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IMPACT OF THERMO-OXIDATION OF ELECTRODE FURNACE ON CHANGES IN ITS COMPOSITION AND PROPERTIES

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Abstract. *The author conducted a series of experiments on obtaining pitch with increased softening point T_p = 110-120 °C by low-temperature thermo-oxidation (up to 300 °C) of electrode coal ash of category B (T_p = 91 °C). The research determines the thermal oxidation conditions for obtaining these types of pitches. The paper also defines the dependences of softening temperature and yields of thermo-oxidation products on process duration and air flow rate.*

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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 (270-360 °C); coal tar pitch (T < 360 °C). The study of the structure and properties of coal pitch and the methods of its production has great prospects for obtaining new technologies in the production of electrode pitch [1].

Coal pitch is mainly used as a binder for electrode products and anode mass. Electrode pitch of categories B1, C are mainly used as a binder for anode mass [2]. Pitches with an increased softening point T_p > 100 °C and high-temperature pitches have great prospects as a binder. Internationally, pitch with T_p = 110-115 °C is used as a binder for anode mass [3, 4]. This type of binder performed well in obtaining electrodes and anode mass according to the results presented in [4].

According to [5-7], pitch with increased softening temperature reduces the destructibility of the anode mass, namely, for pitch with softening temperature T_p = 100-110 °C, the anode mass has a maximum of compressive strength and a minimum of specific electrical resistance. It was experimentally shown in [5-7], that the viscosity value was higher for pitches with T_p = 110-120 °C compared to electrode pitches. Indeed, for these types of pitch at T = 140 °C there is an increase in viscosity from 200 to 500 Pa·s, an increase of the surface tension from 46 to 48·10^{-3} N·m, and an increase of coke yield from 87 to 90.6% [5-7]. For pitches with

T_p = 105-120 °C, the content of α_2 and β-fractions, determining the binding properties of the pitch, had the maximum values [5-7]. Also according to [5-7], the decrease in strength for these pitches is related to the increase in the α_1-fraction. According to [8], the charge for anode mass, presented as a pecocoke composition based on a binder made of high-temperature pitch, had high plasticity and viscosity in comparison with pecocoke compositions based on electrode pitch.

There are many works devoted to obtaining these pitches. In [9, 10], the binder pitch was obtained by thermal oxidation of anthracene fraction with additives of 2B (8.3%) and D (4.8%) coals. Pitches had a softening point of 111 °C for the pitch obtained using anthracene fraction with additives of coal type 2B, and 120 °C for the pitch with additives of coal type D [9, 10].

In [11], a pitch-binder with T_p = 112 °C was obtained by thermal oxidation of coal tar by adding rubber crumb -5% by mass. In [12], a binder pectic with T_p = 114 °C, α = 43.7, α_1 = 7.8, was obtained by extraction of fatty gas coal in anthracene oil in a 1/2 ratio. In [13], binder pitch was obtained by thermal dissolution of gas and fatty coals in anthracene oil followed by thermal oxidation. Furthermore, a pitch with T_p = 119 °C was obtained using gas grade coal and a pitch with T_p = 111 °C using fatty grade coal, respectively [13].

The prospects of obtaining binder pitches using semi-coke tar should also be noted. In [14], a pitch with T_p = 120 °C, α = 34.8% and α_1 = 0.2% was obtained by distillation of semicoke resin to 410 °C and holding at this temperature for one hour. The only disadvantage of this pitch was the high volatile yield of 64% [14]. In [15], a binder pitch with T_p = 104 °C with benz[a]pyrene content 20 times lower than that of coal electrode pitch was obtained by thermooxidation of semicoking resin for 30 min at an air flow rate of 20 l/kg.

The pitches obtained by mixing products of coke-chemical industry with petroleum products are of particular interest and prospective application as a binder. In [16-19] it was shown, that petroleum and petroleum-coal pitches, have low benz[a]pyrene content compared to coal pitch. In [19], binder oil-coal pitches were obtained. Pitches with T_p = 116 °C were obtained by mixing coal and oil in the ratio 70/30, and pitches with T_p = 119 °C – in the ratio of components 55/45. In [20], a pitch with T_p = 110 °C was obtained by mixing coal tar and heavy pyrolysis tar 85:15% with distillation to 410 °C followed by thermooxidation at T = 275 °C for 4 h and air flow rate 26.4 l/kg.

In [7, 21], binder pitches were prepared by thermoxidation of medium-temperature pitches. In [22], a pitch with T_p = 111 °C was obtained by thermal preparation (process temperature 410 °C, duration 3 h) of pitch with softening point 65.8 °C with low-temperature thermooxidation of this pitch for 3 hours. As a result, a pitch with a softening point of 117 °C was obtained. The results indicate the advantage of the medium-temperature thermal oxidation method over other methods in obtaining binder pitches.

Particular attention should be given to obtaining these pitches by thermal oxidation of low temperature or soft pitches. In [23], a pitch with T_p = 46 °C was obtained with T_p = 112 °C and α = 37.1% by thermal oxidation of a low-temperature pitch with Tp = 46 °C for 4 h at 350 °C. These results show the prospect of obtaining binder pitches by thermal oxidation of low-temperature and medium-temperature pitches.

In this study a number of experiments on low-temperature thermo-oxidation of electrode pitch of category B to obtain pitch with T_p = 110-120 °C will be performed. This type of thermal

oxidation reduces the content of benz[a]pyrene in the final product according to the results in [24, 25]. These statements determine the relevance of the study.

For the first time the research determined the optimal conditions for obtaining pitch with increased T_p = 110-120 °C, by thermo-oxidation of electrode pitch of category B. Moreover, for the first time the authors established the dependences of T_p and yields of obtained pitches on duration and air flow rate at thermo-oxidation of electrode pitch of category B. The above statements define the novelty of this research paper.

The purposes of this study are to obtain dependences of pitch yields and their softening point on the duration of low-temperature thermo-oxidation of electrode pitch of category B. We have to determine the conditions under which low-temperature thermo-oxidation of electrode pitch of category B can produce pitch with T_p = 110-120 °C. We plan to determine the technical analysis data for the obtained pitches with increased softening point.

Description of materials, experimental setup and research methods

In this study, the pitches will be heat-treated under air in the unit shown on Fig. 1. Previously, this unit was used to produce high-temperature pitch in [26, 27].

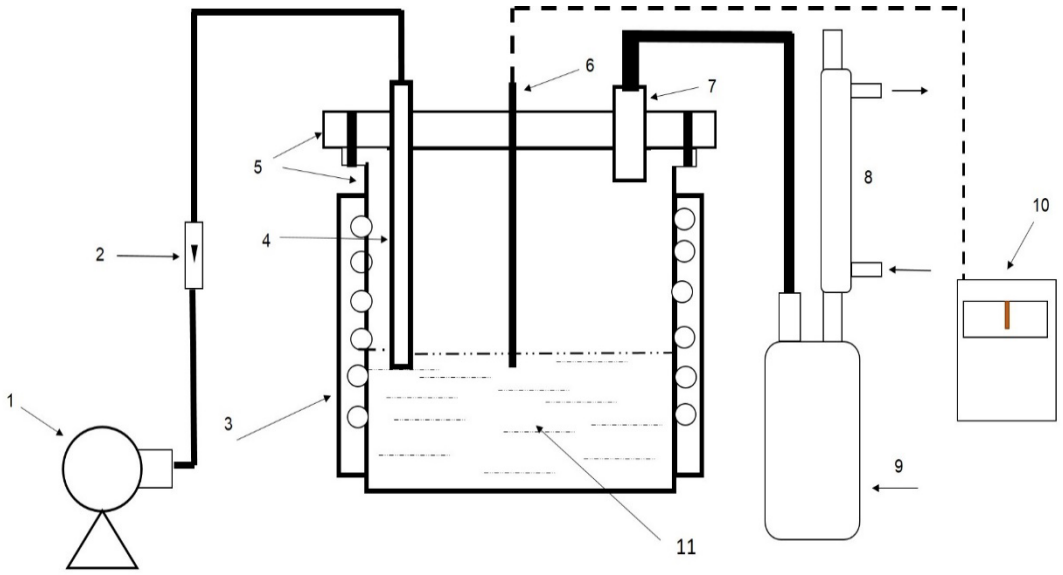

Fig. 1 Scheme of the installation for thermal oxidation of pitch [27]. 1 - compressor; 2 - rotameter; 3 - electric furnace; 4 - cylindrical tube for oxidation; 5 - cylindrical reactor with a cover attached to it by screws; 6 - thermocouple; 7 - outlet pipe for exhaust gases and distillates; distillates receiver; 8 - refrigerator; 9 - distillates collector; 10 - thermoregulator; 11 - pitch.

We put the pitch in a cylindrical reactor with a volume of 4 litres, height of 15 cm, and diameter of 10 cm (5). Air was supplied by a compressor (1) and passed through a cylindrical tube (4) 9 mm in diameter to oxidise and mix the molten pitch with distillate extraction. Under air action from the tube (4) the thermo-oxidation of the pitch took place and exhaust gases-distillates were removed from the reactor.

We used electrode coal pitch, category B (AO 'Altai-Koks', Russia) with softening temperature T_p = 91 °C. The authors conducted a series of experiments with different durations of the thermo-oxidation process up to the maximum process temperature of 300 °C and air flow

rates Q (air flow rate was calculated by the following formula: $q = \frac{Q}{t \times m}$, where t is the duration of thermal oxidation, m is the mass of pitch suspension).

We determined the parameters of thermal oxidation products of B category electrode pitch based on the results of fractional composition of the obtained pitches. We defined the softening temperature T_p of the obtained pitches by the method 'Ring and rod' (GOST 9950-83), and the content of α-fraction insoluble substances in toluene by GOST 7847. The content of α_1-fraction insoluble in toluene and quinoline was ascertained according to GOST 10200 by centrifugation. The α_2-fraction content was determined using the following formula $\alpha_2 = \alpha - \alpha_1$. We performed technical analysis according to known methods and establish the yield of volatile substances X according to GOST 9951-73, ash content of pitch was determined according to GOST7846-73.

Production of pitches with increased softening point

Fig. 1 shows the experiments on obtaining pitches with increased softening temperature T_p = 110-120 °C. A suspension of pitches m = 250 g was used for the pitch. The initial temperature of the experiment was 25-27 °C; air from the compressor was supplied to reach the heating temperature of 260 °C. The oxidation tube was in direct contact with the sample. The molten pitch was mixed with air during the experiments, ensuring homogeneity of the initial product. The air flow rates Q are set equal to 40; 70; 100 l/hour. We obtained dependences of the pitch yields W on the duration of the thermal oxidation process (Fig. 2).

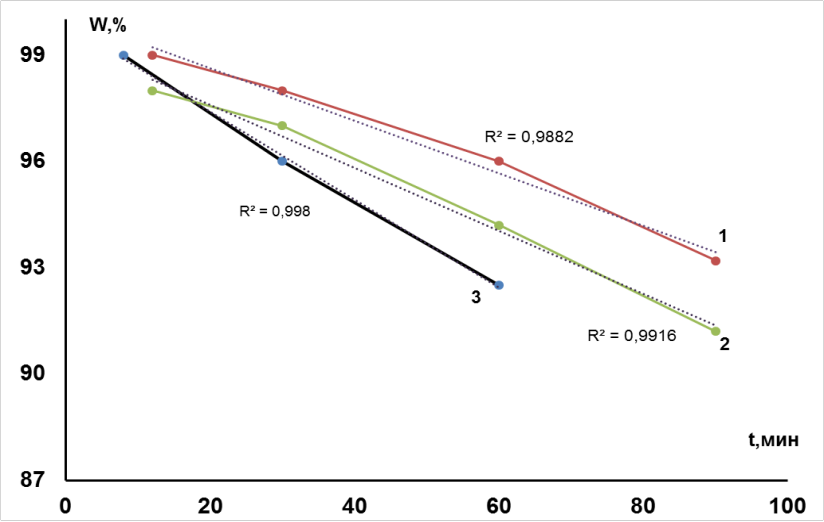

Fig. 2. Dependence of pitch yields W after low-temperature thermo-oxidation on process duration for different air flow rates: 1 - Q = 40 l/h; 2 - Q = 70 l/h; 3 - Q = 100 l/h.

Fig. 2 shows that with increasing duration of thermo-oxidation there is a decrease in the pitch yield. Also the pitch yield decreased with increasing air flow rate. The minimum value of W = 92.5% corresponded to Q = 100 l/h and duration of 60 min. At process durations t > 40 min, the dependencies for Q = 70 and 100 l/h (curves 2 and 3) were monotonically decreasing and the dependence on duration was linear. The W dependence for curve 1 had a sharp decline at t = 60 min, associated with a decrease in W value from 96 to 93% when the duration was increased to 90 min.

For a detailed study, we consider the dependence of the pitch yield on the air flow rate. Fig. 3 shows the dependence of pitch yield after low-temperature thermooxidation on air flow rate q.

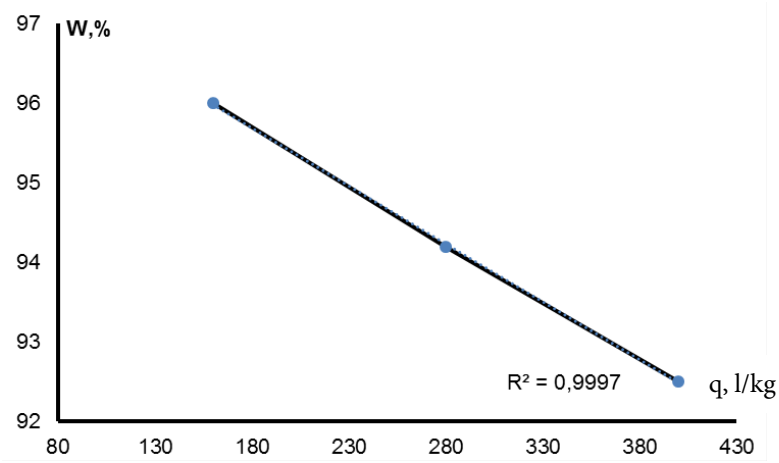

Fig. 3. Dependence of pitch yields W after low-temperature thermooxidation (duration 1 h) on air flow rate q.

Fig. 3 shows that the yield of W pitch at thermo-oxidation of electrode pitch of category B during 1 hour linearly decreased from 96 to 92.5% at increasing air flow rate from 160 to 400 l/kg.

Fig. 4 shows the dependence of T_p on the duration of thermal oxidation.

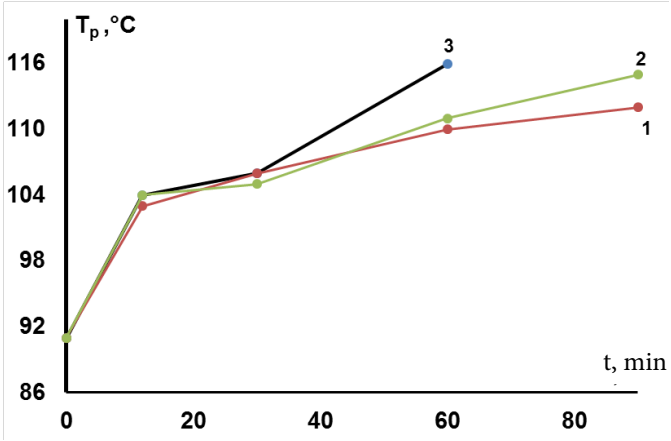

Fig. 4. Dependence of softening temperature of low-temperature thermo-oxidation products on process duration: 1 - Q = 40 l/h; 2 - Q = 70 l/h; 3 - Q = 100 l/h.

Fig. 4 shows a sharp increase of T_p from 91 to 103 °C for Q = 40 l/h and up to 104 °C for Q = 70 and 100 l/h. The increase of T_p from 104 to 106 °C for durations ranging from 12 to 30 min was weakly dependent on the airflow rate. For Q = 100 l/h and increasing the process duration to 60 min, a sharp increase of T_p to 116 °C was observed, while at Q = 40 and 70 l/h the softening temperatures were 110 and 111 °C, respectively. When the duration was increased from 60 to 90 min, there was an increase in T_p to 115 °C at Q = 70 l/h. T_p was 112 °C for this duration at Q = 40 l/h.

Also the detailed consideration of the changes in the value of T_p from the air flow rate requires attention to the dependence on the value of the air flow rate. Fig. 5 shows the dependence of T_p after low-temperature thermal oxidation of electrode furnace B on the air flow rate q.

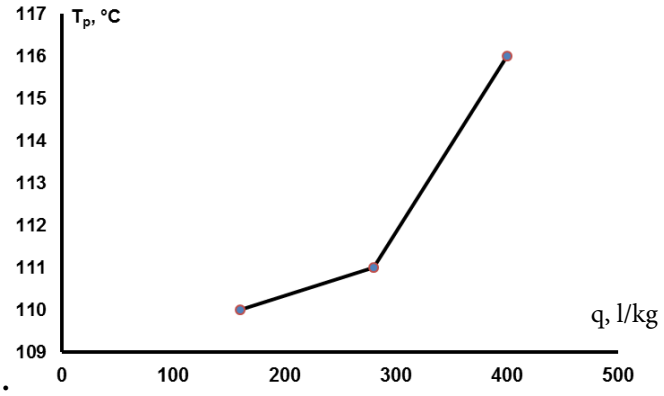

Fig. 5. Dependence of T_p for pitches after low-temperature thermooxidation (duration 1 h) on air flow rate q.

Fig. 5 shows that the value of T_p, increased from 110 °C to 116 °C when the air flow rate increased from 160 to 400 l/kg during thermo-oxidation of electrode pitch of category B for 1 hour.

We obtained the pitch of our particular interest by thermooxidation for 90 min at an air flow rate of 40 l/h (pitch B_{T1}) and 70 l/h (pitch B_{T2}). And pitches B_{T3}, B_{T4} and B_{T5} obtained by thermal oxidation with 60 min duration, with air flow rates of 40, 70, and 100 l/h.

Table 1. Data of pitches obtained by low-temperature TO of medium-temperature pitches

№	Name	t, min	Q, l/h	W, %	T_p, °C	α_1, %	X, %
1	Electrode pitch B				91	7.5	53.0
2	B_{T1}	90	40	93.0	112	7.6	49.6
3	B_{T2}	90	70	91.2	115	7.5	45.1
4	B_{T3}	60	40	96.0	110	7.5	49.8
5	B_{T4}	60	70	94.2	111	7.6	45.3
6	B_{T5}	60	100	92.5	116	7.5	45.5

Table 1 shows that the α_1-fraction content for all pitches remained almost unchanged compared to the original electrode pitch regardless of the air flow rate and process duration. The content of $_\alpha$-fraction was determined for B*T1* pitch. Compared to the initial pitch, the increase of α fraction (increase of α_2 fraction from 26.8 to 34) was low from 34.3 to 41.5%. The growth of α_2-fraction in turn increases the total value of $\beta+\alpha_2$-fraction. It determines the binding properties of pitch.

The volatile yield X decreased from 53.0% to ~ 49% for B_{T1} and B_{T3} when thermo-oxidised with Q = 40 l/h. Also, the value of X decreased from 53 to ~ 45% for B_{T2}, B_{T4} and B_{T5} during thermo-oxidation with Q = 70 and 100 l/h. The value of ash content did not change during thermo-oxidation and remained identical to the value for the initial pitch ~ 0.15%.

We obtained B_{T2} and B_{T5} pitches at a minimum duration of 60 min but with high air flow rates of 70 and 100 l/h. Increasing Q to 70 and 100 l/h makes it possible to obtain pitches with T_p = 115 °C and T_p = 116 °C while reducing t to 60 min.

Discussion and conclusion

We consider the dependence of the change of T_p on the duration of low-temperature thermo-oxidation (Fig. 4). A sharp increase of T_p up to 104 °C occurs at duration t = 12 min. Also the increase of T_p to the values of 101 °C and 103 °C was observed at thermo-oxidation to T = 275 °C (in the first case for the time t = 5 min and Q = 40 l/h, in the second case for t = 3 min

and Q = 100 l/h, respectively). The content of the α fraction increased from 34 to 37% for the first case for pitch, while the α_2 content increased from 26.5% to 29.5%.

Pitches with T_p = 110 °C and 111 °C were obtained by low-temperature thermo-oxidation of 60 min duration, at Q = 40 and 70 l/h. Increasing Q from 40 to 70 l/h for this period of thermooxidation causes an increase in the intensity of stirring of molten pitch and separation of distillates (as can be evidenced by the decrease in the yield of pitch W from 96 to 94% (Fig. 2) and a decrease in the yield of volatile substances X (see Table 1)). However, it does not change the intensity of chemical reactions. Therefore, to increase T_p for the thermo-oxidation product at Q = 70 l/h, an increase in the thermo-oxidation duration t from 60 to 90 min is required.

Increasing t to 90 min, for Q = 70 l/h resulted in an increase of T_p for B_{T2} pitch (T_p = 115 °C) compared to B_{T1} pitch (T_p = 112 °C) obtained at Q = 40 l/h. Increasing Q to 100 l/h ensured the stirring efficiency of the molten pitch and promoted the growth in the number of $\gamma \rightarrow \alpha_2$ reactions in the gas phase (according to [28, 29]), leading to an increase of T_p up to 116 °C in the final product B_{T5}. The absence of growth of the α_1-fraction is due to absence of C-C bond breaking during low-temperature thermooxidation; there is no possibility of reactions in the liquid phase of the following type $\beta \rightarrow \alpha_2 \rightarrow \alpha_1$ leading to a noticeable growth of the secondary α_1-fraction associated with the mesophase [28, 29]. A similar result was observed previously in the thermo-oxidation of electrode pitch B by supplying air to the pitch through an oxidation unit in [30]. According to [30, 31], the growth of T_p during thermo-oxidation was associated with an increase in the α_2 fraction and a decrease in the yield of volatile substances X.

Increasing the air flow rate from 160 to 400 l/kg during thermo-oxidation of electrode pitch of category B decreased the yield of pitch from 98 to 92% (Fig. 3) and reduces the yield of volatile substances X from 49 to 45%.

Conclusions

1. The authors obtained the dependence of softening temperature of products (pitch) of thermo-oxidation of electrode pitch category B on the duration of the process.

2. T_p increases and the yield of pitch decreases with reducing air flow rate of thermooxidation of electrode pitch B.

3. The authors have determined the conditions for obtaining pitch with T_p = 110-120 °C, by thermo-oxidation of electrode pitch of category B.

4. Experimentally shown that the pitches obtained after low-temperature thermo-oxidation of B category pitch did not increase the content of α_1-fraction with increasing the time of thermo-oxidation up to 90 min, but there was a growth of T_p.

5. A pitch with T_p = 112 °C and α-fraction content of 41.5% was obtained by low-temperature thermo-oxidation of B category pitch.

6. A pitch with T_p = 116 °C was obtained at a minimum duration of 1 h but at a maximum air flow rate of 100 l/h.

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