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Effect of hydrothermal pretreatment on product distribution and characteristics of oil produced by the pyrolysis of Huadian oil shale

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ABSTRACT

In this work, Huadian oil shale from China was treated by hydrothermal pretreatment at 200 °C with 1.0–2.5 h in order to investigate the effect of hydrothermal pretreatment on pyrolysis product distribution and characteristics of oil. The differences in the elemental composition and thermal behavior between the untreated and treated oil shale were analyzed and compared. The hydrothermal treatment process could decompose oxygen functional groups and remove some water soluble inorganics in oil shale, which decreased the formation of gas and water during the pyrolysis. However, hydrothermal pretreatment was conducive to increasing shale oil yield. The maximum of oil yield was obtained at the pretreatment time of 2.0 h. The enhancement of the free-radical reactions during the pyrolysis and the reduction of the secondary cracking reactions of the generated oil vapors were considered as the main reasons. The oil obtained by the treated oil shale had a higher H/C ratio, indicating it had high energy content. The analysis results of chemical compositions in oils showed that the relative content of aliphatic hydrocarbons significantly increased after hydrothermal pretreatment. The further analysis demonstrated that the increase in the pretreatment time caused the generated long chain hydrocarbons tended to be directly released from oil shale particles, and were condensed into the oil.

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1. Introduction

The rapid development of the world needs to consume vast amounts of energy. At present, the conventional fossil fuels are still main sources of energy. However, fossil fuel reserves are limited in the world. It is necessary to develop various alternative sources to meet future energy demands. Oil shale, as an important unconventional oil resource, has attracted increasing attentions across the world. It is a fine-graine sedimentary rock containing organic complexes of kerogen throughout its mineral matrix [1], which can be converted into shale oil and other products through various chemical processes. The shale oil is considered to be an ideal alternative fuel for crude oil since the H/C atomic ratio of shale oil is similar with that of petroleum [2].

Pyrolysis is a prevalent method to utilize oil shale resource. Lots of treatment processes and methods for oil shale pyrolysis have been carried out in order to enhance the oil yield and improve its quality. For example, Gerasimov et al. [3] studied the effect of solid heat carrier on the yield of shale oil, and found that the use of solid heat carrier contributed to the increase of oil yield. El harfi et al. [4] pointed out that the oil produced by the pyrolysis of oil shale under microwave irradiation contained less sulfur and nitrogen than that obtained by conventional pyrolysis. The work of Shi et al. [5] focused on co-pyrolysis behavior of oil shale and its ash, and indicated that the addition of oil shale ash promoted the formation of aromatics in the oil. The investigation on co-pyrolysis of oil shale and high density polyethylene was carried out in the work of Aboulkas et al. [6].

Pretreatment before pyrolysis is helpful for promoting the conversion of fossil fuels into liquid products during the pyrolysis. The commonly used method for treating oil shale is acid treatment. Guo et al. [7]investigated the influence of different acids pretreatment (HCl, HNO_3 and HF) on self-heating retorting of oil shale, and showed that after treating with HF, the self-heating temperature was improved and the oil yield was increased. Ballice [8] also noted that the removal of the material soluble in HF increased the conversion in pyrolysis reactions. Furthermore, the thermal behaviors of oil shale treated by different acids were reported in the work of Aboulkas et al. [9]. Although acid treatment can produce some positive effects for the conversion of oil shale in the pyrolysis process, this method is hard to be widely used in oil shale industry due to the strong corrosive of hydrofluoric acid. Currently, hydrothermal pretreatment has paid more and more attentions by researchers.

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Water could act as a catalyst or reactant in the pretreatment process of solid fuels [10]. Some water soluble inorganics and hydrophilic organics containing oxygen functional groups can be leached out during the hydrothermal treatment [11]. So, hydrothermal treatment increases the hydro-liquefaction activity of fossil fuel in the liquefaction process [12]and improves liquid yield [13].

So far, some investigations on the effect of hydrothermal pretreatment on coal pyrolysis have been reported in the literatures [11,12,14]. However, little work focuses on the effect of hydrothermal treatment on oil shale pyrolysis, and the yield and characteristics of shale oil are rare mentioned in the literature. In this work, the hydrothermal pretreatment of oil shale was performed in the autoclave, and the pyrolysis of the treated oil shale was carried out by using the pyrolysis reactor. The aim of our work is to study the effect of different pretreatment time on the product distribution and characteristics of shale oil.

2. Material and methods

2.1. Raw material

Huadian oil shale (OS) from Jilin Province China was used as raw oil shale in this work. It was ground to 0.9–3.0 mm and stored in the wide mouthed bottle at room temperature. The proximate analyses of the Huadian oil shale were respectively listed below: 5.27 wt.% moisture, 37.72 wt.% volatiles, 3.22 wt.% fixed carbon and 53.79 wt.% ash. The ultimate analyses of oil shale were shown in Table 1. The oil shale was dried at 70 °C for 12 h before the further treatment.

2.2. Hydrothermal pretreatment

Hydrothermal pretreatment of oil shale was performed in a 100 mL autoclave. The autoclave was heated by using an oven. In each experiment, 20 g of oil shale and 60 mL of deionized water were placed into the autoclave. After the temperature of the oven reached to 200 °C, the autoclave was placed into the oven. The pretreatment time was 1.0, 1.5, 2.0 and 2.5 h, respectively. After the autoclave was cooled to room temperature, the treated oil shale was filtered and dried at 70 °C for 12 h. The obtained oil shale samples were numbered as OS-1.0, OS-1.5, OS-2.0 and OS-2.5 according to the pretreatment time. The untreated oil shale was labeled as OS. The yield of the treated oil shale was calculated by the formula (1).

$$\mathrm{Y}_t = \frac{M_t}{M_i} \times 100\% \tag{1}$$

where Y_t was the yield of the treated oil shale; M_t was the mass of the treated oil shale and M_i was the initial mass of oil shale.

2.3. Pyrolysis

The thermogravimetric (TG) experiments of the untreated/treated oil shale were performed by the HTC-2 model Beijing Henven thermogravimetry analyzer. About 20 mg of oil shale (<0.9 mm in size) was evenly placed in the crucible and heated from ambient temperature to 600 °C at a heating rate of 10 °C/min. The corresponding differential thermogravimetric (DTG) data was obtained by taking its derivative.

The pyrolysis experiment of oil shale was carried out in a tube reactor. The reactor was made of a stainless steel tube (the internal diameter of 67 mm and the overall length of 120 mm). The liquid collection device was connected with the one side of the reactor. About 10 g of oil shale was evenly placed in the reactor, and heated from ambient temperature to 520 °C kept for 30 min. The pyrolysis temperature was accurately controlled by using a temperature controller. The generated oil vapors and other gaseous products entered into the liquid collector which was immerged in an ice–water bath unit. The generated water and shale oil were separated by the water-toluene distillation method [15]. After the reactor was cooled, solid residues were collected and weighted as the mass of shale semi-coke. The yields of shale oil, water, shale semi-coke and gas were calculated by the following formulas (2)–(5).

$$Y_o = Y_t \cdot \frac{M_l - V_w\rho_w}{M_p} \tag{2}$$

$$Y_w = Y_t \cdot \frac{V_w\rho_w}{M_p} \tag{3}$$

$$Y_s = Y_t \cdot \frac{M_s}{M_p} \tag{4}$$

$$Y_g = Y_t \cdot \frac{M_p - M_s - M_l}{M_p} \tag{5}$$

where Y_o, Y_w, Y_s and Y_g were yields of shale oil, water, shale semi-coke and gas, respectively. M_l and M_s were the mass of liquids and solids; M_p was the mass of oil shale for pyrolysis; V_w was the volume of water and ρ_w was the density of water at 298 K and 0.1 MPa. The same experiment was performed more than twice, and the average yield of product was defined as the final experimental result.

2.4. Analysis methods

The carbon (C), hydrogen (H) and nitrogen (N) contents of oil were analyzed by using a Vario EL Cube Elementar. The oxygen (O) content was calculated by difference. The functional group types in the oil were analyzed by using a SHIMDZU model Fourier transform infrared spectrometer (FTIR). The mixture of the dried KBr powder and shale oil was ground, and then pressed into a pel-

Table 1
The elemental analysis results of raw oil shale and the treated ones.

Sample	C[a]	H	N	O[b]	H/C Ratio	O/C Ratio
OS[c]	30.23(0.07)[d]	4.37(0.23)	0.52(0.04)	64.88	1.73	1.61
OS-1.0	30.96(0.42)	4.32(0.22)	0.63(0.01)	64.09	1.67	1.55
OS-1.5	32.01(0.32)	4.35(0.25)	0.92(0.18)	62.72	1.63	1.47
OS-2.0	32.70(0.18)	3.97(0.08)	0.67(0.03)	62.66	1.46	1.44
OS-2.5	33.12(0.05)	4.21(0.59)	0.89(0.10)	61.78	1.53	1.40

[a] Dry basis, %.
[b] Calculated by difference.
[c] Obtained from the early work.
[d] The standard deviation.

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