



Contents lists available at ScienceDirect

Journal of Analytical and Applied Pyrolysis

journal homepage: www.elsevier.com/locate/jaap

Co-carbonization behaviors of petroleum pitch/waste SBS: Influence on morphology and structure of resultant cokes

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ARTICLE INFO

Keywords:

Styrene-butadiene-styrene copolymer

Mesophase pitch

Co-carbonization

Porous carbon

ABSTRACT

Petroleum pitch was modified by waste styrene-butadiene-styrene copolymer (SBS) and the cokes were obtained via the co-carbonization method. The waste SBS content and the co-carbonization temperature have important effects on the morphology and structure of resultant cokes. It can be found that adding waste SBS can obviously increase the methylene content in the cokes and improve the thermal stability of the co-carbonized products. The coke of MP7.5-450-10 exhibits almost 100 vol% anisotropy region with streamline texture and has the lowest weight loss of 5 wt%. The coke of MP10-450-10-C has the smallest carbon interlayer space and the largest crystallite thickness. Co-carbonized products at 900 °C have the porous structure similar with carbon foam, and the large pore diameter is in the range of 200–1000 μm. Importantly, there are some mesopores existing in the products. Moreover, adding waste SBS can increase the specific surface area and the pore volume of carbonized products. The samples of MP7.5-450-10-C has some mesopores with the diameter of 3–50 nm, and the pore volume is 0.013 cm³ g⁻¹.

1. Introduction

Due to its high coke yield, low softening point and high fluidity, mesophase pitch is regarded as the superior precursor for preparing many carbon materials [1,2]. The mesophase products (such as needle coke, mesocarbon microbeads, carbon fibers and carbon foam) have different structures and properties, which can be applied in C/C composites, electrode materials, catalyst carriers and so on [3–9]. Thus, the mesophase pitch-based carbon materials have attracted the interests of many researchers. The methods for preparing mesophase pitch include thermal polymerization [2], catalytic polymerization [10], hydrogenation treatment [11] and co-carbonization method [12]. It is believed that mesophase spheres can be generated via the polymerization during the liquid phase carbonization of aromatic hydrocarbons, then grow and coalesce continuously to form the bulk mesophase pitch [13,14].

Nowadays, coal tar pitch, petroleum pitch and many other materials containing aromatic compounds are the main feedstock for mesophase pitch [15,16]. The precursor pitch can be modified with polypropylene,

polyethylene, waste plastic, etc., and then the mesophase pitch can be prepared via the co-carbonization [17,18]. In previous researches, styrene-butadiene-styrene copolymer (SBS) as the modifier can improve the rheological and physical properties of precursor pitch [19,20]. More importantly, the morphology and structure of resultant cokes can be modified by the waste polymer via the co-carbonization. Cheng et al. reported that the needle coke could be derived from the co-carbonization of ethylene tar pitch with polystyrene (PS) [21]. Our group have reported mesophase pitch can be obtained by the co-carbonization of petroleum pitch/waste polyethylene (WPE) and petroleum pitch/graphene oxide (GO) [22,23]. Furthermore, the properties of mesophase pitch can be improved greatly due to the increasing content of naphthenic and alkyl groups generated by WPE. However, there are no porosities in resultant cokes.

At present, porous carbon materials have become a hot issue due to its excellent properties and the preparation method mainly includes the carbonization, activation, templating and sol-gel techniques [24,25]. We can infer that porous carbon materials can be derived from some non-porous materials by using appropriate approaches. Lee et al. [26]

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<https://doi.org/10.1016/j.jaap.2017.11.017>

Received 7 September 2017; Received in revised form 19 November 2017; Accepted 20 November 2017

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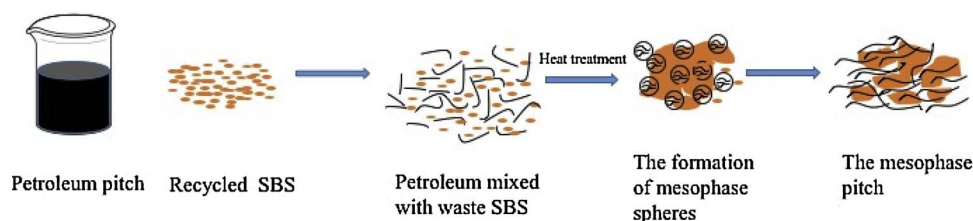


Fig. 1. The illustration of the formation of mesophase pitch derived from petroleum pitch/waste SBS.

reported the porous carbon films have been prepared successfully by using the carbonization method. Singh et al. [27] reported the synthesis of N-doped porous reduced graphene by using carbonization method. Zhang et al. [28] prepared porous carbon materials from rice husk through direct carbonization method. Therefore, these researches give us the inspiration that the porous structure may be produced as the result of the pyrolysis of SBS during the co-carbonization.

In this paper, the petroleum pitch with different contents of waste SBS was heat-treated to obtain mesophase pitch under different conditions. Then, porous structure can be produced in resultant cokes via further co-carbonization. Furthermore, the reuse of waste SBS could decrease the costing of porous carbon materials. The morphology and structure of as-prepared samples were characterized by polarized microscopy, Fourier transform infrared spectroscopy (FT-IR), thermogravimetric analysis (TGA), scanning electron microscopy (SEM), X-Ray diffraction (XRD) and N_2 adsorption.

2. Experimental

2.1. Materials

The 90 A# petroleum was purchased from Xi'an Petrochemical Controlled Company. The recycled waste SBS was obtained from waste rubber.

2.2. Co-carbonization of petroleum pitch/waste SBS

Firstly, waste SBS modified petroleum pitch was prepared by conventional melt blending method [17,29,30]. The typical process was as follows: 500 g petroleum pitch was heated to the fluid state, and then mixed with different contents of waste SBS (0 wt%, 5 wt%, 7.5 wt%, 10 wt%, 12.5 wt% and 15 wt%, respectively) by shearing at a speed of 3750 rpm. The modified temperature kept at 120 °C for shearing 90 min. Then the waste SBS modified petroleum pitch was obtained.

Secondly, about 200 g modified pitch was put into the steel reactor vessel and then treated at the setting temperature for a certain time under N_2 atmosphere. During the heat-treatment, the heating rate of the

system was 2 °C/min and the stirring speed was 1000 rpm. When the reaction system was naturally cooled to the room temperature, the sample of mesophase pitch via the co-carbonization was obtained. The illustration of the formation of mesophase pitch derived from petroleum pitch/waste SBS are shown in Fig. 1.

Finally, as-prepared mesophase pitch was carbonized at 900 °C for 2 h in the tube furnace. The heating temperature was increased from room temperature to 900 °C at the rate of 10 °C/min. After the furnace was naturally cooled to the room temperature, the final product was obtained.

In this experiment, the names of as-prepared all samples under different conditions are shown in Table 1.

2.3. Characterization

The optical anisotropy of mesophase pitch was observed by using the polarized microscopy (Olympus bx51). The samples were placed in round paper moulds, then adding dental base acrylic resin powder to cover the samples and injecting with methyl methacrylate for curing. After grinding and buffing, the samples were observed under the polarized microscopy.

FTIR was performed on a FTIR spectrometer (EQUINOX-55) using KBr wafers of samples. The samples with KBr were pressed to form the wafers (the mass of KBr and as-prepared sample was about 200 mg and 2 mg, respectively).

The morphologies of carbonized products were investigated by scanning electron microscope (SEM Hitachi SU8000).

Thermogravimetric analysis of the samples was performed on a thermogravimetric analyzer (TF-209 F3) in the range of 40–900 °C at the heating rate of 10 °C/min under N_2 flow.

X-Ray diffractions were tested on an X-Ray diffractometer (XRD SHIMADZU LIMITED XRD-700) with a $Cu K\alpha$ irradiation source ($\lambda = 1.54 \text{ \AA}$) within the range of 10–90° at the scan rate of 8°/min.

The N_2 adsorption experiments were performed on a N_2 adsorption instrument (PM2-0648) at 77 K. The specific surface area was calculated by the Brunauer-Emmett-Teller (BET) equation, and the pore distribution was calculated by the Barret-Joyner-Halenda (BJH) method.

Table 1

Names of all samples under different experimental conditions.

Samples	Waste SBS content (wt%)	Heating temperature (°C)	Holding time (h)	Carbonization temperature(°C)	Holding time (h)
MP0-450-10	0	450	10	/	/
MP5-450-10	5	450	10	/	/
MP7.5-450-10	7.5	450	10	/	/
MP10-450-10	10	450	10	/	/
MP12.5-450-10	12.5	450	10	/	/
MP15-450-10	15	450	10	/	/
MP7.5-400-10	7.5	400	10	/	/
MP7.5-450-8	7.5	450	8	/	/
MP0-450-10-C	0	450	10	900	2
MP5-450-10-C	5	450	10	900	2
MP7.5-450-10-C	7.5	450	10	900	2
MP10-450-10-C	10	450	10	900	2
MP12.5-450-10-C	12.5	450	10	900	2
MP15-450-10-C	15	450	10	900	2

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