Characteristics of oil and gas production of oil shale pyrolysis by water vapor injection

Jing Zhao[a,b], Lei Wang[a,b]*, Dong Yang[a,b], Zhiqin Kang[a,b]

[a] Key Laboratory of In-situ Property Improving Mining of Ministry of Education, Taiyuan University of Technology, Taiyuan 030024, P.R. China
[b] The In-situ Steam Injection Branch of State Center for Research and Development of Oil Shale Exploitation, Taiyuan University of Technology, Taiyuan 030024, P.R. China

Abstract. *An appropriate pyrolysis temperature is required to achieve the best-quality oil and gas products via kerogen pyrolysis for the application of the in situ shale exploitation technology. In this study, the oil and gas products obtained at different pyrolysis temperatures via the oil shale pyrolysis process were analyzed using gas chromatography (GC). The results show that as the pyrolysis temperature increases, the content of hydrocarbon gases first increases and then decreases. Meanwhile, the H_2 content in nonhydrocarbon gases gradually increases and reaches 64.07% at 550 °C. In addition, when the pyrolysis temperature is > 400 °C, the content of light components in shale oil rapidly increases. Further, when the pyrolysis temperature exceeds 500 °C, the content of light components in shale oil exceeds 42%. Finally, the H_2 content obtained from oil shale pyrolysis by injecting water vapor is approximately eight times higher than that obtained from direct dry distillation. Additionally, the shale oil quality under water vapor action is better than that under direct dry distillation. The kerogen pyrolysis is performed in the H_2-rich environment and shale oil is prone to hydrogenation reaction.*

Keywords: *oil shale pyrolysis, water vapor, oil and gas products, H_2, light components.*

1. Introduction

Oil shale is a high-ash sedimentary rock containing abundant solid organic matter kerogen. Shale oil and gas can be obtained through dry distillation of oil shale. After the hydrocracking and refining of shale oil, gasoline, kerosene,

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* Corresponding author: e-mail *leiwang0327@163.com*

diesel, paraffin, stone tar and other chemical products can be obtained; thus, oil shale is an important strategic energy material [1, 2]. China has abundant oil shale resources, more than 1 trillion tons, equivalent to the shale oil reserves of 57 billion tons [3, 4], which can be considered as a huge supplement to oil. Therefore, efficient exploitation of oil shale is essential.

Most countries advocate the exploitation of oil shale using in situ retorting technology. Through appropriate control of the in situ pyrolysis process of oil shale, oil and gas products with better quality can be obtained. Lai et al. [5, 6] investigated the oil and gas production law of oil shale using an internal component-fixed bed. They observed that the temperature range of large-scale shale oil production is 350–460 °C. In the initial production stage of shale oil, the pyrolysis product is mainly light oil, whereas in the later stage, a large amount of heavy oil is produced. Wang et al. [7] investigated the effect of final pyrolysis temperature on the properties of shale oil obtained through the dry distillation of Huadian oil shale. The results show that the degree of conversion of naphthenic hydrocarbons to aromatic hydrocarbons is more obvious as the final pyrolysis temperature increases. The increase of the final pyrolysis temperature is conducive to the fracture of long-chain aliphatic hydrocarbons into short-chain aliphatic hydrocarbons. Na et al. [8] investigated the influential characteristics of temperature on the shale oil yield. The researchers observed that when the pyrolysis temperature is between 400 and 600 °C, the shale oil yield increases with increasing temperature. However, the shale oil yield decreases when the temperature exceeds 600 °C. Thus, a high temperature is not conducive to shale oil recovery; it is more appropriate to control the pyrolysis temperature between 500 and 550 °C. The oil and gas production law of oil shale under the condition of direct retorting was achieved from the above research, providing a certain theoretical basis for using oil shale in situ mining technology.

Tucker et al. [9] pyrolyzed oil shale in N_2, CO_2, supercritical CO_2 and water. The results show that the shale oil yield obtained by supercritical water pyrolysis is high. However, the preparation process of supercritical water is complex and the cost is high. Through experimental research, Razvigorova et al. [10] observed that the shale oil produced from Bulgarian oil shale pyrolysis by injecting steam is of good quality and the content of semicoke is low. Lewan and Birdwell [11] conducted an experiment to extract organic matter from oil shale with near-critical water. They noticed that when the temperature is 350 °C, the near-critical water has a high extraction rate of organic matter. The researchers also observed that the C15 alkanes in the extracted material are much higher than those obtained using anhydrous extraction. Sun et al. [12] performed oil shale pyrolysis using a high-pressure power frequency method. The investigators detected that the gas obtained from the oil shale pyrolysis is mainly small molecule hydrocarbon gas and the shale oil is mostly composed of medium C16–C24 molecules. It can be seen that when oil shale is pyrolyzed

with different types of heat-carrying fluid, the oil and gas components produced via organic matter pyrolysis are different.

In 2005, scientists of Taiyuan University of Technology [13] invented the convective heating oil shale technology (MTI Technology) and selected water vapor as the medium for convective heating of oil shale. Additionally, a series of experiments involving high-temperature water vapor pyrolysis of oil shale was conducted. It is found that the pyrolysis of oil shale has high efficiency and recovery rate along with improved quality. The high-temperature water vapor has a complex chemical reaction with kerogen, thus changing the release characteristics of oil and gas. In this study, the pyrolysis tests of oil shale under water vapor action are systematically conducted. The variation characteristics of gas components and oil products are investigated to provide a theoretical basis for the large-scale exploitation of oil shale by injecting water vapor.

2. Methods

Oil shale samples were collected from an open-pit mine in Barkol County, Xinjiang, China. The samples were sealed with asphalt to prevent oxidation. Then, these were transported back to the laboratory. Two samples were selected and sent to the testing center of Shanxi Institute of Coal Geology for conducting oil content tests and industrial analysis. The test results are presented in Table 1.

Table 1. Proximate and Fischer assay analyses of oil shale

Analysis	Proximate analysis, %				Fischer assay analysis, %			
Sample	Moisture	Ash	Volatile matter	Fixed carbon	Oil yield	Water yield	Residue	Gas + loss
1#	0.82	76.45	18.29	4.44	10.14	1.34	82.35	6.17
2#	0.65	79.31	16.64	3.40	9.03	1.13	85.61	4.23

The small water vapor system of pyrolysis oil shale comprises a small water vapor generator, a high-temperature resistant reactor, and oil and gas condensation equipment (Fig. 1). The water vapor generator constitutes a high-temperature resistant autoclave body and a secondary heating device (Fig. 1a). The gas production rate of the water vapor generator is 7 kg/h, and the flow rate of water vapor is 4.6×10^6 mL/min.
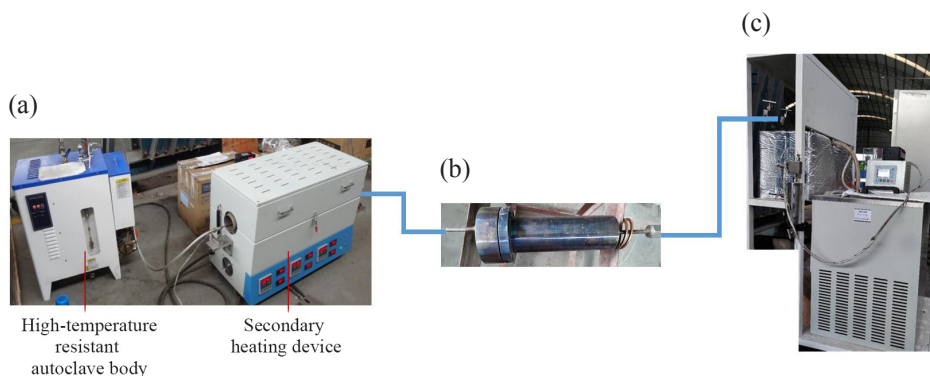
Fig. 1. The water vapor system of oil shale pyrolysis: (a) water vapor generator; (b) high-temperature resistant reactor; (c) oil and gas condensing equipment.

First, the high-temperature resistant reactor is filled with oil shale fragments (mass 4 kg). One end of the reactor is tightly fixed with the flange through high-strength bolts and connected with the small water vapor generator. The other end of the reactor is connected to the oil and gas condensation equipment. Then, the water vapor generator is started and the oil shale pyrolysis tests are conducted. The oil and gas condensing equipment is cooled and oil and gas at different pyrolysis temperatures are collected. It is observed that the oil and gas can only be collected when the pyrolysis temperature reaches 300 °C. Therefore, the oil and gas collection is conducted at six temperatures (300, 350, 400, 450, 500, 550 °C). The pyrolysis is performed for 0.5 h at each temperature to ensure the full pyrolysis of organic matter. Finally, the oil and gas products were analyzed using gas chromatography (GC) and the influence of pyrolysis temperature on the composition and quality of oil and gas products was investigated.

3. Results and discussion

3.1. Variation characteristics of gas components

The Agilent 7820a gas chromatograph (GC) is used for analyzing the pyrolysis gas obtained at different heat injection temperatures. The flame ionization detector (FID) and the thermal conductivity detector (TCD) are equipped with a GC. The former is used to detect hydrocarbon gases, whereas the latter is used to detect other component gases. The gas percentage is obtained by normalizing each gas peak in the spectrum obtained by the FID and TCD detectors. Figures 2a and 2b show the variation laws of hydrocarbon gas and other gas components with pyrolysis temperature, respectively.
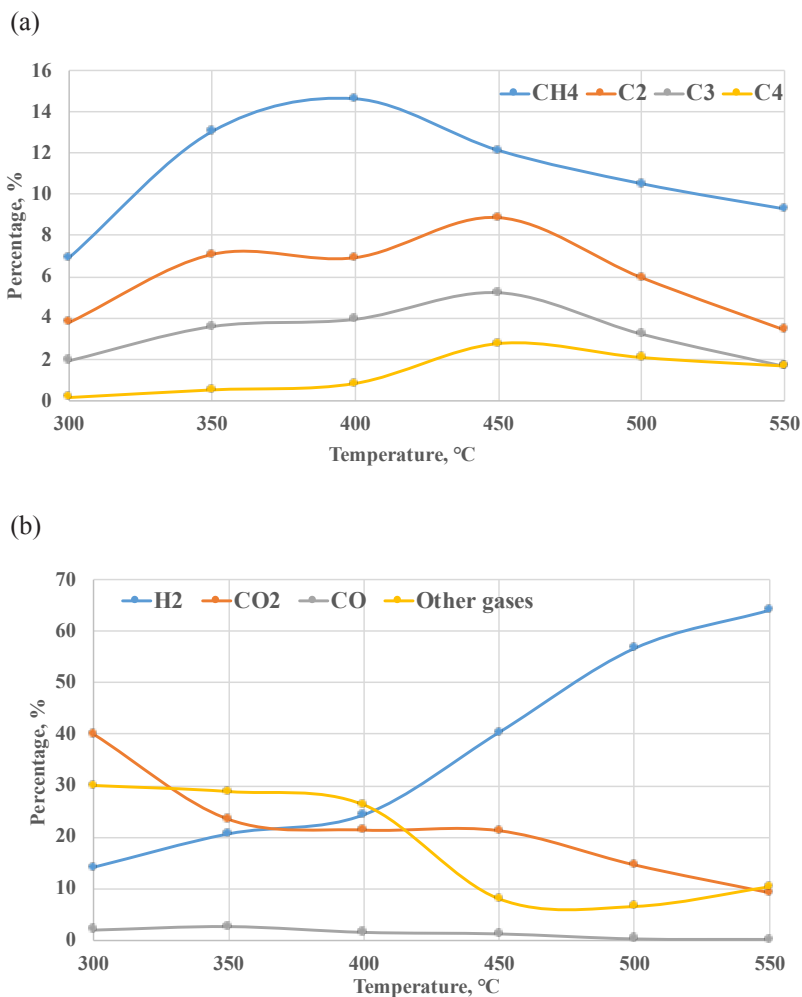
(a)

(b)

Fig. 2. Variation of gas composition with temperature: (a) hydrocarbon gas; (b) other gases.

As shown in Figure 2, many bridge bonds between the kerogen macromolecular structural units of oil shale are broken during the heating process, and the free radical concentration sharply increases. Additionally, the side chains of small molecular aliphatic and macromolecular aliphatic hydrocarbons are broken and cracked to produce numerous hydrocarbon gases. In the temperature range of 300–550 °C, the content of hydrocarbon gas in different carbon numbers first increases and then decreases. The content of CH_4 reaches a peak at 400 °C with a value of 14.63%. Meanwhile, when

the pyrolysis temperature is low, CH_4 is produced due to the breaking of the aliphatic bonds of the long-chain C–C structure. In contrast, when the pyrolysis temperature is high, more CH_4 is produced due to the breaking of the bonds between aromatic methyl and arylalkyl. C2–C4 gases peak at 450 °C, with maximum contents of 8.87%, 5.24% and 2.78%, respectively. The C–C bond of C4 gas easily breaks under the action of temperature to form CH_4.

In other gases, the CO content is always low. As the temperature increases, CO_2 content gradually decreases, whereas the H_2 content gradually increases. When the pyrolysis temperature is < 350 °C, the CO_2 content is higher than the H_2 content. The carboxyl, C–O–C and C=O groups on kerogen macromolecular functional groups have poor stability and break at low pyrolysis temperatures to form more CO_2. Meanwhile, H_2 mainly comes from the cyclization of aliphatic chain hydrocarbons and aromatization of cycloalkanes. Therefore, the H_2 content is low at low temperatures. Moreover, when the pyrolysis temperature is > 400 °C, the H_2 content is higher than the CO_2 content. Furthermore, when the temperature is 550 °C, the H_2 content is the highest at 64.07%. The reactor is placed in a hydrogen-rich environment. The activity of high-temperature water molecules is strong. When the concentration of hydrocarbon gas in the pyrolysis environment is large, the energy required for hydrocarbon gas activation is reduced under the action of numerous active free radicals and high-energy water molecules. The hydrogen-free radicals generated by activation are combined with the hydrogen-free radicals released by high-energy water molecules in high-temperature water vapor and released in the form of H_2. The high-temperature pyrolysis of oil shale forms a large amount of residual carbon, which reacts with water vapor at high temperature to release H_2. Moreover, the CO formed through the reaction between water vapor and residual carbon continues to undergo water vapor conversion reaction [14, 15].

3.2. Comparison of the pyrolysis gas components of oil shale under different heating methods

When oil shale is pyrolyzed using different heating methods, the content of various gases in the mixed gas formed by organic matter pyrolysis differs. The most successful oil shale ground retorts in the world include Chinese Fushun retort, Estonian Galoter furnace and Brazilian petrosex furnace [16, 17]. The methane content in the retort gas obtained by the pyrolysis of Xinjiang oil shale in the Fushun retort is low. In other gases, the content of carbon dioxide is higher, whereas the content of hydrogen is lower. Generally, the calorific value of dry distillation gas is lower. Table 2 presents the contents of the main components of pyrolysis gas of oil shale under different heating modes.

Table 2. Main component content of pyrolysis gas under different heating modes [18, 19]

Heating mode		Direct retorting	Convective heating
		Fushun furnace	High-temperature water vapor
Pyrolysis gas composition, %	H_2	7.9	64.07
	CO	6.5	0.21
	CO_2	18.3	9.24
	CH_4	13.5	9.29
	$\geq C2$	50.2	6.78
	Other gases	3.6	10.41

The direct retorting technology described in Table 2 is gas thermal retorting technology, which uses the gas formed by oil shale pyrolysis and solid residue gasification as the heat-carrying fluid to provide the required heat for oil shale retorting. The heating technology described in this study uses high-temperature water vapor as a medium to pyrolyze oil shale. The different heat carrier fluid selected inevitably leads to the difference in pyrolysis gas composition. The H_2 content in the gas components obtained from oil shale pyrolysis by injecting water vapor is approximately eight times higher than that obtained from direct retorting. H_2 energy is the cleanest secondary energy and the most ideal energy carrier in the postpetroleum era. Furthermore, the high H_2 content in pyrolysis gas provides a good foundation for H_2 energy storage, which is of great significance.

3.3. Variation characteristics of shale oil components

The composition of shale oil formed by the pyrolysis of oil shale was determined by the Agilent 7890b-5977b gas chromatograph-mass spectrometer (GC-MS). The chromatographic column of the instrument is hp-5 ms. Figure 3 shows the gas chromatograms of shale oil formed by oil shale pyrolysis under water vapor action. The constituent substances can be determined by analyzing the characteristic peaks in the gas chromatogram. The percentage content of each constituent substance can be obtained using the full area normalization method.

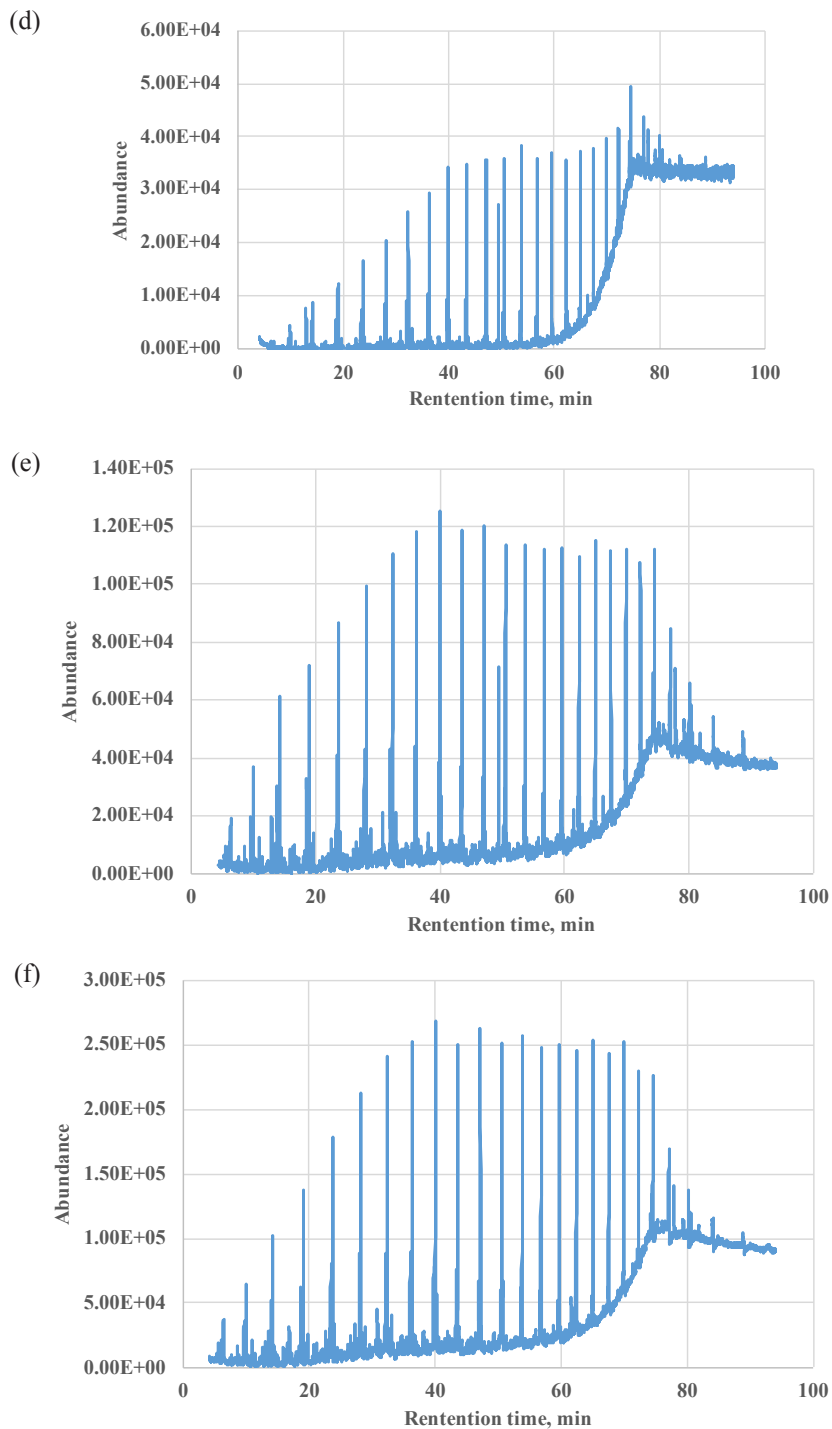(a)

(b)

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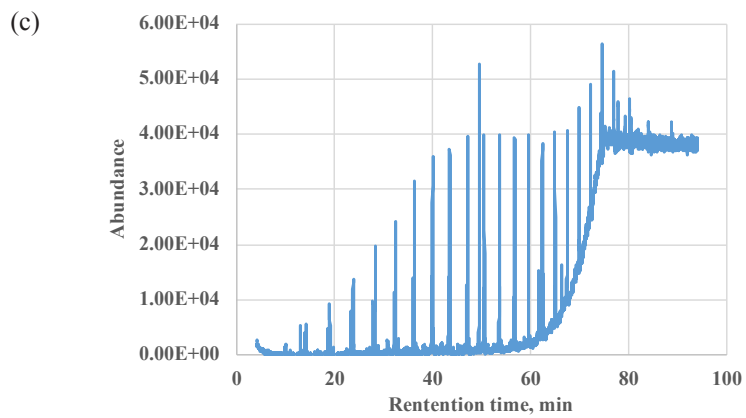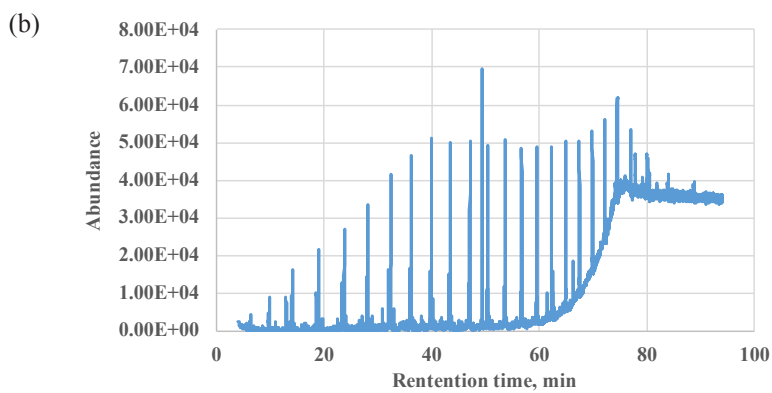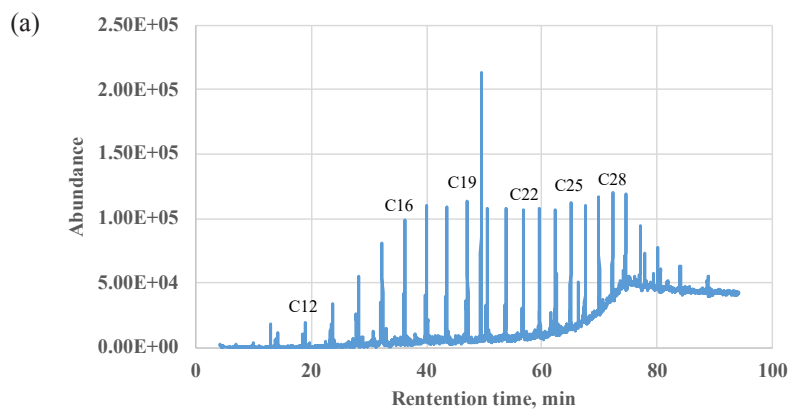
Fig. 3. Gas chromatograms of shale oil formed by oil shale pyrolysis at different temperatures: (a) 300 °C; (b) 350 °C; (c) 400 °C; (d) 450 °C; (e) 500 °C; (f) 550 °C.

When the retention time is 7.69, 31.09 and 61.48 min, the characteristic peak corresponds to the nitrogen-containing compounds. When the retention time is 26.56, 30.76, 49.4, 62.45 and 77.79 min, the characteristic peak corresponds to the oxygen-containing compounds. Figure 3 shows a few types of shale oil components at low temperatures, with the obvious peaks of oxygen- and nitrogen-containing compounds. More chemical bonds break in kerogen macromolecules under the high temperature, resulting in small molecular compounds. When the pyrolysis temperature is 300 °C and 350 °C, the proportion of hydrocarbon components in shale oil is small and the carbon content is mainly between C15 and C25. When the pyrolysis temperature is 550 °C, the carbon content of shale oil is concentrated between C11 and C25. Shale oil can be divided into three categories according to its composition: aliphatic hydrocarbons, aromatic hydrocarbons, and oxygenated and nitrogenous compounds. The boiling points of hydrocarbons with different carbon numbers are different. Thus, the boiling point of crude oil changes with the proportion of hydrocarbons with different carbon numbers. Figure 4 shows the proportion of hydrocarbons with different carbon numbers in shale oil.
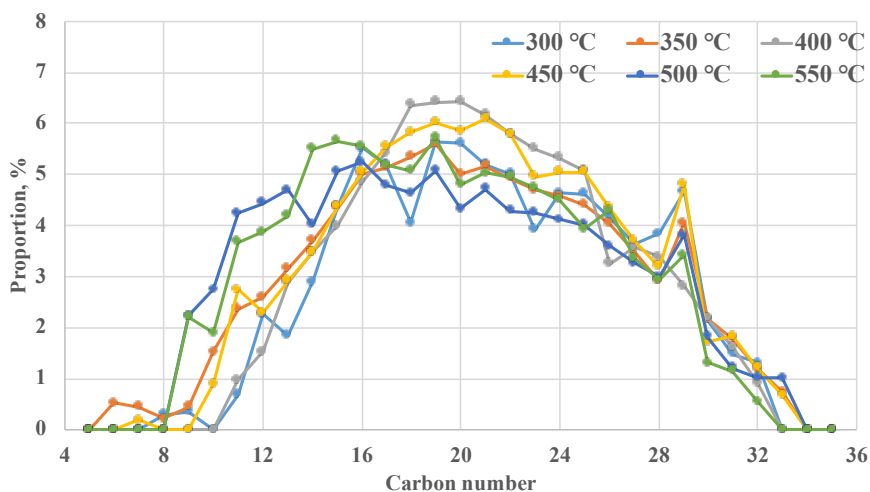

Fig. 4. Proportion of hydrocarbons with different carbon numbers in shale oil at different temperatures.

Based on a similar principle, the dry distillation products of shale oil can be divided into five categories: gasoline (C6–C10), kerosene (C11–C13), diesel (C14–C18), heavy oil (C19–C25) and lubricating oil (> C26) [20, 21]. The better the quality of shale oil is, the lighter the oil is. Therefore, the quality of shale oil can be reflected from the proportion of light components in shale oil.

Gasoline, kerosene and diesel are light oils. Therefore, it is considered that hydrocarbons with carbon numbers below 18 are light components, and those with carbon numbers above 19 are heavy components. Figure 5 shows the variation law of light component content in shale oil at different temperatures.
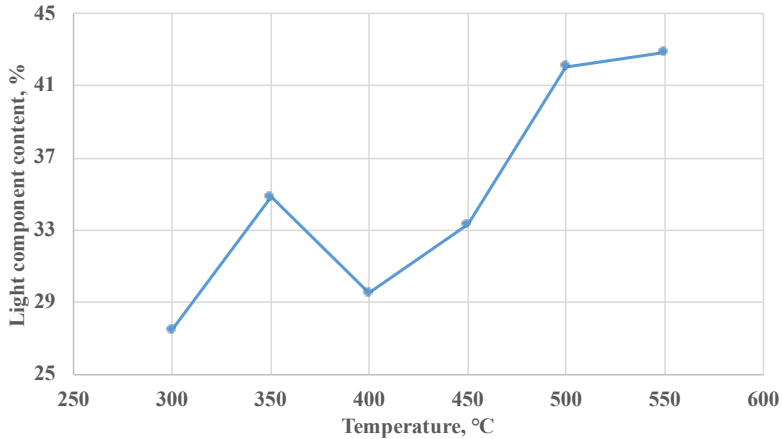

Fig. 5. Relationship between light component content in shale oil and injection temperature.

As shown in Figure 5, when the water vapor temperature is 300 °C, the content of the light components in shale oil is the lowest, approximately 27.49%. However, when the water vapor temperature exceeds 500 °C, the content of light components in shale oil exceeds 42%. As the temperature increases, the content of said components first increases, then decreases and thereafter continues to increase again. According to the variation trend of light component content with temperature, the pyrolysis process can be divided into three stages.

In the first stage (300 °C < temperature < 350 °C), the content of light components increases as the temperature increases. At the low-temperature stage, only weak bonds break in kerogen macromolecules. Moreover, as the temperature increases, chemical bonds with weak bond energy gradually break, forming more chain hydrocarbons with short chain lengths. Shale oil is mainly light; however, its output is small and the oil shale pyrolysis is insufficient.

In the second stage (350 °C < temperature < 400 °C), the content of light components decreases as the temperature increases. At this stage, the internal bond energy of kerogen molecules is strong and the chemical bonds break to form chain hydrocarbons with different lengths. However, due to the low H_2 content and poor activity of H_2 radicals in this temperature range, these radicals hardly participate in the pyrolysis reaction of kerogen, and numerous

short-chain hydrocarbon radicals recombine to form long-chain hydrocarbons with long-chain length, thus reducing the content of light components in shale oil.

In the third stage (400 °C < temperature < 550 °C), the content of light components rapidly increases as the temperature increases. When the temperature increases, the chemical bonds of the long-chain pyrolysis volatile matter will break, resulting in the conversion of long-chain hydrocarbons into short-chain hydrocarbons. Additionally, numerous short-chain hydrocarbon free radicals will be recombined with hydrogen-free radicals to form new short-chain hydrocarbons. Thus, the mutual combination of short-chain hydrocarbon free radicals is avoided to a certain extent, and the content of light components is improved.

3.4. Comparison of shale oil quality under different heating methods

The pyrolysis reaction of kerogen in oil shale is complex. Under different pyrolysis atmospheres, the pyrolysis process of kerogen, as well as the composition and quality of shale oil are different. Tao et al. [22] studied the law of oil and gas production of oil shale under direct retorting. The shale oil yield is relatively high in the temperature range of 490–510 °C. The obtained contents of gasoline, kerosene and diesel in shale oil are compared with those of the shale oil components obtained at 500 °C in this study (Fig. 6). Gasoline has poor stability and low boiling point. During the test, the temperature of liquid products after the heat exchange of the condenser is relatively high, resulting in the loss of gasoline. Therefore, the gasoline in shale oil obtained by direct retorting is slightly high. In general, the content of light components in shale oil is higher in water vapor atmosphere, especially the content of kerosene.
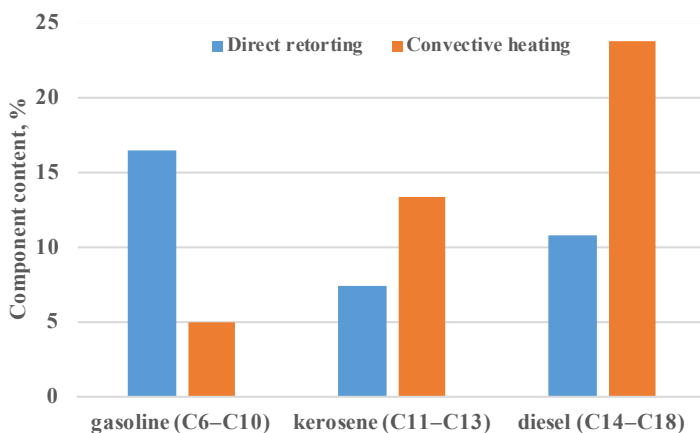

Fig. 6. Comparison of light components in shale oil under different heating modes.

The density of shale oil reflects its quality; the smaller the density is, the higher the quality is. Table 3 presents the density of shale oil obtained from direct distillation and convective heating.

Table 3. Density of shale oil under different heating modes [23, 24]

Heating mode	Retort furnace	Density, kg/m³
Direct retorting	Fushun	0.903
Convective heating	High-temperature water vapor	0.859

Established by the American Petroleum Institute (API), the API gravity is commonly used in the United States to express oil density. The API gravity is numerically opposite to the international density, i.e. the API gravity of light oil with low density is high. The higher the API gravity is, the lower the contents of water, salt and sulfur are and the higher the oil quality is. Therefore, the shale oil quality is characterized by its density. As presented in Table 3, the shale oil density obtained by convective heating is small. Compared with low-temperature dry distillation technology, the pyrolysis of oil shale by injecting water vapor can improve the quality of shale oil products. High-temperature steam has the characteristics of a small hydrogen bond and large specific volume. It can fully penetrate into the micropores in the rock mass and undergo water cracking reaction with the intermediate product (asphaltene) formed by kerogen cracking to reduce the content of high consistency and viscosity asphaltene.

In the petroleum industry, hydrotreating is often used to improve the quality of crude oil. The H_2 atmosphere improves the quality of shale oil. The pyrolysis environment of direct dry distillation has less H_2. Further, numerous short-chain hydrocarbons formed by kerogen pyrolysis are recombined into long-chain hydrocarbons, and the shale oil is heavy. The pyrolysis of oil shale by injecting water vapor is a hydrogen-rich pyrolysis environment. The hydrogenation reaction of shale oil significantly increases the content of short-chain hydrocarbons. The overall density of shale oil is low, and the oil is light.

4. Conclusions

This study analyzed the oil and gas products obtained from the oil shale pyrolysis by injecting water vapor using gas chromatography. It compared the quality of oil and gas products obtained from convective heating with that of the products obtained via direct dry distillation. The conclusions are as follows:

1. In the process of oil shale pyrolysis by injecting water vapor, the content of hydrocarbon gas with different carbon numbers first increases and then decreases. However, the content of low-carbon-number hydrocarbon gas is higher than that of high-carbon-number hydrocarbon gas. In other gases, the CO_2 content gradually decreases, whereas the H_2 content gradually increases. When the pyrolysis temperature is > 400 °C, the H_2 content increases rapidly.

2. The H_2 content in the gas components obtained from the pyrolysis of oil shale under water vapor action is approximately eight times higher than that obtained via direct dry distillation. The H_2 output has considerably increased through the pyrolysis of oil shale using high temperature. The calorific value of gas products increased significantly, and the kerogen of oil shale is pyrolyzed in a hydrogen-rich environment.

3. When the pyrolysis temperature is between 300 and 350 °C, the content of light components in shale oil increases. Moreover, when the pyrolysis temperature is between 350 and 400 °C, the content of light components in shale oil decreases. The content of light components in shale oil increases rapidly when the pyrolysis temperature is > 400 °C. Furthermore, when the temperature exceeds 500 °C, the content of light components in shale oil exceeds 42%.

4. Compared with the low-temperature dry distillation technology, shale oil hydrogenation occurs in water vapor environment, resulting in the low density and high quality of shale oil.

Acknowledgments

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