SECTION 4

IRRADIATION INSTALLATIONS, DIAGNOSTICS AND RESEARCH METHODS

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PRODUCTION OF ACTIVATED CARBON FABRIC FROM COTTON PAPER PRECURSOR

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A method for obtaining activated carbon fabric from its own available raw materials by carbonization with subsequent activation is proposed. Three types of fabric were used as a precursor: a waffle towel, a dense tarpaulin and a harsh cotton tarpaulin "Jute 470". The effect of ammonium chloride and ammonium phosphate as carbonization catalysts, as well as potassium hydroxide as a catalyst-activator, has been studied, the methodology and results of experiments are presented.

INTRODUCTION

Recently, interest has increased in the use of carbon materials in electrode systems of electrochemical devices, such as capacitors, supercapacitors, batteries, filters [1–3]. Such devices require electrode systems with a high specific surface area in combination with a high electrical conductivity of the material, and most often with a low resistance to fluid flow, which is accompanied by a decrease in the ohmic resistance of the electrolyte. For such purposes, electrodes made of carbon fabrics are the most suitable. In this case, a significant factor is also the reduction in the cost of production of electrode materials. A possible way to reduce the cost of production of activated carbon materials can be the use of fabrics made from natural fibers as a precursor. The purpose of this work was to select the type of cotton fabric for carbonization, to study the possibility of using catalysts and to obtain the optimal heating regime during carbonization and subsequent activation of the fabric.

EXPERIMENTS FOR PRECURSOR CHOICE

The experiments were carried out on pyrolysis furnace of the AGAT type [4]. Technological equipment (Fig. 1) was made of carbon-carbon composite materials. It is a tubular carbon-carbon composite heater (reference [1]), closed at the ends with graphite covers (reference [4]). Inside the heater, in a special hole in the bottom cover, a stainless pipe is installed, on which carbon fabric "Ural" is wound up to a diameter of 61 mm. Such a soft winding should provide the necessary deformation during tissue shrinkage during pyrolysis and ensure the preservation of its integrity. The heater walls are perforated, which ensures free gas exchange between the internal working volume of the reactor and the working volume of the pyrolysis unit chamber. To reduce energy consumption, the outer surface of the heater is wrapped with two layers of asbestos fabric.

In the first experiments, **waffle towel fabric** was used as a precursor. Due to the presence of a three-dimensional structure, this fabric achieves free shrinkage of textile fibers during carbonization.

Experiments on carbonization and activation (development of the specific surface) were carried out on the AGAT-2.0 furnace, using a fore vacuum as a process medium.

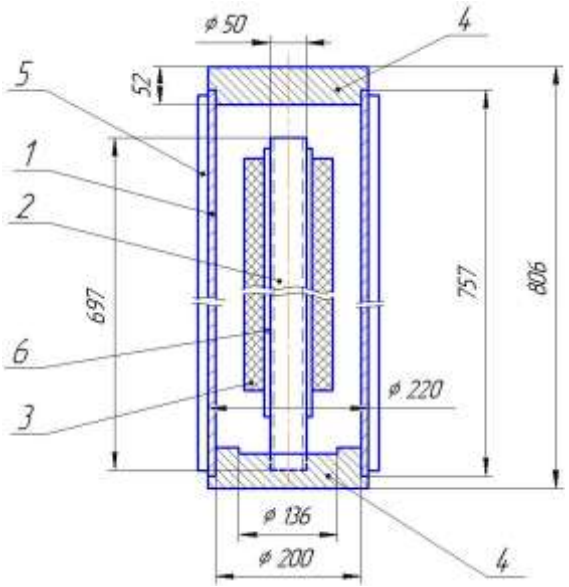

Fig. 1. Tooling for fabric carbonization

At the initial stage of the experiments, a direct carbonization process was carried out without the use of flame retardants, catalysts, and other auxiliary reagents.

The design of the reactor is made in such a way that conditions close to isothermal are realized inside. Temperature control and maintenance is carried out using a thermocouple inserted through one of the holes in the heater directly into the internal volume of the reactor.

The carbonization of cotton fabric was carried out according to the temperature rise graph shown in Fig. 2. At the first stage – heating and carbonization of the

fabric in vacuum to a temperature of 1000 °C for 24 h, at the second stage – activation in a CO_2 environment at 1000 °C for 20 min.

CO_2 was used as an activating agent, which oxidizes carbon fabric at high temperatures. The yield of carbon fabric after carbonization and oxidation was 9.3% of the original fabric.

The specific surface was determined on the obtained samples. To compare the results, control samples of industrial carbon activated materials were used. Samples of the following carbon materials were taken for testing: Busofit, S-AUT (produced in Belarus) and the material obtained at KIPT.

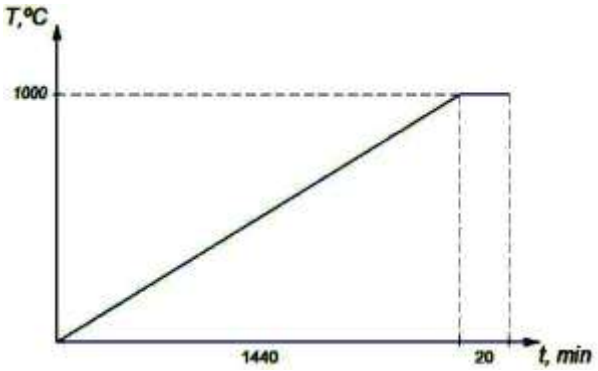

Fig. 2. Temperature chart for carbonization and activation of wafer towel fabric

The specific surface area was determined by the Brunauer-Emmett-Teller (BET) method. The results of measurements of the specific surface are presented in Table 1. The obtained values of the specific surface are averaged over three measurements.

Table 1

Specific surface of activated carbon materials determined by the BET method

Material	Specific surface, m^2/g	Variation in magnitude, m^2/g
Busofit	400	±40
KIPT	800	±80
S-AUT	1000	±100

As can be seen from the presented results, we obtained a material with a sufficiently high specific surface area (at the level of analogues). The main disadvantage is the low strength of the material, which is associated, among other things, with the small thickness of the material obtained.

In continuation of a series of experiments, a **tarpaulin fabric** with a surface density of 245.1 g/m^2, a roll width of 1.518 m was used as a precursor. In these experiments, the final temperature during carbonization and activation was reduced to 900 °C. The maximum yield of carbonized and activated fabric from the tarpaulin increased and amounted to 16.2%, however, the mechanical properties of the resulting carbon fabric are extremely low, which, apparently, is explained by the dense structure of the original fabric, which limits the movement of the threads during their shrinkage during carbonization.

In further experiments for carbonization, a new fabric **"tarpaulin harsh cotton/Jute 470"** (trade name) was used. Said tarpaulin has a surface density of 470 g/m^2. The equipment is left unchanged, as in the previous experiment. The experiment was carried out in vacuum. The mode of carbonization and activation is shown in Fig. 2. Loaded 4510 g fabric, received 887 g. carbon fabric, the output was 19.67% of activated carbon fabric, the resulting fabric is stronger than previously obtained samples, the disadvantage can be considered excessive stiffness of the fabric.

When a large amount of tissue is pyrolyzed, the vacuum depth in the installation drops significantly, which is mainly due to the slow pumping of the evacuated products through a mechanical filter located between the chamber and the fore-vacuum pump. Thus, in this case, it is more correct to call the vacuum medium the medium of rarefied pyrolysis products. A graph of the dependence of the change in pressure in the chamber on temperature is shown in Fig. 3, while the value 245 corresponds to -1 atm on a mechanical vacuum gauge.

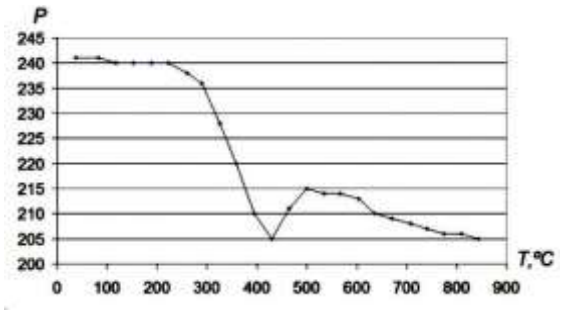

Fig. 3. Graph of pressure change in the furnace from temperature

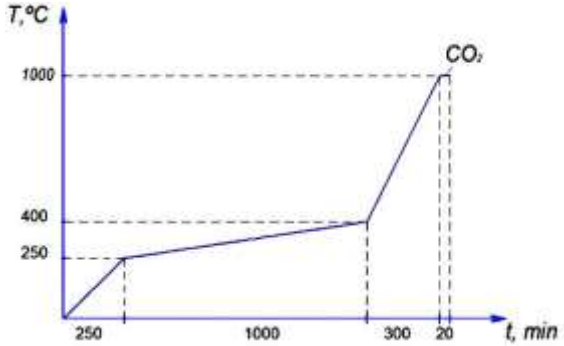

Fig. 4. Temperature rise mode for fabric "tarpaulin harsh cotton/Jute 470"

Based on these data, it can be seen that the process of gas evolution during carbonization occurs unevenly. In the range from 250 to 400 °C, this process is more intense, which indicates the intensity of pyrolysis processes. Considering this fact, for the next experiment, the corrected mode was used, in which the rate of temperature rise was reduced for the indicated range. The graph of temperature rise for the following experiments is shown in Fig. 4. The experiment was carried out in the AGAT-1.6 installation at atmospheric pressure, with the initial filling of the evacuated installation with argon.

USE OF CATALYSTS

In further experiments, the influence of pyrolysis catalysts and the method of their application on the mass yield of carbon fabric after the carbonization

process was tested. The processes of carbonization and activation were separated. For the experiment, 6 pieces of fabric measuring 0.49 x 2 m were used, two in each of the three processes. The first is the control (samples 1 and 2). Pure samples were carbonized to a temperature of 1000 ºC.

In the second process, fabric samples No. 3, 4 **were soaked** in a catalyst solution: a mixture of ammonium chloride, NH_4Cl and ammonium phosphate, $NH_4H_2PO_4$ in a mass ratio of salts 3: 2, 17% solution of a mixture of salts in distilled water.

In the third process, tissue samples No. 5, 6 **were boiled** in the same solution. Carbonization was carried out in an argon atmosphere. The temperature rise plot for this experiment remained the same and is shown in Fig. 4.

After carrying out three carbonization processes, respectively 1, 2, then 3, 4, then 5, 6, the obtained samples were weighed and wound on the same equipment for activation. The activation process was carried out at a temperature of 1000 °C in a CO_2 environment with a holding time of 20 min. The results of the experiment are presented in Table 2.

Subsequent experiments were carried out in a combined process of carbonization and activation. The entire fabric was soaked in a catalyst solution of a mixture of ammonium chloride and diammonium phosphate. Carbonization was carried out in an argon atmosphere. After reaching a temperature of 1000 °C, the activation process was carried out in a CO_2 medium with a holding time of 20 min. At the same time, carrying out the carbonization process with large loads of tissue led to an increase in the maximum yield of carbon material to 35.1%, which is apparently associated with the deposition of solid products of gas-phase reactions on the tissue.

The appearance of carbonized and activated samples is shown in Fig. 5.

Further carbonization was subjected to a mixed load of the fabric "Tarpaulin harsh cotton/Jute 470" and small samples of fabric for waffle towels. There were used 6 pieces of fabric "tarpaulin harsh cotton/Jute 470" measuring 0.49 x 2 m, and 6 pieces of fabric for waffle towels measuring 0.47 x 0.5 m. The fabrics were soaked with distilled water before winding, the catalyst was not used, the equipment and the mode of carrying out the carbonization process were completely similar to the previous experiments. The results of the carbonization and activation processes are presented in Table 3. The last column of the table shows the results of measuring the specific surface area for some of the carbonized and activated tissue samples.

The activation led to a significant increase in the specific surface area of the carbon fabric samples in the outer layers of the winding, regardless of the raw material used. But for the inner layers that were subjected to weak activation, a much larger value of the specific surface area of the carbon fabric obtained from the fabric "tarpaulin harsh cotton/Jute 470" is noticeable compared to the carbon fiber obtained from the fabric for towels, which, according to apparently due to the presence in the composition of jute fibers, in addition to cellulose, significant amounts of lignin and pectin. The heterogeneity of the chemical composition of the fibers apparently leads to an increase in the porosity of the carbonized material.

In further experiments, the processes of carbonization and activation were studied without the use of carbon material oxidation in the gas phase.

To do this, the processes of carbonization and activation of the tissue must be divided into two stages: the stage of preliminary carbonization of the tissue, and the stage of final carbonization-activation of the resulting carbon tissue, using different, incompatible catalysts at each stage of the process.

At the first stage, the fabric "tarpaulin harsh cotton/ Jute (470)" was soaked in a solution of a flame retardant catalyst – 10% ammonium chloride (NH_4Cl) solution in distilled water, then the fabric impregnated with a catalyst solution was wound on a carbonization equipment, dried in oven at 110 °C. Carbonization was carried out in an argon atmosphere. The graph of the temperature rise for the first stage of the experiment is shown in Fig. 6. During the first stage, heat treatment is accompanied by the decomposition of ammonium chloride according to the reaction:

$$NH_4Cl \leftrightharpoons NH_3 + HCl.$$

The hydrogen chloride released during the reaction accelerates the pyrolysis of the tissue and increases the yield of carbon. Complete decomposition of ammonium chloride ends at a temperature of 338 °C. The first stage of tissue carbonization is also accompanied by the release of the main part of the gaseous products of pyrolysis, including the release of a certain amount of carbon dioxide.

Table 2

Results of an experiment on carbonization and tissue activation using catalysts

No.	Weight before carbonation, g	Note	Weight after carbonization, g	Yield of fabrics, %	Weight after activation, g	Yield of tissue after activation, %	Weight before washing, g	Weight after washing, g
1	507	Without catalysts	114	22.48	98	19.3	99	99
2	503		99	19.68	85	16.9	84	85
3	506	Soak	165	32.6	132	26.1	134	132
4	497		156	31.39	123	24.75	124	122
5	532	Boiling	184	34.59	159	29.89	159	155
6	543		167	30.76	125	23.0	121	121

Fig. 5. Appearance of the activated carbon fabric obtained from the precursor "tarpaulin harsh cotton/Jute 470"

Table 3

Results of the carbonization processes and activation of a mixed load of fabric "tarpaulin harsh cotton/Jute 470" and small samples of fabric for waffle towels

No. piece of fabric, from the rod out	Weight before carbonization, g	Weight after carbonization, g	The yield of carbonized fabric, %	Specific surface area of carbonized fabric, BET method, m^2/g
1t*	34	5	14.7	180
1	509	115	22.6	540
2t	32	5	15.6	–
2	512	117	22.9	–
3t	33	5	15.2	210
3	514	117	22.8	530
4t	33	4	12.1	–
4	520	116	22.3	–
5t	32	5	15.6	–
5	516	105	20.3	–
6t	32	4	12.5	800
6	520	72	13.8	1000

* t – sample of fabric for waffle towels.

At the second stage, the catalyst-activator potassium hydroxide is used, which promotes the development of the surface of the carbon fabric. At the second stage, the fabric that has cooled down after preliminary carbonization is impregnated directly on the tooling with a 60% solution of potassium hydroxide, and after draining off excess solution without drying, it is immediately installed in the chamber of the AGAT-1.6 pyrolysis furnace and subjected to heat treatment in an argon medium according to the chart shown in Fig. 7.

To assess the effect of potassium hydroxide on the process of activation of carbon fabric in the first experiment, we wound three pieces of fabric "tarpaulin harsh cotton/Jute 470" impregnated with ammonium chloride, and carried out the process of preliminary carbonization. At the second stage of carbonization-activation, only fabric samples No. 1 and 2 were impregnated, sample No. 3 was separated and left dry as a control, after which the final carbonization-activation process was carried out in accordance with the temperatre rise schedule shown in Fig. 7.

Fig. 6. Temperature rise mode for fabric pre-carbonization step

In the next experiment, the possibility of reducing the pyrolysis time for small loads of tissue was tested, the time of the pre-carbonization stage of the tissue was reduced by 5 times, according to the graph shown in Fig. 8.

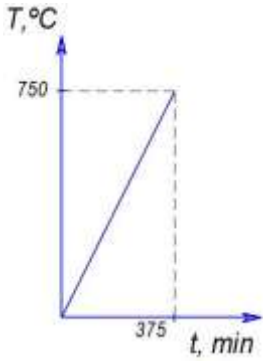

Fig. 7. Temperature rise mode at the stage of final carbonization-activation of carbon fabric

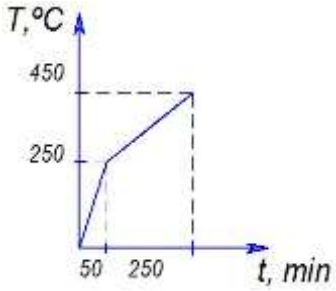

Fig. 8. Reduced temperature rise for the pre-carbonization stage

Table 4

No. piece of fabric	Weight before carbonization, g	Weight after carbonization, g	Yield of carbonized fabric, %	Specific surface area of carbonized fabric, m^2/g
1	514	176	34.2	–
2	513	179	34.9	392
3	517	189	36.6	237
4	527	184	34.9	–
5	523	175*	33.5	200
6	527	202	38.3	165

The second stage of the final carbonization-activation of the obtained carbon fabric was also carried out according to a reduced heat treatment schedule, the temperature rise rate was increased by 2 times, the pyrolysis-activation time was 187 min.

The results of the processes of carbonization and activation for these experiments are presented in Table 4, while tissue samples No. 3 and 6 are control, not impregnated with a solution of potassium hydroxide.

From the data obtained, a preliminary conclusion can be drawn about the admissibility of accelerated tissue pyrolysis.

CONCLUSIONS

In the course of the experiments, a method was proposed for obtaining activated carbon fabric from the own raw materials available in Ukraine. The fabric has a sufficient specific surface area. The disadvantage of the fabric obtained is its low strength.

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ОТРИМАННЯ АКТИВОВАНОЇ ВУГЛЕЦЕВОЇ ТКАНИНИ З БАВОВНЯНОГО ПАПЕРОВОГО ПРЕКУРСОРА

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Запропоновано методику отримання активованої вуглецевої тканини з власної доступної сировини методом карбонізації з подальшою активацією. В якості прекурсора було використано три види тканини: вафельний рушник, щільний брезент і брезент суровий бавовняний «Джут 470». Вивчено дію хлористого амонію і фосфату амонію в якості каталізаторів карбонізації, а також гідрооксиду калію в якості каталізатора-активатора, наведено методика і результати експериментів.