

AUTOCLAVE PYROLYSIS EXPERIMENTS OF CHINESE LIUSHUHE OIL SHALE TO SIMULATE IN-SITU UNDERGROUND THERMAL CONVERSION

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Abstract. Laboratory level autoclave pyrolysis experiments of Chinese Liushuhe oil shale have been conducted at different heating temperatures and pressures to simulate underground in-situ thermal conversion. Effects of water and metallic ions on oil shale pyrolysis have been investigated. In the presence of water, pyrolysis of oil shale generates thermal bitumen as an intermediate product at first, then pyrolysis of bitumen produces shale oil and gas takes place. With increasing temperature, the yields of shale oil and gas increase gradually, while the yield of bitumen initially increases (max. at 300 °C) and then decreases. Compared with the modified Fisher Assay, autoclave pyrolysis temperature of simulated experiment was 100 °C lower when reaching the same conversion level of oil shale. Analysis of the gas chromatography–mass spectrometry (GC-MS) showed that main chemical compounds of the light fraction in shale oil are phenols and benzenes. The analyses of gas chromatography showed high percentage of carbon dioxide and low percentage of hydrocarbons. The research results could provide theoretical information for underground in-situ thermal conversion of oil shale.

Keywords: autoclave pyrolysis, oil shale, bitumen, intermediate product, shale oil, Fischer Assay, GC-MS.

1. Introduction

Oil shale for producing shale oil as a kind of petroleum alternative has now been paid much more attention to at the time of high crude oil prices. Oil

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shale is widely distributed in the world. However, different deposits have different characteristics such as burial depth and shale oil yield, from the utilization point of view [1–3]. Oil shale retorting for producing shale oil is classified into aboveground retorting and underground in-situ retorting. Underground in-situ retorting refers to the direct heating of buried oil shale by introducing air or/and fuel gas or electric rod to pyrolyze it and to produce shale oil vapor, shale gas, and shale char. The shale oil and gas are collected and transported to the surface. For underground retorting, there is no mining of oil shale, hence no cost; but as the shale oil and pyrolysis gas formed underground are easily leaking to surroundings, the recovery rate is lower. Underground in-situ retorting is suitable for processing rich oil shale with deep burial depth. However, due to the complexity of the in-situ processing, there is no commercial production of shale oil by the underground retorting in the world nowadays. It means that more practical work and fundamental research should be done.

However, due to high crude oil prices, oil companies and energy related research institutions have started to pay more attention to underground retorting. Shell Oil Company is continuing oil shale field pilot tests of “In-Situ Conversion Process” in Rio Blanco, Colorado [4]. ExxonMobil is developing the “Electrofrac Process” for in-situ oil shale pilot retorting in western Colorado [5]. Idaho National Laboratory and several US universities are involved in fundamental research work for simulating in-situ retorting [6]. Tallinn University of Technology in Estonia has conducted a study on oil shale pyrolysis in autoclave, which could be taken as reference for in-situ retorting [7]. University of Petroleum in China and Taiyuan University of Technology have conducted laboratory simulation study on underground pyrolysis of oil shale [8, 9].

This paper deals with the autoclave pyrolysis experiments of Chinese Liushuhe oil shale to simulate in-situ underground thermal conversion, including the retorting at different final temperatures and pressures. The effect of water and metallic ions on the pyrolysis yield, and also the chemical characteristics of the shale oil and gas obtained have been investigated. The obtained results could provide basis for the first underground retorting program of oil shale in China.

2. Experimental

2.1. Samples

Liushuhe oil shale samples were analyzed in this research work. Liushuhe is located in Linkou County, Heilongjiang Province, China. The oil shale deposits are buried underground at the depth of about 500 m. The oil shale samples were crushed, sieved and dried to the size of 3–6 mm. The sampling procedure followed the Chinese Standard SH/T 0508-91.

2.2. Fundamental analysis

Fundamental properties of oil shale samples were investigated by using following analytical methods: elemental analysis, Fischer Assay oil yield determination, differential scanning calorimetry (DSC) coupled with thermogravimetric analysis (TGA), determination of total organic carbon (TOC), and analysis of shale oil and gas properties.

The elemental analysis is really the elemental composition determination of the organic matter of the oil shale: 100 g of oil shale was treated repeatedly with 20% HCl to remove carbonate minerals and with 40% HF to remove oxide minerals and silicates; the weighed residue could be considered as the percentage of organic matter contained in the oil shale sample; thus the organic matter was used for determination of elemental composition by using the conventional elemental analysis apparatus.

All the analytical apparatus used were calibrated for temperatures and other parameters (such as gas flow rate, heating rate), according to the relevant Chinese Standards. All the confirmed analytical data met the requirements for reproducibility and repeatability, according to Chinese Standards. For example, by using Fischer Assay apparatus, the difference of the shale oil yield of two repeated tests should not exceed 0.4%.

2.3. Autoclave pyrolysis simulation experiments

The experimental system for simulating autoclave pyrolysis is presented in Fig. 1.

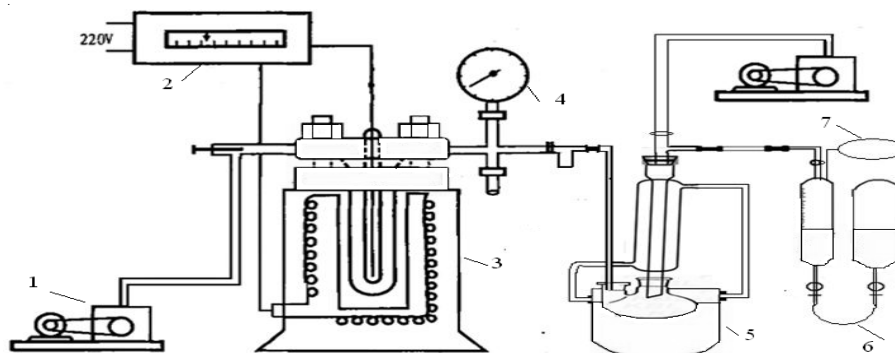


Fig. 1. Autoclave pyrolysis simulation experiment.

1 – vacuum pump, 2 – temperature controller, 3 – autoclave, 4 – pressure gauge, 5 – cooling pump, 6 – collector, 7 – gas bag.

Small quantity of particulate oil shale sample (30 g) and 50 g of pure water were put into the autoclave. Water was oversaturated in autoclave at different temperatures. Autoclave was sealed, filled with nitrogen, and

heated according to the designated heating program: 15–20 °C for 5h; final temperature (200 °C) was reached with heating rate of 2.5 °C/h. After cooling, the exit valve was opened, the pyrolysis oil, gas and water vapor were collected, cooled and condensed. The shale oil and water in the collector were separated and the shale oil was sealed into a bottle for GC-MS analysis. The shale char in the autoclave was taken out after cooling, dried, weighed and extracted with dichloromethane in a Soxhlet extractor to get pyrobitumen as the pyrolysis intermediate. The pyrobitumen was put into a sealed bottle for GC-MS analysis. The pyrolysis gas was collected into a gas bag for the gas chromatographic analysis. The water of pyrolysis was collected for pH analysis.

3. Results and discussion

3.1. Fischer Assay and TOC data

Fischer Assay and TOC data of Liushuhe oil shale are presented in Table 1.

Table 1. Fischer Assay and TOC data of Liushuhe oil shale, dry basis

Oil yield, %	Shale char yield, %	Water yield, %	Pyrolysis gas yield, %	TOC, %
11.63	64.88	8.75	14.73	49.54

As seen in Table 1, Liushuhe oil shale has high yield of shale oil and pyrolysis gas and high TOC content. Due to its burial depth (500m underground), it is suitable for in-situ retorting.

3.2. Elemental analysis of the oil shale

It was determined that the Liushuhe oil shale is composed of organic matter (57.03%) and mineral matrix (42.97%). Elemental composition of the organic matter of Liushuhe oil shale is presented in Table 2.

Table 2. Elemental composition of organic matter of Liushuhe oil shale, wt%

Basis	Total organics %	C%	H%	O%	N%	S%	H/C, mol ratio	O/C, mol ratio
On oil shale	57.02	37.27	3.46	15.05	1.05	0.19	1.11	0.30
On organics	100.00	65.37	6.07	26.39	1.84	0.33	1.11	0.30

As seen in Table 2, Liushuhe oil shale has low H/C mol ratio and high O/C mol ratio, belonging to humic-sapropelic type, i.e., Type II oil shale, according to van Krevelen diagram.

3.3. Organic matter distribution in the pyrolysis process

Organic matter distribution in the Liushuhe oil shale by Fischer Assay retorting is presented in Table 3.

Table 3. Organic matter distribution in the Liushuhe oil shale pyrolysis process, wt%

Sample	Basis	Shale oil*	Shale gas	Shale char**	Pyrolysis water	Total
Organic matter	On organics	20.40	25.83	38.43	15.34	100.00
Organic matter	On oil shale	11.63	14.73	21.91	8.75	57.02

* The shale oil yield for Liushuhe oil shale on the basis of its organic matter content is not high.

** The shale char is composed of 21.91% organics and 42.97% minerals.

3.4. Fischer Assay pyrolysis gas composition analyzed by gas chromatography (GC)

Pyrolysis gas composition after the analysis of GC by the method of Fischer Assay is presented in Table 4.

Table 4. Fischer Assay pyrolysis gas composition analyzed by GC

Component	Fraction, vol%	Component	Fraction, vol%
CH ₄	5.6128	C ₃ H ₆	0.7909
C ₂ H ₆	1.3997	i-C ₄ H ₁₀	0.0647
C ₂ H ₄	0.8148	n-C ₄ H ₁₀	0.2325
C ₂ H ₂	0.1401	t-2-C ₄ H ₈	0.0814
C ₃ H ₈	0.7739	n-C ₄ H ₈ -1	0.1426
i-C ₄ H ₈	0.1701	C ₅ =	0.0287
c-2-C ₄ H ₈	0.0630	n-C ₆ H ₁₄ & C ₆₊	0.2814
1,3-C ₄ H ₆	0.0600	CO ₂	72.2805
i-C ₅ H ₁₂	0.0802	CO	15.4642
n-C ₅ H ₁₂	0.1246	H ₂	1.4538

As seen in Table 4, the pyrolysis gas contains high percentage of carbon dioxide and monoxide, and lower percentage of hydrocarbons. It indicates that the Liushuhe oil shale is of younger formation; it contains plenty of carboxylic functional groups and forms many carbon oxides during pyrolysis.

3.5. GC-MS analysis of Fischer Assay shale oil

The content of aromatics and phenols of the Fischer Assay shale oil as analyzed by GC-MS apparatus is presented in Table 5.

Table 5. Content of aromatics and phenols of the Fischer Assay shale oil obtained at 520 °C by GC-MS analysis

Component	Fraction, wt%	Component	Fraction, wt%
p-dimethyl benzene	3.11	1,3-dimethyl phenol	1.51
phenol	3.70	1,2,3,4-tetramethyl benzene	0.92
1,3,5-trimethyl benzene	1.02	p-ethyl phenol	0.93
1,2,4-trimethyl benzene	1.59	o-ethyl phenol	0.92
m-methyl phenol	1.74	dimethoxy benzene	1.14
p-methyl phenol	3.95	o-dimethoxy benzene	1.90
m-methoxyphenol	1.97	2-methoxy-4 carbonyl-phenol	1.17

The high content of aromatics and phenols presented in Liushuhe shale oil also indicates that this oil shale is of younger formation.

3.6. TGA/DSC analysis of Liushuhe oil shale

The TGA/DSC curves of Liushuhe oil shale are shown in Figure 2. The left y-axis indicates the scale for TGA mass loss curve; the right y-axis indicates the scale for thermal reaction DSC curve. In Figure 2, it can be seen that the hydrocarbon generation from Liushuhe oil shale mainly proceeds in the temperature ranging from 300 to 600 °C, with 80% of total weight loss. It is shown in detail that at about 100 °C some water evaporates, from 410 to 520 °C large amount of pyrolysis products evolves due to the pyrolysis of oil shale, and after about 700 °C a peak on DSC curve appears, indicating the decomposition of carbonates present in oil shale.

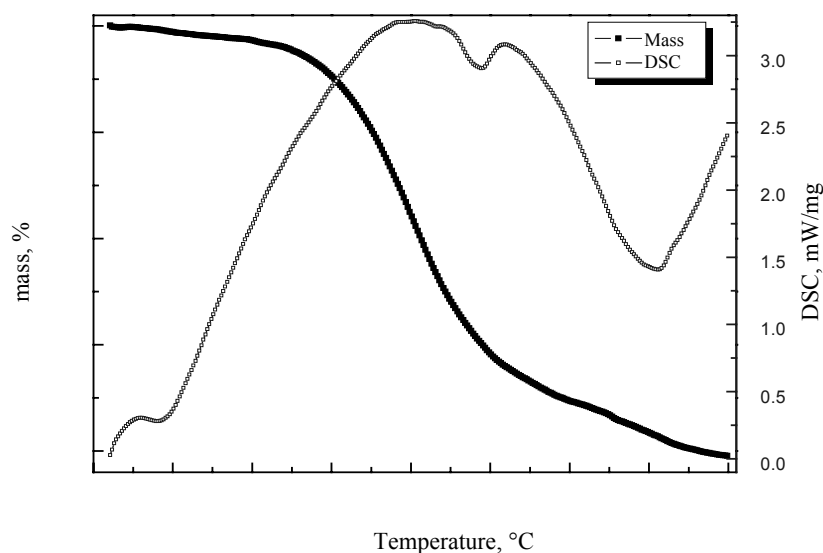


Fig. 2. TGA/DSC analysis of Liushuhe oil shale.

3.7. Simulation testing of autoclave pyrolysis

Product yields of autoclave pyrolysis at different final temperatures are presented in Table 6. As mentioned above, the pyrobitumen was extracted from the remaining shale char after each autoclave test.

Table 6. Autoclave pyrolysis yields of Liushuhe oil shale at different final temperatures

Final temperature, °C	Pyrolysis gas, wt%	Shale oil, wt%	Pyrobitumen, wt%
275	6.76	0.00	1.33
300	7.37	0.01	1.76
320	10.91	0.82	0.79
340	12.36	3.47	0.80
360	13.96	5.94	0.31
370	14.72	7.90	0.32

In Table 6, it can be seen that for Liushuhe oil shale the pyrolysis gas and the shale oil yields increase with increasing temperature; as at first the pyrobitumen yield increases with the increase of temperature, reaching the maximum at 320–340 °C, and then decreases, it means that the pyrobitumen is the pyrolysis intermediate.

3.8. Comparison of the autoclave pyrolysis yields with the modified Fischer Assay results

The autoclave pyrolysis yields at different final temperatures under different final pressures compared with the modified Fischer Assay yields are presented in Table 7. It should be noted that for simulating underground in-situ retorting, the oil shale is mixed with pure water for autoclave pyrolysis, while the modified Fischer Assay tests are conducted with the dried oil shale, but with the same temperature rising program as that of autoclave experiments, under atmospheric pressure.

Table 7. Comparison of autoclave pyrolysis yields with the modified Fischer Assay results

Item	Autoclave	Fischer	Autoclave	Fischer	Autoclave	Fischer	Autoclave	Fischer
Temperature, °C	300	300	320	320	350	350	370	370
Pressure, MPa	6.4	0.1	8.8	0.1	13.8	0.1	17.8	0.1
Shale oil, wt%	0.01	0.00	0.82	0.81	4.16	1.85	7.90	3.33
Shale gas, wt%	7.37	4.13	10.91	5.43	12.93	7.27	14.72	8.54
Shale char, wt%	89.17	93.93	85.87	91.37	79.54	87.71	73.36	83.76
Water, wt%	3.45	1.94	2.40	2.39	3.37	3.17	4.02	4.37

It is shown in Table 7 that despite of high pressure, the shale oil and gas yields of autoclave pyrolysis are higher than that of Fischer Assay at the same final temperatures, (at 350 °C oil yield is 2.31% higher, gas yield is 5.66% higher; at 370 °C oil yield is 4.57% higher, gas yield is 6.18% higher and the initial pyrolysis temperature for autoclave retorting is lower than that

of Fischer Assay. It may be due to the presence of water promoting the oil shale pyrolysis.

3.9. Autoclave shale oil composition at different final temperatures

The content of alkanes, cyclohexanes and olefins in the light fractions of shale oil after the autoclave pyrolysis (with oversaturated water) at different final temperatures, was detected by GC-MS analysis and presented in Table 8.

Table 8. Content of alkanes, cyclohexanes and olefins in the light fraction of the shale oil after autoclave pyrolysis at different final temperatures

Temperature, °C	300	320	340	360	370
Alkanes, wt%	3.0	3.0	2.8	2.7	2.6
Cyclohexanes, wt%	1.8	2.0	1.8	1.3	1.3
Olefins, wt%	2.8	3.2	2.6	2.0	1.8

It can be seen that the content of alkanes decreases with the increasing temperature, while the content of cyclohexanes and olefins increases with the increasing temperature at first to a maximum value and then decreases.

3.10. Autoclave shale gas composition at different final temperatures

The composition of shale gas obtained by autoclave pyrolysis of Liushuhe oil shale (with oversaturated water) at different final temperatures was analyzed by GC and results are presented in Table 9.

Table 9. Composition of shale gas obtained by autoclave pyrolysis of Liushuhe oil shale at different final temperatures

Vol, %	300 °C	320 °C	340 °C	360 °C	370 °C
H ₂	0.4778	0.6623	0.9178	1.1246	1.2147
CH ₄	0.5742	1.0340	1.3129	2.0092	2.4723
C ₂ H ₆	0.0451	0.1003	0.2207	0.4241	0.6036
C ₂ H ₄	0.0607	0.0649	0.0557	0.0459	0.0398
C ₂ H ₂	0	0.0215	0.0249	0.0344	0.0407
C ₃ H ₈	0.0663	0.1327	0.2492	0.4126	0.5604
C ₃ H ₆	0.0708	0.0972	0.1015	0.1065	0.1053
i-C ₄ H ₁₀	0	0.0142	0.0309	0.0619	0.0900
n-C ₄ H ₁₀	0	0.0114	0.0237	0.0518	0.0818
t-2-C ₄ H ₈	0	0.0107	0.0128	0.0202	0.0237
n-1-C ₄ H ₈	0	0.0125	0.0125	0.0146	0.0154
i-C ₄ H ₈	0.0272	0.0411	0.0421	0.0385	0.0336
c-2-C ₄ H ₈	0	0	0.0096	0.0177	0.0236
i-C ₅ H ₁₂	0	0.0106	0.0129	0.0289	0.0485
n-C ₅ H ₁₂	0	0	0.0072	0.0156	0.0262
C ₅ ⁼	0.0224	0	0.0249	0.0344	0.0121
CO ₂	97.9452	97.3660	96.6226	95.2022	94.5319
CO	0.7102	0.4073	0.3035	2.0092	2.4723
Calorific value, kJ/mol	15	25	32	40	60

It can be seen that the shale gas contains mainly CO₂, followed by CO, with the total hydrocarbon content of no more than 2-6%. However, the CO₂ and CO content slightly decreases with increasing temperature and the calorific value of gas increases with the increase of temperature.

3.11. Analysis of the autoclave pyrolysis shale water

The pH values of the autoclave pyrolysis shale water produced at different final temperatures are presented in Table 10.

Table 10. pH values of the autoclave pyrolysis shale water at different final temperatures

Temperature, °C	300	320	340	350	360
pH	4.12	4.09	4.01	3.96	3.94

It can be seen in Table 10 that the shale water has acidic characteristics. The pH value decreases slightly with the increasing temperature.

The concentrations of metals in the shale water that was produced at 360 °C are presented in Table 11.

Table 11. Concentrations of metals in the shale water that was produced at 360°C

Metal	Ca	Al	Fe	Na	K	Ba	Zn	Mg
ppm	17.3	0.4	0.1	6.0	15.5	0.4	0.6	1.0

Hazardous metals were not found.

3.12. Effect of different metal sulfates on the shale oil yields of autoclave pyrolysis and its characteristics

Based on the metal ion concentrations present in the underground water of Liushuhe oil shale region, several corresponding metal sulfates, including Cu, Fe, Ni and Na sulfate, were added into the water and oil shale mixture for autoclave pyrolysis tests at the final temperature of 360 °C (final pressure about 15 MPa). The quantity of the metal ions accounts for 0.5% of the oil shale used. The pyrolysis yields for each test with different kind of metallic sulfate addition compared with the autoclave oil shale pyrolysis with pure water are listed in Table 12.

Table 12. Effect of different metal ions on the autoclave oil shale pyrolysis yields (final temperature 360 °C)

Metallic ion	Without metal	Cu	Ni	Na	Fe
Shale oil, wt%	5.94	3.93	4.79	4.98	6.04
Shale gas, wt%	13.96	16.07	15.63	16.58	14.30
Pyrobitumen, wt%	0.31	0.38	0.27	0.75	0.33

It can be seen in Table 12, that the presence of Fe ion promotes slightly the formation of shale oil, while the others prevent the shale oil formation; however, the presence of all metal ions promote the formation of shale gas.

The alkanes, cyclohexanes and olefins present in the light fractions of the shale oil obtained from the autoclave pyrolysis, oversaturated with water, and with and without different metallic ions, at final temperature of 360 °C are presented in Table 13.

Table 13. Alkanes, cyclohexanes and olefins in the light fractions of the shale oil obtained from autoclave oil shale pyrolysis, oversaturated with water, with and without addition of different metallic ions

Metallic ions	Without metal	Cu	Ni	Na	Fe
Alkanes	2.7	3.2	2.6	2.7	2.6
Cyclohexanes	1.3	1.7	1.4	1.6	1.4
Olefins	2.0	1.6	2.0	2.2	2.0

It can be seen in Table 13 that the presence of different metallic ions has small effect on the group composition of light fraction in shale oil.

The composition and calorific value of shale gas obtained from autoclave pyrolysis of Liushuhe oil shale, oversaturated with water, with and without addition of different metallic ions, at final temperature of 360 °C are presented in Table 14.

Table 14. Composition and calorific value of shale gas from autoclave pyrolysis of Liushuhe oil shale, oversaturated with water, with and without metallic ions, at final temperature of 360 °C

Composition	Without metal	Cu	Ni	Na	Fe
H ₂	1.1246	1.1888	1.0876	0.9294	1.1061
CH ₄	2.0092	1.7636	1.8319	1.9148	1.8370
C ₂ H ₆	0.4241	0.3006	0.3227	0.3362	0.3474
C ₂ H ₄	0.0459	0.0320	0.0378	0.0341	0.0374
C ₃ H ₈	0.4126	0.2763	0.3093	0.3129	0.3522
C ₃ H ₆	0.1065	0.0715	0.0802	0.0764	0.0825
i-C ₄ H ₁₀	0.0619	0.0457	0.0487	0.0494	0.0565
n-C ₄ H ₁₀	0.0518	0.0409	0.0460	0.0471	0.0620
t-2-C ₄ H ₈	0.0202	0.0190	0.0188	0.0197	0.0215
n-1-C ₄ H ₈	0.0146	0.0108	0.0122	0.0137	0.0136
i-C ₄ H ₈	0.0385	0.0310	0.0337	0.0316	0.0353
c-2-C ₄ H ₈	0.0177	0.0162	0.0169	0.0179	0.0193
i-C ₅ H ₁₂	0.0289	0.0241	0.0274	0.0272	0.0329
n-C ₅ H ₁₂	0.0156	0.0144	0.0173	0.0166	0.0237
C ₅ =	0.0344	0.1164	0.1655	0.1330	0.1358
CO ₂	95.2022	95.7573	95.5927	95.7970	95.4638
CO	2.0092	0.2445	0.2949	0.2817	0.3072
Calorific value, kJ/mol	40.00	38.56	40.77	41.34	44.02

Above table shows that the composition and calorific values of the pyrolysis gas do not vary much in the presence of different metallic ions.

4. Conclusions

Fundamental analysis of Chinese Liushuhe oil shale has been conducted. The analysis shows that the Liushuhe oil shale has high content of organics, high shale oil yield and high shale gas yield with low H/C and high O/C organic mol ratio, belonging to humic-sapropelic type of kerogen. The analysis also shows that the Liushuhe oil shale is suitable for in-situ underground pyrolysis, mainly due to its high oil yield and deep burial depth.

Laboratory level autoclave pyrolysis experiments were conducted to simulate underground in-situ thermal conversion of the Liushuhe oil shale. Tests were run at different heating temperatures and pressures, with and without inclusion of some metallic ions; and oversaturation with water was used. The results are summarized as follows:

1. The autoclave pyrolysis gas and the shale oil yields increase with the increasing temperature; the yield of pyrobitumen as intermediate increases with increasing temperature at first, reaching maximum at 320 °C–340 °C, and then decreases.

2. Although the pressure in the autoclave pyrolysis is higher compared to the atmospheric pressure used in the modified Fisher Assay test, the shale oil and gas yields are higher in the autoclave pyrolysis when the same final temperature is applied in both tests. The initial pyrolysis temperature for autoclave retorting is lower than that of Fischer Assay. It may be due to the presence of water promoting the oil shale pyrolysis.

3. GC-MS analysis of the light fraction of shale oil obtained from autoclave pyrolysis of oil shale has been carried out. The results indicate that the content of alkanes decreases with increasing temperature, while the content of cyclohexanes and olefins increases with increasing temperature to a maximum value and then decreases.

4. GC analysis of the shale gas obtained from autoclave pyrolysis was carried out. The results indicate that the shale gas mostly contains CO₂, followed by CO, and the total hydrocarbon content is no more than 2–6%. However, the content of CO₂ and CO slightly decrease with increasing temperature. The calorific value of gas increases with temperature increase.

5. In the autoclave pyrolysis, the effect of metallic ion addition (Cu, Ni, Na, Fe sulfate) on shale oil yield and the product characteristics is small compared to the effect of increasing pyrolysis temperature.

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