CARBON 2019
LEXINGTON, KY

OBTAINING THE COMPOSITIONAL FIBERS BASED ON COAL TAR MESOPHASE PITCH USING ELECTROSPINNING METHOD

Zulkhair Mansurov[1,2], Gaukhar Smagulova[1,2], Bayan Kaidar[1,2],
Aldan Imangazy[2], Meruyet Artykbayeva[1,2], Renata Nemkayeva[2]

[1]*Institute of Combustion Problems, Almaty, Kazakhstan*
[2]*al-Farabi Kazakh National University, Almaty, Kazakhstan*
*Presenting author's e-mail: zmansurov@kaznu.kz

Introduction

Among a wide class of carbon-based materials, carbon fibers occupy a special position due to the uniqueness of their physicochemical properties and prospects for practical application: in construction, industry, aerospace, etc. Carbon fibers are characterized by high-tension force, low specific weight, low thermal expansion coefficient and chemical stability [1, 2]. The process of electrospinning is a complex physico-chemical process that depends on many factors and, largely, on the choice of the precursor and its preparation [3,4]. Polymers are the most common fiber-forming materials. However, the main goal of our work was to obtain fibers from alternative precursors, such as coal tar using electrospinning method.

Coal tar is a complex mixture of aromatic hydrocarbons, heterocyclic sulfur-, oxygen- and nitrogen-containing compounds. By further coal tar processing, such valuable products are obtained: benzene, toluene, xylenes, etc., as well as sleeper impregnation oil, plastics, pitched-based electrodes, carbon fibers, binding pitch, etc. [5].

There are two main types of pitch: isotropic (usual non-mesophase) and anisotropic (mesophase). Mesophase pitch is obtained by heat treatment, as a result of which chemical reactions occur with a change in structural characteristics - the formation of mesophases. Heat treatment was carried out in a wide range of temperatures in inert nitrogen atmosphere, argon or helium. Formation of liquid crystal structures (mesophase) occurs in the temperature range of 300-500^{o}C. Mesophase crystallites are composed of condensed high molecular weight aromatic compounds with an interplanar distance of 0.344 nm. The mesophase transformations appearance depends on the physicochemical characteristics of the initial raw material and the temperature regime of the treatment. The transition of carbon pitch into the mesophase structure occurs through the formation of an intermediate isotropic-mesophase structure under the influence of temperature. The transition is accompanied by the removal of gaseous products and a change in the H/C ratio.

Materials and Methods

Mesophase pitches were obtained by coal tar heat treatment in an argon atmosphere in the temperature range from 200 to 500°C. Argon consumption was 92 cm^3/min.

The pre-dried and weighed porcelain boat was filled with the initial coal tar, then this boat was placed in a quartz reactor and blew off with argon to remove air from the reactor to avoid contact with oxygen. Argon was run for 5 minutes, and then the reactor was heated up in series to 200, 300, 350, 400, 500°C. The heating rate was 13°C/min. The thermal treatment time was 2 hours, after the heat treatment process, the heating of the reactor was stopped, the sample was cooled to room temperature without being removed from the reactor in argon atmosphere. After removal from the reactor, the boat with the final product was weighed to determine the mass loss.

Results and Discussion

To determine the effect of temperature treatment on the initial coal tar composition, an elemental analysis for coal tar-derived pitch was carried out. The coal tar-derived pitch samples, obtained at 200, 300 and 350°C, the sulfur content was 0.24-0.26 wt.%. The increasing of treatment temperature to 400°C resulted in the complete sulfur removal from the coal tar-derived pitch composition. Sulfur is contained in the coal tar in the form of sulfur-containing heterocyclic aromatic compounds.

Analysis of SEM images showed that for 200°C obtained coal tar pitch, the formation of a porous structure and active mesophase centers was observed. When the treatment temperature rises up to 300°C, an increase in the degree of surface degradation was observed. After thermal processing, the sample surface had a more relief structure, and the number of mesophase centers increases per unit area. At a treatment temperature of 350°C, a transition from an isotropic to anisotropic structure was observed. For this sample, all volatile fractions were removed. The sample surface was homogeneous, the size of the mesophase centers was about 2 microns. For a coal tar pitch sample obtained at 400°C, a similar anisotropic structure was observed, in some regions a layered structure, which was associated with an increase in the degree of the sample graphitization was observed. The size of the mesophase particles increased up to 3.5-5 microns. A treatment temperature of coal tar equal to 500°C leads to a complete transition from an isotropic to anisotropic structure.

Conclusions

Based on the conducted research, a correlation between the temperature of the heat treatment and the structure of the obtained coal tar-derived pitch was established. The results of optical microscopy and scanning electron microscopy have shown that while the heat treatment temperature increased, the number of mesophase centers per volume unit of the obtained pitch also increased. The diameters of the mesophase particles were 3-16 μm. The highest number of mesophase particles was observed for pitch obtained at 500°C. The results of energy-dispersion analysis showed that heat treatment at 400°C led to complete sulfur removal.

The electrospinning method does not show high requirements for the chemistry of the process, does not require high temperatures for fiber solidification, and therefore, allows the creation of fibers from long and complex molecules. It is a complex, profitable and simple method for nanofibers production.

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