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Effect of ionic liquid 1-butyl-3-methyl-imidazolium dihydrogen phosphate pretreatment on pyrolysis of Shengli lignite

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ABSTRACT

The pyrolysis behaviors of Shengli lignite (SL) pretreated with ionic liquid 1-butyl-3-methyl-imidazolium dihydrogen phosphate ([Bmim]H_2PO_4) was studied in this paper. The effect of pretreatment temperature and the ratio of [Bmim]H_2PO_4 to SL on the pyrolysis behaviors of lignite were mainly investigated. Also the mechanism of the influence of [Bmim]H_2PO_4 pretreatment on the pyrolysis of lignite was investigated by FTIR analysis. It is found that [Bmim]H_2PO_4 pretreatment results in the drastical increase of the yield of tar, especially the yield of oil fraction. The yield of tars obtained from pyrolysis of SL treated in [Bmim]H_2PO_4 at 200 °C with the ratio of [Bmim]H_2PO_4 to SL at 1 increases by 1.7 times compared to that of SL. [Bmim]H_2PO_4 pretreatment results in the increase of yield of oil fraction, especially the yield of arenes and phenolics. The FT-IR analysis shows that [Bmim]H_2PO_4 pretreatment results in the increase of the content of carbonyl group and ether bond in SL.

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1. Introduction

Pyrolysis is considered as a simple method for direct production of low-carbon fuel and high value chemicals from low rank coal resources. However, the existing lignite pyrolysis has common problems such as low tar yield and poor tar quality due to high solid and pitch contents in tar, which is not suitable for industrial applications and continuous operation of the pyrolysis processes.

It is well accepted that pyrolysis of coal starts with the generation of volatile radical fragments derived from thermal cleavage of covalent bonds in coals. Also many studies showed that the cross linking at the low temperatures influence the pyrolysis at the temperatures between 400 °C and 500 °C, leading to the decrease of tar yield [1]. The above studies suggest that thermal cleavage of covalent bond and noncovalent bonds affects the yield and quality of tar derived from lignite pyrolysis.

Large number of studies found that pretreatment of coal can destroy weak bonding structure in coal and it's an effective method to improve tar yields and regulate the composition and distribution of pyrolysis products [2–5]. During the study of the dissolution of coal in ionic liquids, Lei et al. found that ionic liquids can efficiently destroy the hydrogen bonding in low rank coal [6–8]. Based on the high depolymerization performance of ionic liquids, Lei et al. found that the pretreatment of lignin by ionic liquid 1-butyl-3-methyl-imidazolium chloride is effective to increase the total yield of liquid [9]. At the same time, he found that ionic liquids pretreatment can significantly improve the tar yield and change the distribution of pyrolysis products [10]. Also Nawshad et al. [11] found that pretreatment biomass in the ionic liquid 1-butyl-3-methyl-imidazolium chloride can significantly increase the bio-oil yield. Lei et al. [8] has studied the effect of ionic liquids type on the extraction of Xiangfeng lignite and found that ionic liquids type, especially the anion of ionic liquids, significantly affects the dissolution of lignite. The performance of imidazolium-based ionic liquids towards the dissolution of lignite follows the order as $Cl^- > H_2PO_4^- > Br^- = OH^- > BF_4^- > BrO_3^- > PF_6^-$. Since the type of ionic liquids significantly affect the thermal dissolution of lignite, different types of ionic liquid pretreatment is bound to change lignite pyrolysis behavior in varying degrees. 1-butyl-3-methyl-imidazolium dihydrogen phosphate ([Bmim]H_2PO_4) as an acidic ionic liquid has been reported to be used in oil to extract nitrogen-containing compound [12]. To the best of our knowledge, however, the information of [Bmim]H_2PO_4 pretreatment on lignite pyrolysis is scarce in the literature.

In this paper, the effect of [Bmim]H_2PO_4 pretreatment on the pyrolysis of lignite is studied. The emphasis is to study the effect of pretreatment temperature and the ratio of [Bmim]H_2PO_4 to lignite on the pyrolysis of lignite, especially on the yield of liquid and oil fraction. Also the [Bmim]H_2PO_4-treated lignite is characterized by FTIR to

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investigate the mechanism of the increase of total volatile matter and liquid yield through the ionic liquid treatment.

2. Experimental

2.1. Properties of samples

Shengli lignite (SL) sample was used in this study. It was ground to pass through a 200 mesh, stored under nitrogen atmosphere, and dried under vacuum at 80 °C overnight before use. The proximate and ultimate analyses of SL are showed in Table 1. The ionic liquid used in this study was [Bmim]H_2PO_4 purchased from Shanghai Cheng Jie Chemical Co. LTD. and dried at 100 °C in a vacuum oven for 48 h prior to the experiments.

2.2. [Bmim] H_2PO_4 treatment of SL

The pretreatment of SL was carried out in a microwave apparatus with a thermometer, timer, and stirrer. In a typical example, SL (5.0 g) and a certain amount [Bmim] H_2PO_4 were charged into a Teflon tube to be treated at 150 °C, 200 °C and 250 °C for 60 min, respectively. After [Bmim]H_2PO_4 treatment, the SL/[Bmim] H_2PO_4 slurry was cooled down to room temperature. Then the dissolved lignite was precipitated by pouring the lignite/[Bmim]H_2PO_4 slurry into a beaker containing water. The precipitate formed was filtered through filtration using membrane filter paper with a pore size of 0.45 μm. Finally the [Bmim]H_2PO_4-treated SL was dried in vacuum at 353 K overnight. These samples treated at 150 °C, 200 °C, and 250 °C were abbreviated to SL_{150}, SL_{200}, and SL_{250}, respectively. The samples pretreated at 200 °C with ratio of [Bmim]H_2PO_4 to SL at 0.5, 1, and 2 were abbreviated to $SL_{0.5}$, SL_1, and SL_2, respectively.

2.3. Pyrolysis of SL samples

The pyrolysis of SL was performed in a vertical fixed-bed quartz reactor ($\Phi = 25 \times 500$ mm) coupled with a U-shaped quartz insert (U-tube, in short). The lower portion of the U-tube was placed in an ethanol–water bath to reach a temperature of -10 to -5 °C to condense the liquid products. The experimental apparatus and the procedure were described in detail in Ref. [13]. In each run, 3 g of SL with or without [Bmim]H_2PO_4 pretreatment was loaded into the reactor and heated in an N_2 flow of 100 ml/min. The temperature-program was from room temperature to 110 °C at a rate of 10 °C/min and 45 min at 110 °C for removal of moisture, and then from 110 °C to 800 °C at a rate of 10 °C/min. The tar was collected at temperatures of 300, 400, 500, 600 and 800 °C by quickly replacing the U-tube, respectively. Five tar samples obtained in such a way represent tars generated in the temperature ranges of 110–300 °C, 300–400 °C, 400–500 °C, 500–600 °C, and 600–800 °C in sequence. The weight of the tar was measured by weight difference in U-tube, with and without the tar. The amount of residual ionic liquid is estimated by the amount of nitrogen of ionic liquid pretreated coal. In order to eliminate the influence of pyrolysis of residual ionic liquid on the calculation of tar yield, the liquid product yields resulted from pyrolysis of residual liquid ionic in five stages were also studied. Therefore the tar yield of the pretreated coal (without residual ionic liquids) is obtained by the difference between the tar yield resulted from ionic liquid treated coal (with residual ionic liquids) and that of residual ionic liquid. The U-tubes with the tar sample were then filled with n-hexane and settled for 2 h. All of the hexane solution was then poured into a volumetric flask and supplemented with hexane to 50 mL.

Table 1
Proximate and ultimate analyses (wt.%) of SL.

Proximate analysis			Ultimate analysis (daf)				$S_{t,d}$
A_d	M_{ad}	V_{daf}	C	H	O[a]	N	
15.1	15.1	33.3	68.25	5.23	>24.34	1.12	1.05

daf: dry and ash-free base; M_{ad}: moisture (air dried base); A_d: ash (dry base, i.e., moisture-free base); V_{daf}: volatile matter (dry and ash-free base); $S_{t,d}$: total sulfur (dry base).

[a] By difference.

2.4. Analysis of samples

FTIR spectra of the SL and [Bmim]H_2PO_4 treated SL were measured on a Nicolet 6700 spectrometer (USA Thermo Scientific Co.) using the KBr-pellet technique.

Hexane-soluble portions (HS) were separated by column chromatograph with n-hexane, n-hexane/dichloromethane (1:2.5 v:v), and dichloromethane/tetrahydrofuran (1:3 v:v) in turn into alkanes, arenes, and phenolics, respectively. Alkanes, arenes and phenolics were analyzed by gas chromatograph (GC-6820, TechCom) equipped with a SE-54 column (30 × 0.25 × 0.33 μm) and a flame ionization detector. Alkanes were analyzed by gas chromatograph (GC-6820, TechCom) equipped with an OV-1 column (30 × 0.25 × 0.33 μm) and a flame ionization detector. The separated components were determined by GC/MS (Thermo Scientific TRACEGCULTRA-ISQ) analysis. The initial oven temperature was set at 50 °C and then increased to 240 °C at a rate of 3 °C/min and held for 10 min. The compounds were identified through GC/MS analysis by comparing mass spectra with NIST08 and NIST08s library data.

3. Results and discussion

3.1. Effect of [Bmim]H_2PO_4 pretreatment on the tar yield of SL pyrolysis

As shown in Fig. 1, the yield of liquid obtained from pyrolysis of SL at temperature ranges of 100–300, 300–400, 400–500, 500–600, and 600–800 °C is 17 mg/g_{coal}, 12 mg/g_{coal}, 45 mg/g_{coal}, 25 mg/g_{coal}, and 0.7 mg/g_{coal}, respectively. After the dissolution of SL at 150 °C, it can be found that the yield of liquid obtained from pyrolysis of SL_{150} at temperature ranges of 300–400, 400–500, and 600–800 °C increased by 2.8, 1.5, and 14.8 times, respectively, compared to that of SL. The total yield of liquid obtained from pyrolysis of SL_{150} is 1.4 times larger than that of SL. With the increase of dissolution temperature, the yields of liquid obtained from pyrolysis of SL_{200} at temperatures ranges of 300–800 °C increase compared to that of SL. The total yield obtained from pyrolysis of SL_{200} reaches 125 mg/g_{coal}, which is 1.3 times larger than that of SL. However, with the dissolution temperature increase to 250 °C,

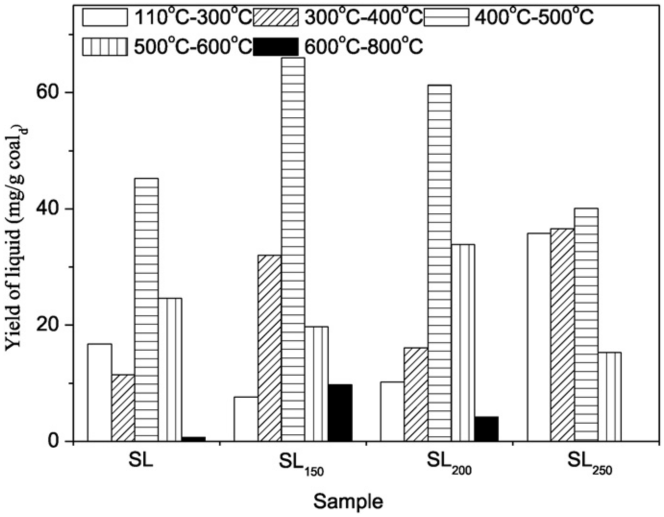

Fig. 1. Yields of liquid produced during different stages of coal pyrolysis for SL and SL treated under different temperature. Pretreatment conditions: 60 min and ratio of SL to [Bmim]H_2PO_4: 1:1.

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