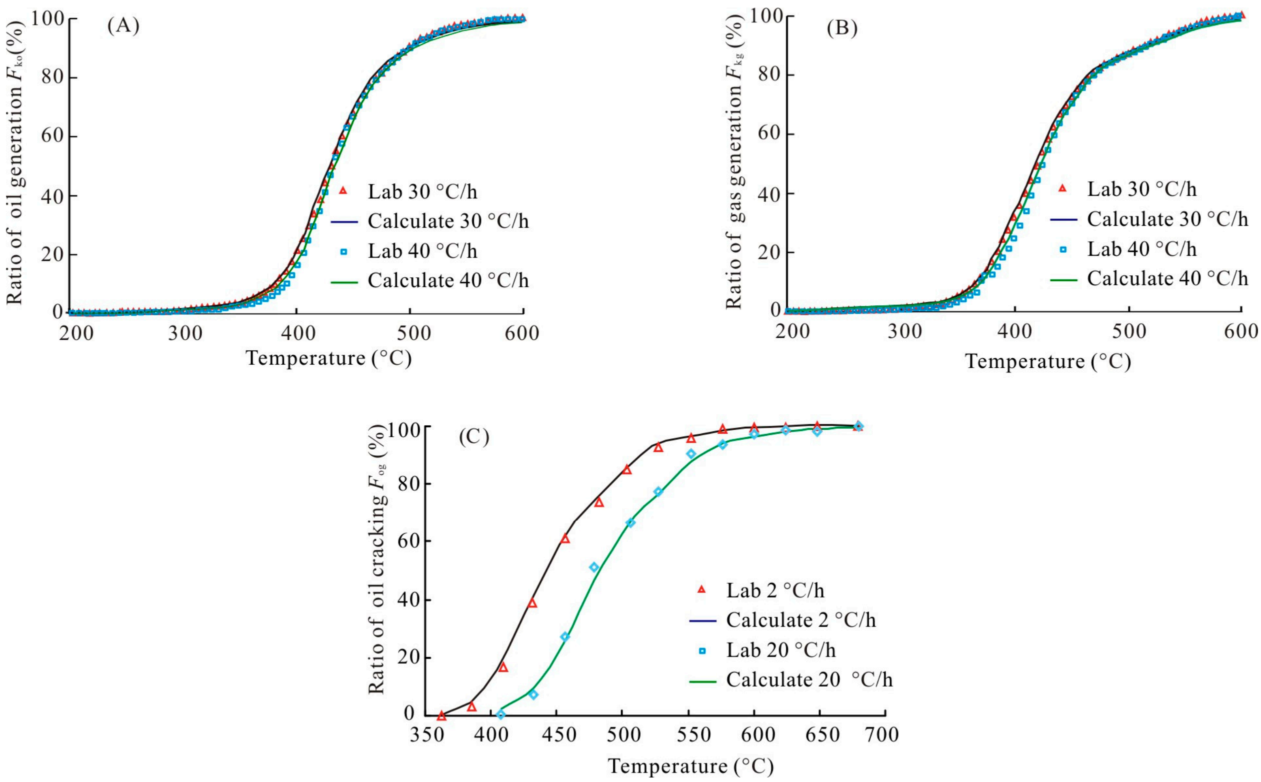

Figure 4. The conversion ratios of hydrocarbon generation from OM in thermal simulation experiments: (**A**) conversion ratio of oil generation from OM; (**B**) conversion ratio of gas generation from OM; (**C**) conversion ratio of gas generation from oil cracking.

3.4. Characteristics of Hydrocarbon Generation

3.4.1. Conversion Ratio Section of OM

Based on the thermal simulation experiments on the conversion ratios of hydrocarbon generation (including oil and gas generated from kerogen and gas generated from oil cracking) and the sedimentary and burial history and thermal history of the fourth member of K_1yc^4 (Figure 5), the relationship between the conversion ratio of hydrocarbon generation and present burial depth was calculated according to the methods of chemical kinetics for hydrocarbon generation [21–23].

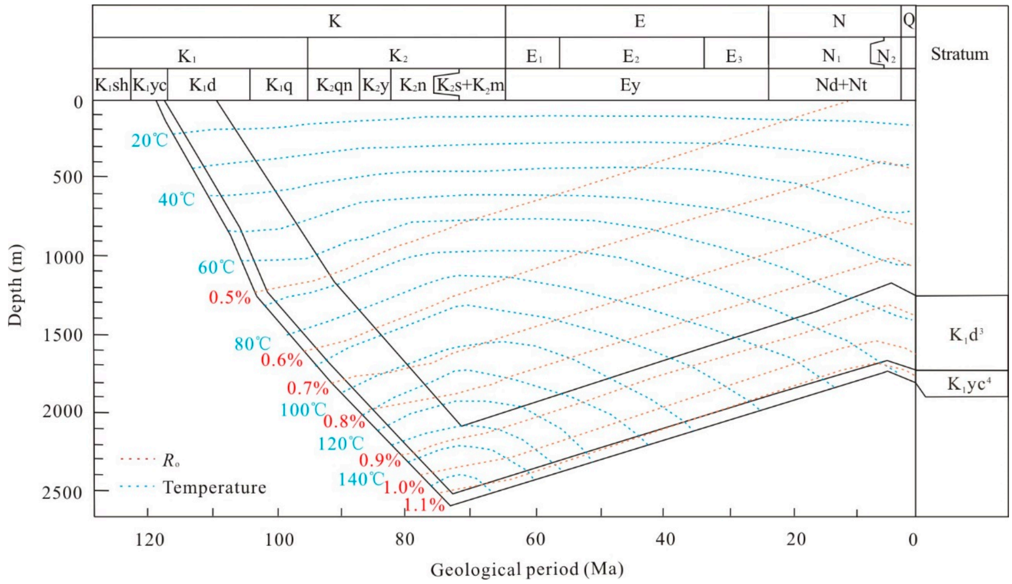

Figure 5. The sedimentary and burial history and thermal history of K_1yc^4 in the South Shuangcheng Fault Depression.

The hydrocarbon generation profile shows that the threshold of hydrocarbon generation from shale in K_1yc^4 in South Shuangcheng corresponds to a burial depth of approximately 750 m (Figure 6). The maximum oil-generation stage corresponds to a burial depth of approximately 1380 m, and the conversion ratio of oil generated from kerogen is 71.06%. The conversion ratio of gas generated from OM (including gas generated from kerogen and gas generated from oil cracking) exceeds the conversion ratio of oil generated from kerogen when the burial depth is larger than 1950 m.

3.4.2. Amount and Period of Hydrocarbon Generation

After obtaining the relationship between the conversion ratio of hydrocarbon generation from OM and the burial depth of K_1yc^4 in the South Shuangcheng Fault Depression, the original TOC and hydrogen index of the shale were recovered based on the burial depth, TOC content and hydrogen index according to the method of hydrocarbon generation chemical kinetics [23,24]. Figure 7 shows the residual and original TOC and hydrogen index of shale in K_1yc^4 for the S59 well. The recovery coefficients of the TOC and hydrogen index of the shale were in the ranges of 1.08–1.29 and 1.31–2.38, with average values of 1.19 and 1.20, respectively.

According to the evaluation method of hydrocarbon generation from shale [23], the amounts of oil and gas generated from shale in K_1yc^4 in the South Shuangcheng Fault Depression during the main geological period were determined (Figure 8). The amounts of oil and gas generated from shale in K_1yc^4 were 2.417×10^8 t and 0.546×10^{11} m^3, respectively. The amounts of oil generated during the sedimentary periods of the Quantou Formation, Qingshankou Formation, Yaojia Formation and Nenjiang Formation were 0.016×10^8 t, 0.934×10^8 t, 0.868×10^8 t and 0.598×10^8 t, respectively. The amounts of gas generated during the sedimentary periods of the Qingshankou Formation, Yaojia Formation and Nenjiang Formation were 0.006×10^{11} m^3, 0.043×10^{11} m^3 and 0.497×10^{11} m^3, respectively.

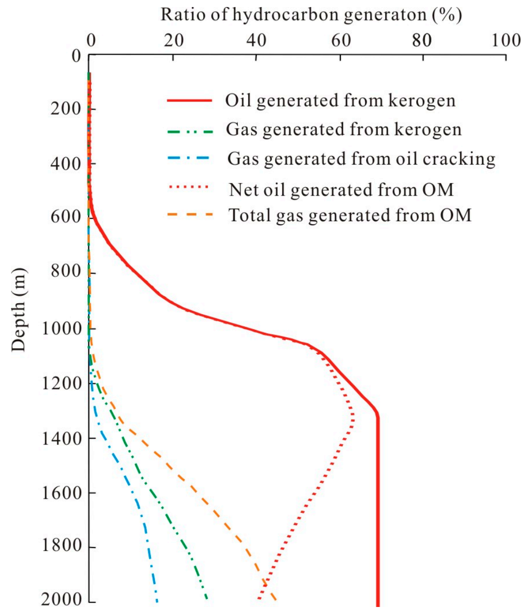

Figure 6. The hydrocarbon generation profile of K_1yc^4 in the South Shuangcheng Fault Depression.

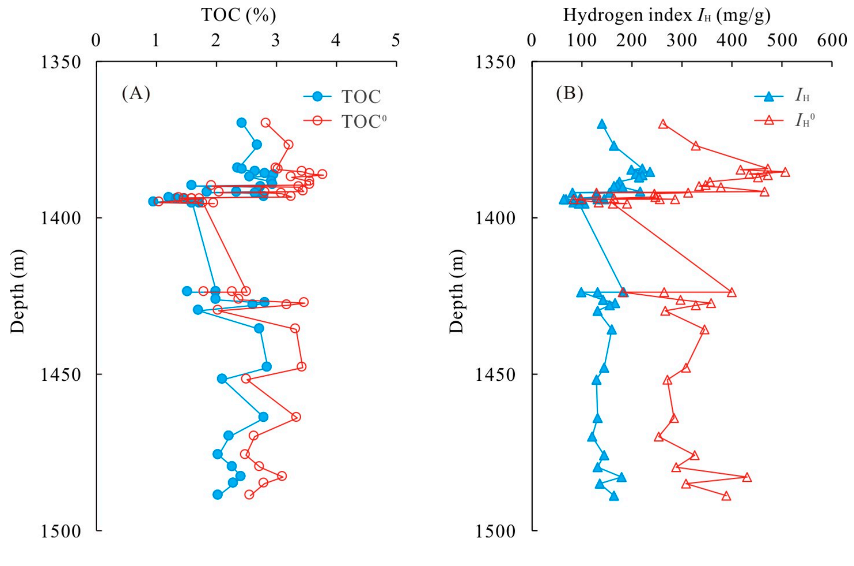

Figure 7. The original TOC and I_H of K_1yc^4 from well S59 in the South Shuangcheng Fault Depression: (**A**) TOC and original TOC; (**B**) I_H and original I_H.

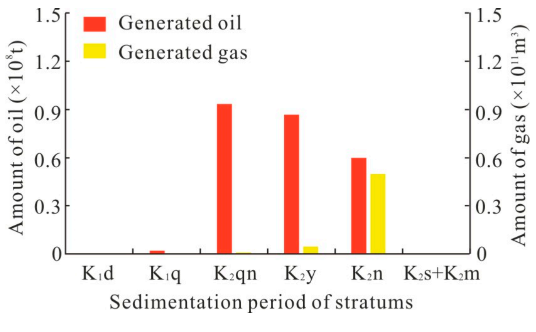

Figure 8. The amounts of hydrocarbon generation from K_1yc^4 in the South Shuangcheng Fault Depression.

4. Discussion

4.1. Comparison of Generated Oil and Gas Amounts

The amounts of oil and gas generated from source rocks are not only related to the abundance and type of OM but also affected by the degree of thermal evolution. Associated with the history (sedimentary, burial and thermal) of well S71, located in the sag zone, the vitrinite reflectance of the source rock in K_1yc^4 was up to 1.1%. This indicates that the source rocks in the center and vicinity of the sag zone have entered the highly mature stage of thermal evolution, and a large amount of crude oil was generated with a certain amount of wet gas. The amounts of oil and gas generated from shale in K_1yc^4 in the South Shuangcheng Fault Depression are estimated to be 2.417×10^8 t and 0.546×10^{11} m^3, respectively. The amount of generated oil for this set of source rocks is much greater than the amount of generated gas. In addition, after the sedimentary period of the Nenjiang Formation, the study area experienced multiple periods of intense tectonic activity, and the strata continued to be uplifted and denudated for a long period (Figure 5), which was relatively unfavorable to the enrichment and preservation of natural gas in the formations of K_1yc^4 and K_1d^3. Therefore, in the South Shuangcheng Fault Depression, exploration and development should be focused on crude oil in the K_1yc^4 and K_1d^3 formations.

4.2. Comparison of Oil and Gas Generation Periods

In addition to the type of OM, the oil and gas generation periods of source rocks are mainly controlled by the thermal evolution stage, which is closely related to the sedimentary, burial and thermal history. After the sedimentary period of K_1yc^4 in the South Shuangcheng Fault Depression, K_1yc^4 continued to descend until the late sedimentary period of the Nenjiang Formation, continued to uplift until the late Tertiary, and finally descended slowly to the present (Figure 5).

The vitrinite reflectance of the source rocks reached 0.5%, 0.7% and 0.9% in the sedimentary periods of the early Quantou Formation, the middle Qingshankou Formation and the early Nenjiang Formation, respectively. The thermal evolution of the source rocks was the low mature stage (0.5%–0.7%) during the sedimentary period from the early Quantou Formation to the middle Qingshankou Formation. It was in the early middle mature stage (0.7%–0.9%) during the sedimentary period from the middle Qingshankou Formation to the early Nenjiang Formation. It was in the later middle mature stage (0.9%–1.3%) after the sedimentary period of the early Nenjiang Formation. The thermal evolution of the source rocks stopped at the end of the sedimentary period of the Nenjiang Formation due to stratum uplift.

The source rocks of K_1yc^4 in the South Shuangcheng Fault Depression mainly generated crude oil during the sedimentary periods of the Qingshankou Formation, the Yaojia Formation and the Nenjiang Formation (Figure 8), and the proportions of generated oil were 38.66%, 33.66% and 24.73%, respectively. They mainly generated natural gas during the sedimentary period of the Nenjiang Formation, followed by the sedimentary period of the Yaojia Formation (Figure 8), and the proportions of generated natural gas were 91.09% and 7.85%, respectively. K_1yc^4 was uplifted in the sedimentary period of the late Nenjiang Formation, and the hydrocarbon generation and thermal evolution of source rocks stopped. The crude oil no longer cracked to generate natural gas, which is conducive to the enrichment of crude oil resources.

4.3. Favorable Areas for Oil Exploration

In the South Shuangcheng Fault Depression, K_1yc^4 contains organic-rich shale and sandstone, and its overlying stratum is K_1d^3 including sandstone and sandy conglomerate reservoirs. The reservoirs in K_1yc^4, including shale oil, tight oil and conventional oil reservoirs, have the properties of self-generation and self-accumulation. The reservoir in K_1d^3 mainly developed conventional oil accumulation of lower generation and an upper reservoir. In the South Shuangcheng Fault Depression, the high parts of the local structure are the favorable areas for oil and gas exploration of K_1yc^4 in the sag zone, which could be used for the combined production of shale oil, tight sandstone oil and conventional oil. After successful exploration, it was expanded around the area. The areas with weak fault activity intensity are favorable targets for conventional oil exploration of K_1yc^4 in the slope zone. The source rocks of K_1yc^4 generated a certain amount of natural gas in the sedimentary period of the Nenjiang Formation, but no natural gas reservoir has been found in K_1yc^4 or K_1d^3 in the current exploration. It is speculated that part of the generated natural gas exists in the form of oil-soluble gas, and the other part seeped and dissipated into the overlying strata. Oil-soluble gas is very beneficial to shale oil development, reducing the viscosity of shale oil, maintaining the fluid pressure of the shale oil reservoir, and keeping the reservoir energy at a slow decline in the process of mining. The high parts of the local structure in the sag zone and the trap with weak fault activity intensity in the slope zone are the favorable areas for conventional oil exploration of K_1d^3.

5. Conclusions

(1) In the South Shuangcheng Fault Depression, the OM in K_1yc^4 has higher abundance, mainly type II_2 and II_1, and higher maturity. The hydrocarbon generation threshold of shale in K_1yc^4 in the South Shuangcheng Fault Depression corresponds to an approximate buried depth of 750 m, and the maximum oil-generation stage corresponds to an approximate buried depth of 1380 m.

(2) Determined using the chemical kinetics method, the amounts of generated oil and gas from shale in K_1yc^4 were approximately 2.417×10^8 t and 0.546×10^{11} m^3, respectively. The shale in K_1yc^4 generated crude oil mainly during the sedimentary period of the Qingshankou Formation, Yaojia Formation and Nenjiang Formation, and the proportions of generated oil were 38.66%, 33.66% and 24.73%, respectively. It mainly generated natural gas during the sedimentary period of the Nenjiang Formation, followed by the sedimentary period of the Yaojia Formation, and the proportions of generated natural gas were 91.09% and 7.85%, respectively.

(3) In the South Shuangcheng Fault Depression, the high parts of the local structure are the favorable areas for oil and gas exploration of K_1yc^4 in the sag zone, which could be used for the combined production of shale oil, tight sandstone oil and conventional oil.

Author Contributions: Conceptualization, L.S. (Lidong Shi) and F.C.; methodology, L.S. (Lidong Shi); software, L.Y.; validation, L.S. (Lidong Shi), L.S. (Lidong Sun) and J.X.; formal analysis, C.D.; investigation, J.X.; resources, L.S. (Lidong Sun); data curation, F.C.; writing—original draft preparation, L.S. (Lidong Shi) and F.C.; writing—review and editing, F.C.; visualization, L.Y.; supervision, F.C.; project administration, L.S. (Lidong Sun); funding acquisition, L.S. (Lidong Sun) and F.C. All authors have read and agreed to the published version of the manuscript.

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