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STUDY OF THE HEAT TREATMENT EFFECT OF MEDIUM-TEMPERATURE ELECTRODE PITCH ON CARBONISATE YIELDS

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Abstract. *Coal tar pitch is a residue of coal tar separation. It is widely used as a binder in the production of electrodes and anode mass. The production of pectic carbonisates and the development of methods to increase the value of its yield is of scientific and applied interest. The author conducted experiments on heat treatment in oxidising medium of medium-temperature electrode pitch of category BsT$_{melt}$ = 71.5 °C (AO Altai-Koks, Russia). We conducted the process of heat treatment of the pitch in a 5.6 litre reactor heated by using an integrated electric heating system. To enhance the increase of T$_{melt}$ during heat treatment, we pumped the gas phase products from the reactor to a collection tank where they condensed. We performed heat treatment at T > 400 °C using both thermal oxidation of pitch and pumping of distillates. We formed the oxidising environment by supplying air by a compressor to the molten pitch. The air supply process started after the holding temperature was reached. The temperature controller provided the holding temperature. The author determined the yield of thermo-oxidation products, melting temperatures T$_{melt,}$ and volatile yields X for the resulting products. We obtained pitch with T$_{melt}$ = 140 °C and 158 °C. However, this heat treatment significantly reduced the yield of volatile substances in the final products. The temperature of carbonisation of heat-treated pitch was T = 650 °C and T = 850 °C. Therefore, research determines the carbonisate yields. Moreover, carbonisation proceeds with the additional holding times at 450 °C and 650 °C. Hence, heat treatment increased the carbonisate yield by 10%. Holding times at 450 °C and 650 °C also quantitatively increased the yield of carbonisate.*

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Introduction

Coal tar pitch (CP) is a residue of coal tar separation into fractions: light fraction (T<170°C); phenolic fraction (T = 170-210 °C); naphthalene fraction (T = 210-230 °C); absorption fraction (T = 230-270 °C); anthracene fraction (270 °C-360 °C); coal tar pitch (T < 360 °C). Pitch is a multicomponent structure consisting of the following fractions: γ-fraction, soluble in hexane, isooctane; β-fraction, insoluble in hexane but soluble in toluene; α-fraction, insoluble in toluene, divided into quinoline-soluble α_2-fraction and quinoline-insoluble α_1-fraction [1].

The main products of pitch carbonisation are mesophase pitch (400-450 °C) [2-6]; pitch semi-coke (450-800 °C) [3, 7] and pitch coke (> 800 °C) [4, 6].

Mesophase pitches (MPs) have wide applications in the production of needle coke [4, 6] and carbon fibres [8-10]. MPs can also be used for production of:

- construction materials [11];
- foam carbon [12-13];
- electrode in a lithium-ion battery [14].

According to earlier studies, high-grade MP is obtained by low-temperature carbonisation (LTC) of both coal pitches [1-5] and synthetic pitches [10, 15], petroleum pitches [6, 11, 16] and oil-coal pitches [17].

Most foreign studies have conducted LTC by heating [2, 5, 10] or thermostatting of pitch or other hydrocarbon feedstock [3-6, 15-17] in the temperature range of 400-450 °C. It was found that the processes of mesophase formation in pitch stop at 540 °C [18].

According to [19], anisotropic semicokes were obtained by heating to 500 °C in argon atmosphere and holding at this temperature for 1 hour. The paper [20] describes the modification of coal pitch with polyethylene glycol with the addition of a catalyst in the form of p-toluene sulfonic acid, and the heat treatment (T = 800 °C) of this modification yielded pitch semi-coke. In [21], semi-coke was obtained by heat treatment at temperatures up to 470°C for 4 hours.

In [22] carbonisation of pitch and pitch with penographite additives at 800-900 °C results in materials. Their X-ray structural characteristics are close to those of graphite. In [23], the yield of carbonisate 64% was determined at carbonisation of electrode pitch of category T = 900 °C. Carbonisate yield of 60-64% was at heat treatment of pitch at T =900 °C. With holding time of 1 h each at 320 °C, 400 °C, 450 °C, 500 °C, and 600 °C, the carbonisate yield was 60-64%. The carbonisate yield was 50-53% at the same final temperature but with holding time of 1 h each at 320 °C and 3 h at 600 °C [24].

According to [25-26], carbonisation of pecks by heating at temperatures above 300 °C results in the growth of α_1-fraction in the pitch. Mesophase transformations occur at temperatures of 400-500 °C according to [2-3, 10]. Enhanced growth of α_1-fraction can occur in this temperature range according to the results of [10]. The processes of semi-coking and coking of pitch occur at temperatures above 500 °C [3, 4, 19]. Hence, it can be suggested that the α_1 fraction may play a major role in carbonisation of pitch and quantitatively increase its yield.

According to [27, 28], dehydrogenation polymerisation reactions followed by dehydrocyclisation predominate during carbonisation. These reactions result in cross-linking. It eventually causes to the formation of coke. This corresponds with the data obtained in [29] – pitch coke or carbonizate is the final product of polymerisation reactions at T>500°C.

The determination of the heat treatment effect of pitch in the temperature range (T = 400-500 °C) for the low temperature carbonisation on the carbonisate yield (T = 850 °C) is very interesting reaction. In this study we conducted an experiment to determine the effect of heat treatment of medium-temperature coal pitch of category B on the carbonisate yield. It determines the novelty of this study.

Purpose of the study is to determine the effect of pre-treatment of medium-temperature electrode pitch in an oxidising environment at T > 400 °C on the yield of pitch carbonisate.

Main body

Medium-temperature electrode pitch of category B (AO Altai-Koks, Russia) was used as a feedstock. The selected pitch had the following characteristics: melting point T_{melt} = 71.5 °C, volatile yield X = 61.1%, ash content = 0.2%, toluene insoluble content α = 25.8%, quinoline insoluble content α_1 = 4.5%.

Fig.1 shows the scheme of heat treatment of pitch in oxidising medium.

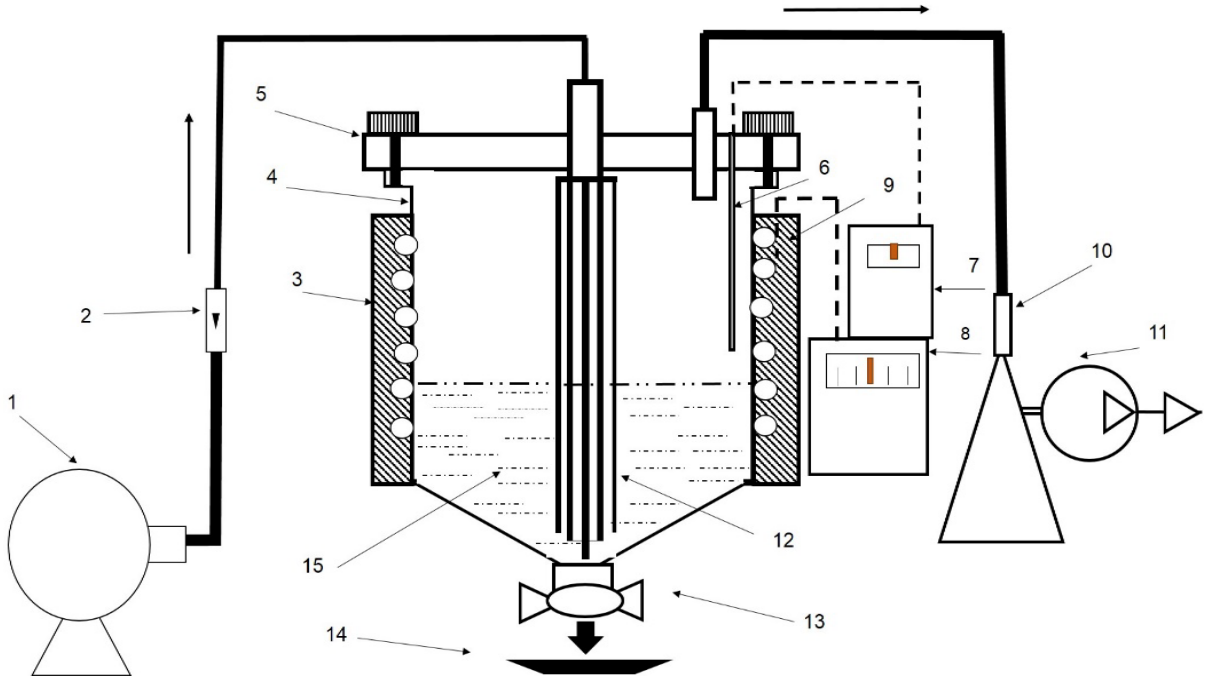

Fig. 1. Scheme of the installation for heat treatment of pitch in oxidising medium: 1 - compressor; 2 - rotameter; 3 - electric heating; 4 - reactor; 5 - reactor cover; 6 - thermocouple for measuring the temperature in the reactor; 7 - secondary device; 8 - thermoregulator; 9 - thermocouple for measuring the heating temperature; 10 - Bunsen flask; 11 - pump; 12 - air supply unit consisting of 6 tubes of 3 mm diameter; 13 - drain valve; 14 - tray; 15 - pitch.

We loaded a 100 g weight of pitch into a stainless steel reactor (*4*) closed with a metal lid (*5*) with a volume of 5.6 litres. The reactor cover was fastened tightly to the vessel with screws. Heating of the pitch in the reactor was performed using an electric heating system (*3*), the temperature in the reactor was increased to the set value on the thermoregulator (*8*).

The temperature in the reactor was measured using a thermocouple (*6*); the indications were displayed on a secondary device (*7*). To mix the pitch to a homogeneous mass, air was supplied by a compressor. Air was supplied through the gas supply unit (*12*), consisting of six tubes with a diameter of 3 mm, deep into the volume of molten pitch. The compressor and pump were switched on during the thermostatting process at the set temperature. Pumping out of light components of the pitch by the pump was required to increase the growth of T_{melt}. The maximum air flow rate (30 l/h) was set by rotameter (*2*). Under the air pressure, the products from the gas phase left the reactor through a pipe in the reactor lid and were transported to a receiver in the form of a Bunsen flask (*10*) by the pump (*11*). After the experiment, the pitch was drained through a drain valve (*13*) into a stainless steel tray (*14*).

The electric heating system (*3*), thermoregulating (thermocouple *9* and thermoregulator *8*), and thermo-measuring unit (thermocouple *6* and secondary device 7) of the unit allow ones to conduct heat treatment of pitch by heating and thermostatting in the temperature range of 400-500 °C.

Air supply exactly at a given temperature range is required to exclude reactions at the temperature range 260-400 °C. It causes a sharp increase in α_2 and α_1 in the pitch [30-31], and use of air to provide a more economical oxidising medium for low-temperature carbonisation. The selected optimum air flow rate (30 l/h) was required to ensure efficient mixing of the molten pitch to obtain a homogeneous product. However, it reduced possibility of increasing the number of thermo-oxidative reactions in the pitch. Indeed, the pitch was thermostated with air treatment. Under the action of air, the distillates from the reactor were intensified and pumped out by a pump into the receiver. The melting point T_{melt} by 'Ring and rod' method according to GOST 9950-2020, yield of volatile substances X was measured according to GOST 9951-2023 for heat-treated pitches were measured. Ash content was measured according to GOST 7846-73.

Heat-treated pitches (heat-treated products) were carbonised in muffle furnace crucibles with lapped lids. Carbonisation was conducted under the following modes: *mode 1*: heating to T = 850 °C and holding for 1 hour; *mode 2*: heating to 650 °C and holding for 1 hour; *mode 3*: heating to 450 °C and holding for 1 hour, with further heating to 850 °C and secondary holding also for 1 hour; *mode 4*: heating to 450 °C and holding for 1 hour, with further heating to 650 °C and holding also for 1 hour, and heating to 850 °C and holding for 1 hour. We determined the carbonisate yield K as the percentage of the mass of carbonisate obtained to the mass of pitch. The extracted liquid distillates were viscous liquid of dark brown colour and completely dissolved in chloroform.

Table 1 shows the heat treatment conditions of B category pitch.

Table 1. Heat treatment conditions for coal pitch B

Name	BTO-1	BTO-2
Process temperature, °C	410	445
Duration, min	60	60
Air consumption rate, l/h	30	30

The values of T_{melt} and X were obtained for the products (BTO-1 and BTO-2) for heat treatment of medium-temperature electrode pitch of category B.

Table 2 shows the melting point T_{melt}, volatile yield X for the initial pitch, and pitch after heat treatment.

Table 2. Value of T_{melt} and changes in volatile yield X for initial pitch and heat-treatment products

No.	Name	T_{melt}, °C	X, %	Pitch yield, %
1	Pitch B	71.5	61.1	———————
2	BTO-1	158	36.2	62
3	BTO-2	140	44.1	67

According to Table 2, the value of X for BTO-2 is higher than that of BTO-1. This may be a result of pumping of gaseous products and distillates at obtaining PK-2 was conducted at a higher temperature of 445 °C than at obtaining PK-1. However, at T > 410 °C, part of the distillates went to the pitch, as evidenced by the value of BTO-2 pitch yield (67%). It is higher than that of BTO-1 pitch yield (62%). The ash content of the pitch was 2%. It was identical to the ash content of the original pitch.

Carbonisation by heating and thermostatting for 1 hour at 850 °C was conducted for initial electrode pitch of category B, the yield of carbonizate is K = 51%. The yield of carbonisate is K= 53.3% at low-temperature carbonisation by heating to 650 °C and holding at this temperature for 1 hour. Table 3 shows the results of carbonisate yields for BTO-1 and BTO-2 for different carbonisation modes.

For example, for *mode 1*, the carbonisate yield is K (BTO-1) = 62.4%. It is higher in value than that for BTO-2 (60.4%). Moreover, heat treatment (at T > 400 °C) of category B electrode pitch in an oxidising medium increases the K values for the carbonisation product at T = 650 °C by 14-16%. Also, heat treatment increases the K value for the carbonisation product at T = 850 °C by 9-10%. It is also evident from the data on K values for carbonisation modes *3* and *4* that additional holding at T = 450 °C and 650 °C, increases the K values. Additional holding at 450 °C for 1 hour (*mode 3*) increases the K values for further carbonisation at T = 850 °C from 62.4% to 69.3% for BTO-1 and from 60.4% to 65.2% for BTO-2. Two additional holding times at 450 °C and 650 °C (*mode 4*) increase the K values for BTO-1 (from 62.4% to 72.3%) and BTO-2 (from 60.4% to 68.1%), respectively.

Table 3. Carbonisate yields under different carbonisation conditions.

Carbonisation mode		K (BTO-1), %	K (BTO-2), %
1	*Mode 1*	62.4	60.4
2	*Mode 2*	69	67
3	*Mode 3*	69.3	65.2
4	*Mode 4*	72.3	68.1

The increase in carbonisate yield (Table 3) may be due to heat treatment of the pitch at T > 400 °C leads to a marked increase in the value of α_1 fraction according to [10]. It increases the value of K. Additional temperature control at 450 °C and 650 °C resulted in a marked increase of the K value. Therefore, the additional hold times increased the time of chemical reactions (polymerisation according to [27, 29]). It could increase quantitatively the yield of carbonisate K. Also, additional curing at 450 °C could further increase the α_1-in pitch according to [10]. It significantly increased the K value.

The volatile yield V^{daf} was determined for carbonisates obtained from BTO-1 according to GOST R 55660-2013. Volatile yield is Vd^{af} = 2.87% for the carbonisate obtained by carbonisation according to *mode 1*. Volatile yield is Vd^{af} = 1.88% for carbonisate obtained by carbonisation according to *mode 4*. For carbonisate (*mode 1*) of initial B category pitch, the volatile yield is V^{daf} = 4.01%. Hence, heat treatment in an oxidising medium at 410 °C of pitch causes a decrease in the yield of volatile substances in the obtained carbonisate. The combination of thermal oxidative heat treatment at 410 °C and multiple holding times during carbonisation reduces the volatile yield for the final product by 50%. This improves the quality of the resulting carbonisate.

Conclusions

Thermal treatment of medium-temperature electrode pitch of category B in an oxidising medium makes it possible to obtain high-temperature pitch.

Preliminary thermal heat treatment in oxidising medium of medium-temperature electrode pitch of category B increases the value of carbonisate yield in comparison with the value for initial pitch.

Preliminary thermal heat treatment in oxidising medium of medium-temperature electrode pitch of category B reduces the volatile yield for carbonisate compared to the value for the initial pitch.

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