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Effects of retorting factors on combustion properties of shale char. 3. Distribution of residual organic matters

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ABSTRACT

Shale char, formed in retort furnaces of oil shale, is classified as a dangerous waste containing several toxic compounds. In order to retort oil shale to produce shale oil as well as treat shale char efficiently and in an environmentally friendly way, a novel kind of comprehensive utilization system was developed to use oil shale for shale oil production, electricity generation (shale char fired) and the extensive application of oil shale ash. For exploring the combustion properties of shale char further, in this paper organic matters within shale chars obtained under different retorting conditions were extracted and identified using a gas chromatography–mass spectrometry (GC–MS) method. Subsequently, the effects of retorting factors, including retorting temperature, residence time, particle size and heating rate, were analyzed in detail. As a result, a retorting condition with a retorting temperature of 460–490 °C, residence time of <40 min and a middle particle size was recommended for both keeping nitrogenous organic matters and aromatic hydrocarbons in shale char and improving the yield and quality of shale oil. In addition, shale char obtained under such retorting condition can also be treated efficiently using a circulating fluidized bed technology with fractional combustion.

1. Introduction

Oil shale, a fine-grained sedimentary material with organic matter called kerogen, is rich and widespread in the world. Based on the current data reported by Qian et al. [1], shale oil (converted from the in situ oil shale) accounts for about 400 billion tons, higher than that of crude oil (more than 300 billion tons). The uncertainty of petroleum prices, its growing worldwide consumption and limited availability have motivated more countries rich in oil shale resources to investigate means to produce and use shale oil as an alternative. By now, there have been three countries producing shale oil commercially: China, Estonia and Brazil. Total annual production of shale oil in the world accounts no more than one million tons currently, and it is predicted that till 2015, it may reach 3.5 million tons [1].

Shale char, formed in retort furnaces of oil shale, is a source of severe environmental pollution and is classified as a dangerous waste, containing several toxic compounds, such as water-soluble phenols, sulphide sulphur, polynuclear aromatic hydrocarbon (PAH) [2]. Provided that oil shale is exploited and retorted largely, it will become a serious issue how to treat shale char efficiently and in an environmentally friendly way. Some studies [3,4] have shown that shale char could be fired in circulating fluidized bed (CFB) furnaces, due to its fixed carbon and residual organics. Thus, the authors [5,6] recently recommended a new comprehensive utilization system of oil shale for shale oil production, electricity generation, oil shale ash utilization, economical efficiency and environmental protection. The system involves three subsystems: retort subsystem, where coarse oil shale is retorted to shale oil, hydrocarbon gases and shale char; combustion subsystem, where a mixture fuel of shale char and fine oil shale is fed to a CFB furnace to burn, in order to generate high-pressure steam which is used to supply heat and generate electricity via a traditional steam-electric power mode; and ash processing subsystem, where oil shale ash from the CFB furnace is treated [7] to produce chemical materials, building materials, etc. Compared with conventional retorting and burning technologies of oil shale, this comprehensive utilization technology has many advantages: (1) higher utilization efficiency of oil shale resources; (2) obtaining diversified products, such as shale oil, fuel gas and electricity; (3) lower pollutants emission; (4) successive use of the waste from one process as the input to another, which can decrease mass loss and energy loss. And for obtaining shale oil with a high yield as well as treating shale char efficiently in this system of oil shale, a series of experiments have been conducted for studying the effects of retorting factors on the shale oil yield [8] and shale char characteristics [9].

In this paper, organic matters within shale chars obtained under different retorting conditions were identified using a gas chromatography–mass spectrometry method, and then the effects of retorting factors, including retorting temperature, residence

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Table 1
Proximate and ultimate analysis of Dachengzi oil shales (wt.%, ar).

Proximate analysis		Ultimate analysis, wt.%	
Moisture, wt.%	11.54	C	27.33
Volatile matter, wt.%	36.21	H	3.59
Ash, wt.%	48.24	O	7.89
Fixed carbon, wt.%	4.01	N	0.57
Low heating value, kJ kg^{-1}	11076.07	S	0.84

O content was calculated by difference, ar: as received basis.

time, particle size and heating rate, were analyzed in detail, providing information necessary to study the combustion characteristics of shale char and design a circulating fluidized bed boiler of shale char.

2. Experimental

2.1. Samples

A representative oil shale sample used in this work was obtained from Dachengzi mine located in Huadian, Northeast China, the analytical data of which are given in Table 1. The samples were ground as received, sieved to the desired mesh, and dried at 45–50 °C to constant weight, and then stored in a desiccator for use.

As used herein, the term “shale char” refers to oil shale from which a portion of organic matters has been removed during retorting. An experimental retorting system of oil shale is presented in Fig. 1. Detailed instructions have also been given in reference [8]. An aluminum retort (inner diameter is Φ54, and height is 75 mm) was made according to the National Standards of China (SH/T 0508-92). 50 g of dried oil shale samples were placed inside the aluminum retort before each test and heated from ambient temperature to a retorting temperature. During the heating process, steam, non-condensable gases and shale oil were formed and passed into a conical flask immersed in a low-temperature trough with a temperature around ~0 °C. Both steam and shale oil were cooled and condensed at the bottom of the conical flask, and non-condensable gases exited the conical flask from a small mouth. These phenomena could be observed through the transparent conical flask. After the retorting temperature was held for a specified residence time *t*, the experiment ceased. Shale char and a mixture of shale oil and water were collected and weighted, and then the shale oil and water were separated and weighted.

In the present work, the studied retorting parameters include retorting temperature, residence time, heating rate and particle size. The effects of one retorting factor on organic matters within shale char was investigated while keeping the others constant. Table 2 gives the mass content of the products obtained under different retorting conditions.

2.2. Gas chromatography–mass spectrometry analysis

2.2.1. Extraction of organic matters within shale char

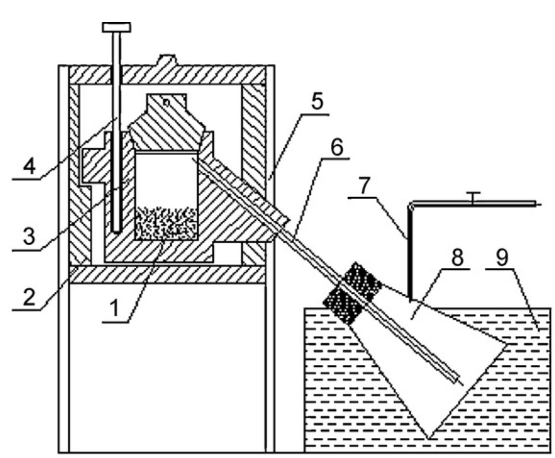
Organic matter of oil shale is present mostly as a complex combination of carbon, hydrogen, nitrogen, sulfur and oxygen—named kerogen that cannot be extracted with organic solvents. Upon heating, the kerogen is initially converted to bitumen at retorting temperatures ranging from 300 to 425 °C [10,11], and then to shale oil, non-condensable gas and coke, for which inorganic matter within oil shale supplies a certain catalysis. In this work, it was regarded that kerogen has decomposed almost completely and residual organic matters within shale char particles contained pyrolytic bitumen and shale oil coagulation, based on the following causes:

(1) Shale char was retained in the aluminum retort chamber for a specific time at a retorting temperature (≥430 °C) exceeding the pyrolysis temperature of kerogen.
(2) The studied particle size (<3 mm) easily produced a uniform temperature distribution within solid particles.

For extracting organic matter within shale char samples, solid samples of 0.5 g were first taken and plunged into a test tube containing 6 ml mixture solvent of acetone and hexane (3 ml:3 ml). Next, the test tube was fully stirred for 1 min and immersed in an ultrasonic container for 20 min. Then, the solid residue was separated from the solution by a high acceleration centrifuger, and the clear solution was collected and condensed to 200 μl. Finally, the obtained condensed solution was analyzed using a gas chromatography–mass spectrometry apparatus.

2.2.2. Gas chromatography–mass spectrometry (GC–MS)

GC–MS measurements were performed on each sample in an AutoSystem XL GC/Turbomass MS analyzer (Perkin-Elmer, USA). The capillary column was a 30 m × 0.25 mm DB-5MS with 0.25 μm film thickness. The gas chromatograph oven was held at 50 °C for 2 min and then programmed to 300 °C at 4 °C/min, held for 20.5 min. Injection temperature and injection amount was 280 °C and 1.0 μl, respectively, and helium was used as the carrier gas with a flow rate of 1 ml/min. The temperature of the GC–MS transfer line was



1 Oil shale; 2 Electric heater; 3 Aluminum retort; 4 Thermocouple; 5 Steel stand;
6 Copper tube; 7 Non-condensable gas tube; 8 Conical flask; 9 Low-temperature trough

Fig. 1. Experimental retorting system of oil shale.

Table 2
Mass content of products obtained under different retorting conditions.

Shale char	Retorting conditions				Retorting products/wt.%			
	Particle size/mm	Average heating rates, °C/min	Residence time/min	Retorting temperature/°C	Shale char	Shale oil	Water	Gas + mass loss
1#	<0.6 mm	7.33	6	430	80.06	4.88	11.63	3.44
2#	<0.6 mm	7.33	20	430	75.49	8.73	11.70	4.09
3#	<0.6 mm	7.33	40	430	70.24	13.51	11.78	4.47
4#	<0.6 mm	7.33	60	430	68.24	15.47	11.82	4.48
5#	<0.6 mm	7.33	40	460	65.14	17.97	11.86	5.03
6#	<0.6 mm	7.33	40	490	64.00	18.58	11.87	5.55
7#	<0.6 mm	7.33	20/40	520	63.35	18.78	11.88	6.00
8#	<0.28 mm	7.33	20	520	63.45	18.72	11.88	5.96
9#	<1.20 mm	7.33	20	520	63.48	18.93	11.88	5.70
10#	<3.0 mm	7.33	20	520	63.77	19.20	11.88	5.15
11#	<0.6 mm	9.57	20	520	63.41	18.79	11.88	5.92
12#	<0.6 mm	3.67	20	520	63.93	18.39	11.87	5.81

260 °C. The MS was operated at 200 °C in the electron impact mode (70 eV), scanning from m/z (mass to charge ratio) 33–600 in 0.4 s with 0.05 s interval time of the scan; the voltage of the photoelectric multiplier tube was 400 V. The mass spectral identifications were conducted by comparing to the NIST98 (National Institute of Standards and Technology, Gaithersburg, MD) mass spectral library as well as to the Wiley 7.0 (Wiley, New York, NY) mass spectral library.

3. Results and discussion

3.1. Retorting temperature

Fig. 2 presents GC–MS chromatograms of organic matters extracted from shale chars 3#, 5#, 6#, 7# obtained at different retorting temperatures. Combining the characteristics of organic matters with the comprehensive utilization technology of oil shale, the organic matters found in this work are classified as follows:

(1) Aromatic hydrocarbons (AH), which are very toxic to human being.
(2) Nitrogenous organic matters (NOM), which will be related to NO_x emissions from the CFB boiler of shale char.
(3) Hydrocarbons except aromatic hydrocarbons (HEAH), which play an important role in improving the quality and quantity of shale oil.
(4) Other organic matters, including ketone, alcohol and a few organic matters that cannot be identified accurately.

In addition, any organic matters with sulfur element cannot be found within shale char in this work. Trikkel et al. [2] found the same locations of Fe and S within shale char by the EDAX analysis, showing that S element of shale char mainly exists in pyrite.

Table 3 gives the distribution and content of organic matters within shale chars 3#, 5#, 6#, 7#, calculated from Fig. 2. The content of HEAH decreases evidently with elevating retorting temperature, of which the content of C16− organic matters decreases slowly and that of C16+ organic matters decreases dramatically, attributed to the cracking of high molecular weight HEAH within shale char to low molecular HEAH. Since hydrocarbon is an important component of shale oil and usually amounts to 40–50 wt.% of shale oil, it was recommended to try to extract the hydrocarbon out from shale char by increasing retorting temperature. However, higher retorting temperature easily results in cracking and coking of a portion of hydrocarbons, making shale oil yield decrease and non-condensable gases yield increase [12–16]. AH content

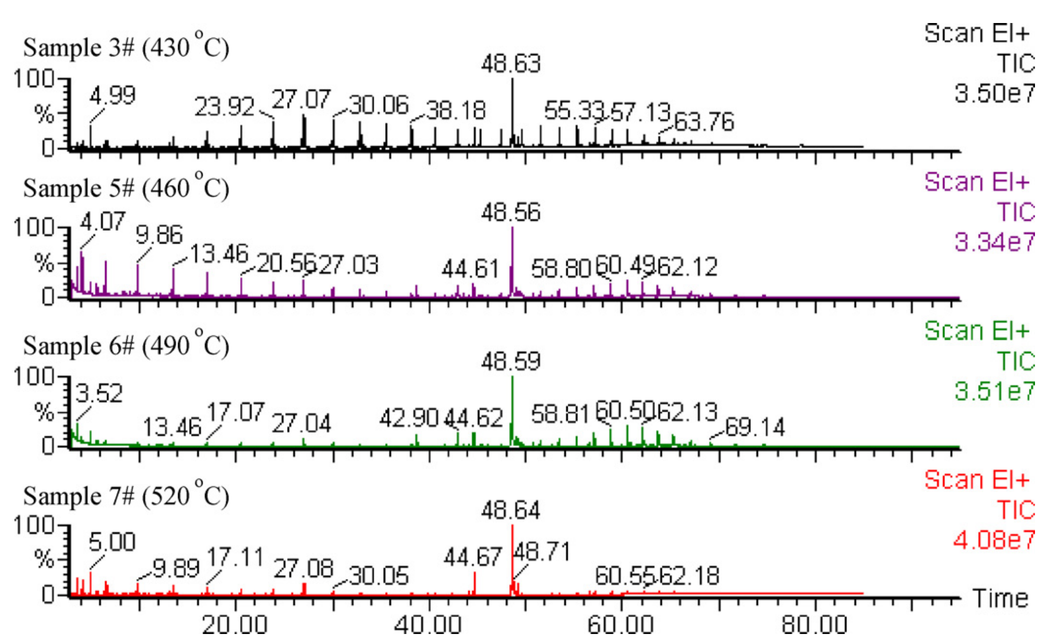


Fig. 2. GC–MS chromatograms of organic matters within shale char samples 3#, 5#, 6# and 7#.

Table 3
Distribution and content (area%) of organic matters within shale chars 3#, 5#, 6# and 7#.

Organic matters		Peak area, %			
		Sample 3#	Sample 5#	Sample 6#	Sample 7#
Total		31.700%	26.827%	25.320%	25.137%
AH	Monocyclic	0.659%	2.189%	0.886%	1.249%
	Polycyclic	0.358%	0.354%	0.522%	0.106%
	Total	1.018%	2.543%	1.408%	1.355%
HEAH	C16−	5.551%	8.218%	2.112%	5.199%
	C16+	12.899%	6.160%	8.920%	3.600%
	Total	18.449%	14.378%	11.032%	8.799%
NOM	–	4.822%	5.310%	7.814%	9.772%
Others	–	7.411%	4.596%	5.067%	5.212%

Table 4
Ultimate and proximate analysis of shale chars 3#, 5#, 6#, 7# (wt.%, ar).

	Sample 3#	Sample 5#	Sample 6#	Sample 7#
C	22.43	18.86	17.37	17.88
H	1.71	1.16	0.90	0.88
O	6.03	4.48	4.91	5.47
N	0.71	0.78	0.71	0.70
S	1.08	0.95	0.95	1.08
Moisture	0.91	0.88	1.01	0.21
Ash	67.13	72.89	74.15	73.79
Volatile matter	22.38	17.28	15.40	16.16
Fixed carbon	9.58	8.95	9.43	9.84
Remaining ratio of N	87.30	89.33	79.82	78.31

O content was calculated by difference, ar: as received basis.

within shale char initially increases and then decreases with the increasing retorting temperature, and its maximum value is 2.543% occurring at the retorting temperature of 460 °C. Since AH has an opposite effect on the stability of shale oil [17], a low retorting temperature may be recommended for retaining AH in shale char during retorting oil shale and then innocuously treating it with the CFB combustion technology. Nitrogen element within shale char mainly exists in $C_{16}H_{33}ON$ (∼44.67 min), $C_{18}H_{35}ON$ (∼48.63 min) and $C_{18}H_{37}ON$ (∼49.13 min) containing amide group (ONH_2). From Fig. 2 and Table 3, the content of these NOM increases evidently with increasing the retorting temperature, showing that they have higher boiling point and pyrolysis temperature than other organic matters, and do not easily decompose and vapor. Moreover, Table 4 gives the ultimate and proximate analysis of shale chars 3# and 5#–7# and the remaining ratio of N element within shale char, showing that there is still 78.31 wt.% of N of oil shale remaining in shale chars even when the retorting temperature is elevated to 520 °C, though the remaining ratio of N gradually decreases with an increase of the retorting temperature. The remaining ratio of N element herein is defined as follows:

$$\eta_N = \frac{C_s}{C_0} \times Y_{sc} \tag{1}$$

where, Y_{sc} describes the yield of shale char, seeing Table 2, C_0 and C_s are the content of N of oil shale and shale char, respectively, given in Tables 1 and 4. Since nitrogenous compounds have a toxic effect on the catalyzer used in refining shale oil, it was also suggested to keep organic nitrogen in shale char by selecting a low retorting temperature and then treat it efficiently using the subsequent CFB technology with fractional combustion. Considering the effects of retorting temperature on the organic matters within shale char mentioned above, a low retorting temperature range of 460–490 °C may be recommended for the comprehensive utilization system of oil shale, where the shale oil yield can exceeds 93.58% [8].

3.2. Residence time

Curves in Fig. 3 illustrate GC–MS chromatograms of organic matters within shale char samples 1#–4# obtained at the retorting temperatures of 430 °C in different residence time. Since organic matters within oil shale distribute widely and compactly within the framework of inorganic matters, its release needs adequate time and energy. As a result, the content of organic matters decreases gradually with the increasing the residence time, just as shown in Table 5.

As the residence time increases at the retorting temperatures of 430 °C, the content of HEAH decrease slightly, of which the content of C16− organic matters increases largely and that of C16+ organic matters decreases evidently, showing that lengthening the residence time at a low retorting temperature will make high molecular weight HEAH within shale char crack to low molecular HEAH remaining in the shale char, rather than be vaporized and leave shale char particles. The AH content changes slightly, showing that they have closer relation with retorting temperature than with residence time. The NOM content initially increases as residence time increases and then decreases after the residence time exceeds 40 min. Table 6 gives the ultimate and proximate analysis of shale chars 1#–4# and remaining ratio of N element within shale char. Ignoring small measure errors, it was found that more than 85.0 wt.% N element of oil shale still remains in shale chars at the retorting temperature of 430 °C with an increase of the residence time to 60 min, which is beneficial for improving the quality of shale oil and reducing the difficulty of refining shale oil. Among the four kinds of organic matters classified in this paper, only other organic matters, including ketone and alcohol, decrease largely, showing that the quality of shale oil obtained under such low retorting temperature should be low-grade. Besides, lengthening the residence time will increase input heat and heat loss of retort system, and make organic matters in shale char crack to non-condensable gas, resulting in decreasing shale oil yield [8]. So, it was recommended to elevate the retorting temperature properly and

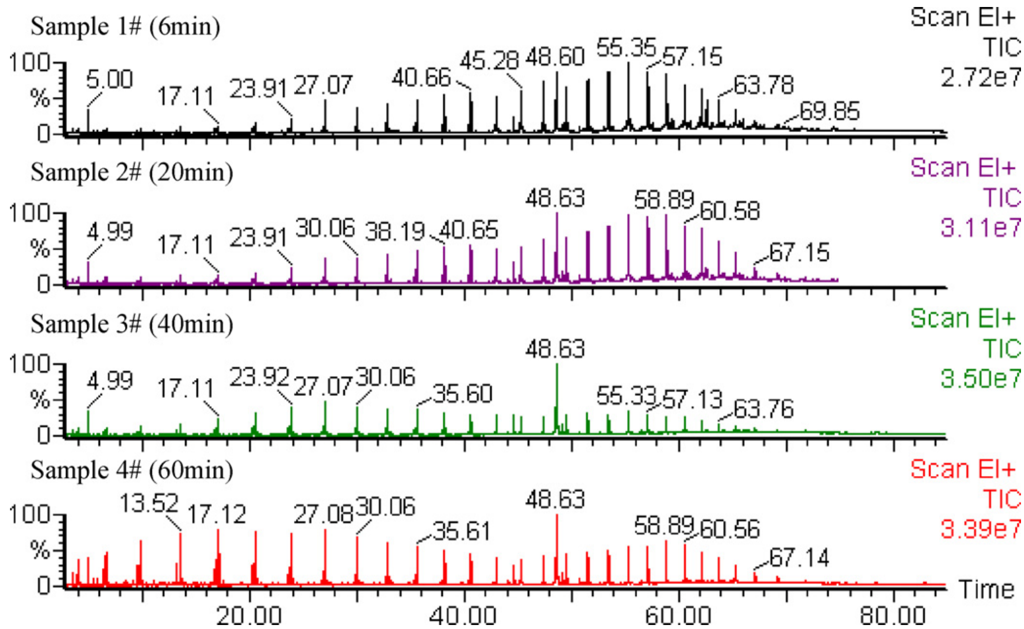


Fig. 3. GC–MS chromatograms of organic matters within shale char samples 1#–4#.

Table 5
Distribution and content (area%) of organic matters within shale chars 1#–4#.

Organic matters		Peak area, %			
		Sample 1#	Sample 2#	Sample 3#	Sample 4#
Total		40.170%	36.390%	31.700%	30.730%
AH	Monocyclic	0.514%	0.473%	0.659%	1.229%
	Polycyclic	0.534%	0.535%	0.358%	0.415%
	Total	1.048%	1.008%	1.018%	1.644%
HEAH	C16−	3.077%	2.806%	5.551%	9.314%
	C16+	21.109%	21.041%	12.899%	12.148%
	Total	24.186%	23.846%	18.449%	21.462%
NOM	–	2.495%	3.523%	4.822%	2.922%
Others	–	12.441%	8.013%	7.411%	4.702%

Table 6
Ultimate and proximate analysis of shale chars 1#∼4# (wt.%, ar).

	Sample 1#	Sample 2#	Sample 3#	Sample 4#
C	28.88	25.80	22.43	22.03
H	3.02	2.50	1.71	1.69
O	6.59	6.65	6.03	5.10
N	0.74	0.76	0.71	0.79
S	0.89	0.93	1.08	1.11
Moisture	0.82	0.89	0.91	0.88
Ash	59.06	62.48	67.13	68.40
Volatile matter	32.89	28.25	22.38	20.09
Fixed carbon	7.24	8.38	9.58	10.63
Remaining ratio of N	103.69	100.39	87.30	94.50

O content was calculated by difference, ar: as received basis.

shorten the residence time to <40 min. Considering the enrichment of AH and NOM within shale char, it is suitable that the subsequent CFB combustion technology is adopted for treating shale char cleanly.

3.3. Particle size

Similarly, from the GC–MS chromatograms of organic matters within shale char samples 7#–10# prepared from oil shale with different particle sizes, the distribution and content of these organic matters are given in Table 7. It was found that there exist differences in the content and distribution of HEAH: for shale char 8# (<0.28 mm), HEAH are small and distribute uniformly; with increasing particle size, C16+ HEAH increase largely and C16− HEAH decrease evidently. In addition, the content of NOM decreases with increasing the particle size.

Usually, small particles have a greater pore volume and surface area per unit mass, which are beneficial for the pyrolysis and diffusion of organic matters within particles. With increasing particle size, the bad heat conductivity of oil shale results in a large temperature difference between center and surface of particle during heating: the temperature of particle surface is high and that of par-

Table 7
Distribution and yield (area%) of organic matters within shale chars 7#–10#.

Organic matters		Peak area, %			
		Sample 8#	Sample 7#	Sample 9#	Sample 10#
Total		25.107%	25.137%	23.920%	23.380%
AH	Monocyclic	0.181%	1.249%	0.548%	0.325%
	Polycyclic	0.055%	0.106%	0.553%	0.360%
	Total	0.236%	1.355%	1.100%	0.685%
HEAH	C16−	1.356%	5.199%	0.770%	0.704%
	C16+	1.122%	3.600%	9.094%	6.979%
	Total	2.478%	8.799%	9.865%	7.683%
NOM	–	18.654%	9.772%	8.659%	10.989%
Others	–	3.739%	5.212%	4.296%	4.024%

Table 8
Distribution and yield (area%) of organic matters within shale chars 7#, 11# and 12#.

Organic matters		Peak area, %		
		Sample 11#	Sample 7#	Sample 12#
Total		24.740%	25.137%	25.720%
AH	Monocyclic	0.260%	1.249%	0.103%
	Polycyclic	0.299%	0.106%	0.252%
	Total	0.559%	1.355%	0.355%
HEAH	C16−	1.017%	5.199%	0.877%
	C16+	8.637%	3.600%	8.305%
	Total	9.654%	8.799%	9.182%
NOM	–	7.736%	9.772%	8.686%
Others	–	6.791%	5.212%	7.497%

ticle center is low, resulting in that high molecular weight HEAH near the center of particle decompose late and incompletely. In addition, a portion of high molecular weigh HEAH near the particle center will possibly crack, agglomerate and form intermediate products with good heat stability together with metals element [18], due to the resistance of ash layer. These reactions will result in the increase of high molecular weigh HEAH with the increasing particle size. Since smaller particle size will result in decreasing yield of shale oil [8], a middle particle size is helpful for increasing the yield and quality of shale oil.

3.4. Heating rate

Table 8 gives the distribution and content of organic matters within shale char samples 7#, 11# and 12# obtained at different heating rates, showing that the heating rate lower than 10 °C/min has little effect on the distribution and content of organic matters within shale char.

4. Conclusions

Shale char, a dangerous waste formed in oil shale retort furnaces, may be treated efficiently with a circulating fluidized bed technology. For exploring its combustion properties further, organic matters within shale chars obtained under different retorting conditions were extracted and identified using a gas chromatography–mass spectrometry (GC–MS) method, and the effects of retorting factors were analyzed in detail. The main conclusions and recommendations are given below:

(1) Combining the characteristics of organic matters with the comprehensive utilization technology of oil shale, organic matters found in this work are classified as aromatic hydrocarbons, nitrogenous organic matters, hydrocarbons except aromatic hydrocarbons, and other organic matters including ketone and alcohol. Any organic matters with sulfur element cannot be found within shale char.
(2) A low retorting temperature ranging from 460 to 490 °C was recommended for both keeping nitrogenous organic matters and aromatic hydrocarbons in shale char and improving the yield and quality of shale oil.
(3) Excessively lengthening residence time at a low retorting temperature will result in the enrichment of aromatic hydrocarbons and nitrogenous organic matters within shale char and the low-grade quality of shale oil. As a result, it was recommended to elevate the retorting temperature properly and shorten the residence time to <40 min.
(4) Particle size has an effect on nitrogenous organic matters and hydrocarbons except aromatic hydrocarbons within shale char. For increasing the yield and quality of shale oil, a middle particle size was recommended.
(5) Heating rate lower than 10 °C/min has little effect on the distribution and content of organic matters within shale char.

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