



Full Length Article

Characteristics of bio-oil produced by the pyrolysis of mixed oil shale semi-coke and spent mushroom substrate



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HIGHLIGHTS

- The pyrolysis of mixed shale semi-coke and spent mushroom substrate is studied.
- The existence of shale semi-coke influences yield and characteristics of bio-oil.
- The shale semi-coke mass fraction of 50% is recommended in this work.
- The amount of aliphatic compounds in the obtained bio-oil is increased.

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ABSTRACT

In this work, spent mushroom substrate (SMS) was co-pyrolyzed with oil shale semi-coke (OSS) to investigate the effects of the OSS on yield and chemical compositions of bio-oil product. Oil shale semi-coke was mixed with biomass in the OSS/SMS mass ratios of 0:1, 1:4, 1:1, 4:1 and 1:0. The pyrolysis experiment was carried out by using a lab-scale pyrolysis reactor. The experimental results showed that the addition of the OSS led to an increase of bio-oil yield. The maximum of bio-oil yield was obtained at the OSS/SMS mass ratio of 1:1. The characteristics of the obtained oils were studied by elemental analyzer, FTIR and GC-MS. It was found that the bio-oil obtained from the pyrolysis of mixed SMS and OSS had higher carbon and hydrogen content, lower oxygen content than those produced by the pyrolysis of biomass alone. Moreover, there were more aromatics and aliphatic compounds in the obtained bio-oil product. These variations indicated that the existence of the OSS significantly influenced the chemical compositions of bio-oil.

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1. Introduction

Oil shale, as an important potential energy source, arouses the attention of the world. In China, oil shale reserves are about 720 billion tons [1], mainly distributed in northeast regions such as Huadian Basin, Fushun Basin, and Songliao Basin [2]. Through thermochemical conversion, oil shale is decomposed to generate oil, gas and shale semi-coke. Currently, liquid and gas products have been widely used in many fields. The utilization of these pyrolysis products is beneficial to alleviate the depletion of fossil fuels and promote the development of society. However, it is worth noting that a large number of shale semi-coke are produced after the retorting of oil shale. The unreasonable treatment of these solid residues not only has some negative impacts on the environment, but also hinders the development of oil shale industry [3]. At

present, Oil shale semi-coke is used as the absorbent [4] or the building material [5]. Some researchers also investigate the influence of its addition on the combustion of fossil fuels or biomasses. For example, Wang et al. [6] investigated the co-combustion behavior of oil shale and its shale semi-coke, and found that the addition of shale semi-coke decreased ignition temperature of oil shale. The similar results were also reported in the work of Sun et al. [7]. In their work, shale semi-coke was mixed with bituminous coal. Kuusik et al. [8] discussed the combustion characteristic of shale semi-coke by using a circulated fluidized. Moreover, Liu et al. [9] found that there were some interactions in the co-combustion process of torrefied cornstalk and shale semi-coke. Wang et al. [3] pointed out that the existence of shale semi-coke hindered the combustion of fixed carbon in the cornstalk.

Biomass also gains a growing interest for researchers, because it is rich, inexpensive and renewable [10]. There are various biomass resources in the world, such as woods, microalgae and

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agricultural residues. So, it is considered to be one of the most sustainable carbon sources [11]. As an agriculture country, China has a large number of agricultural residues. Among these solid wastes, spent mushroom substrate is an organic solid residue remaining after harvest of mushrooms [12]. The total yield of spent mushroom substrate is over 13 million tons [13]. However, there is no effective treatment method at present. The produced spent mushroom substrate usually is incinerated or dumped in the field directly, resulting in serious pollution of environment. Pyrolysis is considered to be an appropriate method to convert biomass into high value-added products such as bio-oil and gas.

So far, the study on pyrolysis behavior of oil shale semi-coke is poor. In particular, the influences of shale semi-coke on bio-oil yield and its characteristic are rarely reported in the literature. In this paper, the effects of oil shale semi-coke on yield and characteristics of bio-oil produced by spent mushroom substrate were studied. The phase composition of shale semi-coke was analyzed by the X-ray diffraction, and the energy-dispersive X-ray spectroscopy was used to analyze the contents of metal elements in shale semi-coke. The thermal behavior of shale semi-coke was studied by using a thermogravimetric analyzer. The influence of the mass fraction of shale semi-coke in the mixture on bio-oil yield was investigated by using a lab-scale retorting reactor. The characteristics of the obtained bio-oils were analyzed by the elemental analysis, FTIR and GC–MS. The purpose of our work is to provide foundation study for the effective utilization of oil shale semi-coke and spent mushroom substrate in the thermochemical conversion.

2. Materials and methods

2.1. Raw material

The used spent mushroom substrate in this paper was obtained from Jilin province, China. Oil shale semi-coke was obtained after the retorting of Huadian oil shale at 540 °C. The particle size of raw materials was below 0.9 mm. After biomass was mixed thoroughly with shale semi-coke, the mixed samples were stored in the wide-mouth bottles. The samples were labeled as S₁, S₂, S₃, S₄ and S₅ with the SMS/OSS mass ratios of 1:0, 4:1, 1:1, 1:4 and 0:1 (the OSS mass fraction of 0%, 20%, 50%, 80% and 100%), respectively. The basic characteristics of the SMS and OSS were listed in Table 1.

2.2. Experimental apparatus and procedure

The thermal behavior of the OSS was studied by using thermogravimetric analyzer (HTG-2 model, Henven, Beijing). About 10 mg sample was evenly distributed in the crucible, and then heated from the room temperature to 750 °C at the heating rate of 10 °C/min. The nitrogen was used to ensure the hypoxia environment during pyrolysis. The flow rate of inert gas was kept at 50 ml/min. The differential thermogravimetric (DTG) data of sample was calculated by taking the derivative of thermogravimetric (TG) data.

The pyrolysis experiment of the mixed sample was carried out by a lab-scale reactor. The schematic diagram was shown in Fig. 1. The experimental apparatus consisted of three sections: the feeding section, the pyrolysis section and the collection section. Through the rotation of the spiral feeder, the solid sample was sent to the pyrolysis section. The pyrolysis section was composed of two stainless steel tubes. The total length of tubes was 170 mm, and inner diameter was 20 mm. The pyrolysis temperature was kept by using a temperature controller. The gaseous products generated in the pyrolysis process were cooled through a vertical orientation counter-current condenser tube, and condensed in the liquid collection device. The produced solid residues were collected in the solid collection device. In this work, the final pyrolysis temperature was 470 °C, and the feeding rate of the sample was 0.52 g/min. The nitrogen flow rate was 200 ml/min. The obtained liquid products were weighted and recorded as M₁. The mass of water was determined by using the water-toluene distillation method [15], and recorded as M₂. The weight of bio-oil (M₃) was calculated by the formula (1), and the yields of pyrolysis products were obtained by the formula (2). The gas yield was calculated by difference.

$$M_3 = M_1 - M_2 \quad (1)$$

$$Y_i = \frac{M_i}{M} \times 100\% \quad (2)$$

where M was the mass of biomass, M_i and Y_i were the mass and yield of product i (bio-oil, water and solid residues).

2.3. Analysis methods

The contents of carbon (C), hydrogen (H) and nitrogen (N) elements of oils were tested by a Vario EL Cube Elemental analyzer. The oxygen (O) content was calculated by difference. An energy-dispersive X-ray spectroscopy (EDS) was used to analyze the contents of metal elements in the OSS. The X-ray diffraction (XRD) spectrum of the OSS was obtained by using an Ultima IV X-ray diffractometer equipped with a fixed monochromator and a Cu tube (Cu K α radiation), operating at 40 kV and 30 mA with the scan range from 10° to 70° at 0.02° intervals. The Fourier transform infrared spectra (FTIR) of bio-oils were recorded in the scan range of 400–4000 cm⁻¹ by using the SHIMDZU 1.50SU1 FTIR spectrometer. The Agilent 7890 gas chromatograph equipped with the Agilent 5975 N mass spectrometer (GC–MS) was used to detect chemical compositions of oils, which was fitted with a 30 m × 0.32 mm capillary column coated with a 0.25 μm thick film of 5% phenyl-methylpolysiloxane (HP-5MS). The oven temperature was initially held at 50 °C for 3 min, and then programmed from 50 °C to 280 °C at 5 °C/min and held 10 min. The flow rate of Helium was 1 ml/min. The split ratio was 1:50. The analysis of chemical compounds in the oil products was performed by National Institute of Standards and Technology mass spectrum library search (version 2.0). The schematic diagram of the whole experiment process was shown in Fig. 2.

Table 1
Proximate and ultimate analysis of the SMS and OSS.

Sample	Proximate analysis (air-dry basis, wt%)				Ultimate analysis(dry basis, %)			
	Moisture	Volatiles	Fixed carbon	Ash	C	H	N	O ^b
SMS ^a	10.7	72.1	8.1	9.1	40.96	4.93	1.15	52.96
OSS	0.95	29.62	1.44	67.99	18.29	1.52	0.48	79.71

^a Obtained from our early work [14].

^b Calculated by difference.

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