

## Features of implementation options for the process of high-temperature activation of carbon material

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**Abstract:** The paper shows the relevance of research in the development of activated carbon materials with a high specific surface area and porosity, which can be used as materials for the separation or storage of various liquid and gaseous media. A plurality of approaches to the implementation of the activation process, characterized mainly by the accumulation of experimental data and laboratory studies, is noted. The possibility of realizing the process of carbonization activation in two variants has been established: the first is high-temperature alkaline activation, the second is high-temperature alkaline activation with an additional activator in the form of water vapor. The most rational temperature regimes for the implementation of this activation process are determined, which are 750 °C for the first variant, and 600 °C for the second, as well as their key features are defined. The characteristics of the carbon material obtained as a result of two variants of activation – specific surface area and porosity – are analyzed, and their dependence on the temperature of this process is established. The possibility of industrial implementation of the considered activation options is substantiated, taking into account the peculiarities of their implementation to obtain an activated highly porous carbon material with a specific surface area of more than  $2700 \text{ m}^2 \cdot \text{g}^{-1}$  and a porosity in the micro- and mesosize range of more than  $1.3 \text{ cm}^3 \cdot \text{g}^{-1}$ .

**Keywords:** high-temperature activation; options for carrying out activation; characteristics of the carbon material; features of the technological design of the process.

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## Особенности вариантов реализации процесса высокотемпературной активации углеродного материала

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**Аннотация:** Показана актуальность исследований в области разработки активированных углеродных материалов с высокой удельной поверхностью и пористостью, которые могут быть использованы в качестве материалов для разделения или хранения различных жидких и газообразных сред. Отмечена множественность подходов к осуществлению процесса активации, характеризующая преимущественно накоплением опытных данных и лабораторными исследованиями. Установлена возможность реализации процесса активации карбонизата по двум вариантам: 1) высокотемпературная щелочная активация, 2) высокотемпературная щелочная активация с дополнительным активатором в виде водяного пара. Определены наиболее рациональные температурные режимы реализации данного процесса активации, составляющие для первого варианта 750 °C, для второго – 600 °C, а также их ключевые особенности. Проведен анализ характеристик полученного в результате двух вариантов активации углеродного материала – удельной поверхности и пористости, и установлена их зависимость от температуры проведения данного процесса. Обоснована возможность промышленной реализации

рассмотренных вариантов активации с учетом особенностей их проведения, для получения активированного высокопористого углеродного материала с удельной поверхностью более  $2700 \text{ м}^2/\text{г}$  и пористостью, приходящейся на микро- и мезоразмерный диапазон более  $1,3 \text{ см}^3/\text{г}$ .

**Ключевые слова:** высокотемпературная активация; варианты проведения активации; характеристики углеродного материала; особенности технологического оформления процесса.

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## 1. Introduction

The analysis of works devoted to the development and study of highly porous carbon materials with a large specific surface area and porosity with a predominance of micro- and mesopores is an actual direction of many studies, the results of which are in demand in a number of branches of modern industry. These materials are the most promising as universal sorbents for liquid and gaseous media, devices for storing and transporting gaseous fuels, catalysts, fertilizer carriers, fuel cells, solving environmental problems in such industries as petrochemistry, energy, radio electronics, medicine, and agriculture. This is explained, first of all, by the presence of a balanced combination of a developed system of micro- and mesopores, with a significant specific surface, corresponding accessible pores and their large volume, with the presence of sufficiently large transport pores that ensure rapid diffusion of sorbed substances, chemical inertness and stability in real conditions of using such materials [1–7].

To activate carbon materials, various carbon raw materials, such as phenol-formaldehyde resin, hydroquinone, natural coals, carboxymethylcellulose, furfural, dextrin, urotropine, as well as carbon nanotubes, graphene, etc. are pre-carbonized, and then subjected to activation by various gas-phase or liquid-phase reagents: various acids or alkalis, water vapor, etc. [8–13].

Moreover, one of the most effective activation methods is alkaline, which ensures the formation of high specific surface area and porous structure with a predominant content of micro and mesopores [14–17].

High-temperature alkaline activation is a process of heat treatment of the initial carbonizate consisting of pre-carbonized carbon raw materials with alkali in a given ratio, usually at a temperature in the range from 400 to 900°C in an inert atmosphere. During the heat treatment of the reaction mixture, consisting of carbonizate and alkali, a number of chemical reactions occur. This process is accompanied by significant gas evolution and the formation of a porous structure of the activated material.

Preliminary studies carried out by the authors have shown that the balance of ongoing processes largely depends on both the feedstock used to produce carbonizate and the technological activation modes – the duration and temperature of the process, the size of the activation reactor, the amount of the activated substance and the activator, their ratios, etc. [18–20].

For example, in [21], the activation process of carbon raw materials at 300–500 °C in a continuous flow of an inert gas was considered, in [22, 23] chemical activation was carried out at 750 °C, while in [24–26] activation was carried out at 800 °C, and in [27] at 900 °C.

The authors of [28] studied the effect of alkaline heat treatment on the specific surface area and porosity structure in the temperature range of 600–800 °C. And in [29], activation was studied at temperatures from 700 to 900 °C.

In [14, 30–32], the influence of the purge rate of the reaction zone with an inert gas was studied, and the efficiency was improved with an increase in the flow rate, while in [28] a decrease in the quality of the obtained activated material was noted, considering the specific surface area and volume of micro-mesopores as the main qualitative criterion. In [18], a clear extremum was established when analyzing the effect of an inert gas flow on the properties of the activated material.

The authors of [33] used sodium hydroxide (NaOH) as an activator, while in most studies it was demonstrated that activation with potassium hydroxide (KOH) seems to be a more effective method of increasing the specific surface area and porosity [10–13, 16, 32, 34–36].

In [37–39] it was shown that various processing conditions, and especially the nature of oxidizers, can have a significant positive effect on the characteristics of a carbon material. Thus, the source [40] considers the multifactorial effect on the activated carbon material not only of alkali, but also of water vapor, which acts as a mild oxidizing agent, while alkali promotes the occurrence of complex redox reactions between carbon and compounds

formed as intermediate products in activation time. Moreover, in [37–40] it is noted that the beginning of the activation process falls already at 500–600 °C.

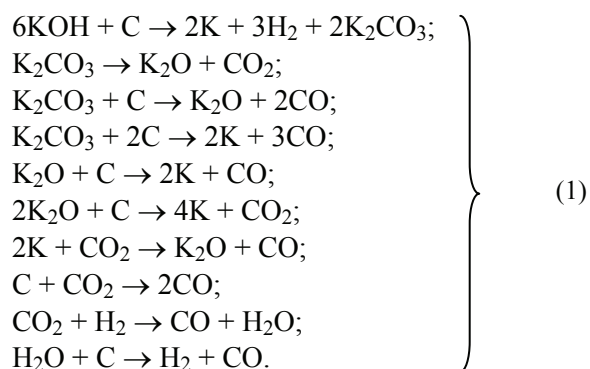
The analysis of literary sources and preliminary works of the authors showed that the results of studies of high-temperature alkaline activation are very contradictory, in addition, they are considered mainly in the framework of laboratory conditions and do not take into account the specifics and features of real production.

Thus, the authors of this research set the goal to evaluate two options for the carbon material activation: high-temperature activation with one activator – alkali and with two activators – alkali and water vapor in relation to the conditions of real industrial implementation by analyzing the characteristics of the obtained activated material and the features of the technological process.

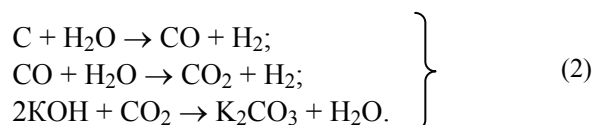
The preliminary studies of the authors made it possible to select the most effective ratios of the reaction mixture – carbonizete and alkali, as well as the most rational operating parameters of the activation process [41, 42], due to the possibility of implementing this process in a fairly wide range of technological regimes:

*The first activation option* (activation with one activator – alkali at high temperatures) is the process of heating the reaction mixture, consisting of carbonizete with alkali in a given ratio, to 400 °C, followed by holding for one hour and further heating and holding at 750 °C in within two hours. During the entire activation process, an inert atmosphere is maintained in the reactor due to the supply of argon.

Previous experiments made it possible to establish that during the activation of the reaction mixture, various chemical reactions take place, which is accompanied by intense gas evolution occurring in the ranges of approximately 400–550 and 610–750 °C and ultimately determining the surface parameters and porosity of the resulting material. The high-temperature alkaline activation process can be described by the following equations (1):



According to *the second activation option*, in which, in addition to alkali, vapor is used as an activator (as a result of the dosed supply of distilled water to the activation reactor and its evaporation in it), the Boudouard redox reaction proceeds, in addition to those reactions discussed above. This reaction occurs on the surface of the activated reaction mixture along the phase boundary and has the following form (2):



This variant is also characterized by lower temperatures of intensive processes, 450–500 and 550–600 °C, respectively.

Since the key difference between the activation options, first of all, is the temperature of the process, the process and the result of its implementation, as well as characteristics of the activated carbon material, will be considered with respect to this parameter. The most relevant characteristics, i.e. specific surface and porosity: total pore volume and micropore volume, will be compared in practical application (preliminary studies have shown that when using carbonizete, a predominantly microporous carbon material is obtained).

## 2. Materials and Methods

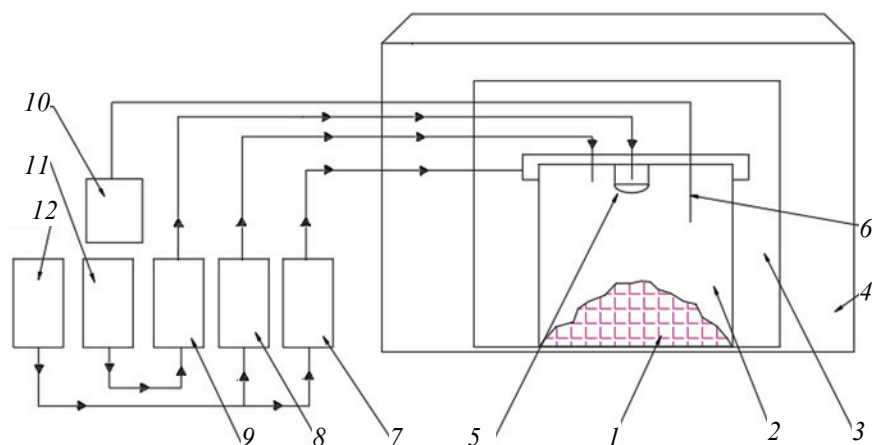
### 2.1. Initial materials and obtaining carbonizete

The following consumables were used as initial materials: initial carbon raw material is carbonizete (consisting of a mixture of dextrin, 75 % (CJSC “Dekstrinzavod”, Murom, Russia) and graphene oxide, 25 % (NanoTechCenter Ltd., Tambov, Russia). The components were mixed for one hour to dissolve and obtain a homogeneous mixture, and then heat-treated in a reactor in a stream of argon for 8 hours at 400 °C. Upon activation, a mixture of carbonizete and potassium hydroxide (RM Engineering, Moscow, Russia) was taken in a ratio of 1:3, inert gas is argon.

### 2.2. Experimental activation setup

To study the process of high-temperature activation according to *activation options 1 and 2*, an experimental laboratory setup was developed (Fig. 1).

The experimental setup for activation consisted of a muffle furnace 3 installed in a fume hood 4, where there was an activation reactor 2, with an activated reaction mixture 1. The tightness of the reactor was ensured by the supply of inert gas from



**Fig. 1.** Scheme of the experimental laboratory setup:

1 – activated material; 2 – activation reactor; 3 – muffle furnace; 4 – fume hood; 5 – bowl for water evaporation; 6 – thermocouple; 7 – inert gas flow meter for supply to the gas lock; 8 – inert gas flow meter for supply to the activation reactor; 9 – dispenser; 10 – programmer; 11 – tank for distilled water; 12 – inert gas cylinder

cylinder 12 to its internal volume and gas lock, the gas supply rate was regulated by laboratory flow meters 7 and 8. A bowl 5 was installed on the inner surface of the reactor lid to evaporate distilled water supplied to the reactor with the help of a dispenser 9 from the tank 11. The furnace heating was controlled using a thermocouple 6 and a programmer 10.

Experimental studies included several series of activations at temperatures of 600, 750, 800 and 900 °C. Each series consisted of five activations carried out according to *options 1 and 2*, respectively.

The temperature range of activations is from 600 to 900 °C, with the lower temperature limit due to the possibility of the process, and the upper one based on data from the literature, preliminary studies of the authors and the capabilities of the technological equipment used.

Diagnosis of the specific surface and porosity parameters of the obtained activated carbon material was carried out by nitrogen adsorption using the Nova Quantachrome E1200 analytical complex (Quantachrome, Boynton Beach, USA). In this work, for the analysis of the material, the BET models were used to determine the specific surface area and DFT models were used to determine the specific pore volume and their size distribution. The processing of the obtained data was carried out according to the method of measuring the specific surface area and processing the results of studies of nanosized materials [43].

### 3. Results and Discussion

As a result, activated carbon materials were obtained with a specific surface area according to  $S_{\text{BET}}$  – more than 2700 m<sup>2</sup>·g<sup>-1</sup>, according to  $S_{\text{DFT}}$  –

more than 2000 m<sup>2</sup>/g and a specific pore volume  $V_{\text{DFT}}$  – more than 1.3 cm<sup>3</sup>·g<sup>-1</sup>, where micropores accounted for more than 80 %. Analyzing the experimental data, it can be noted that activated carbon materials with very similar parameters were obtained. However, at the same time, they were with a pronounced shift in regime extrema due to the peculiarity of the implementation of a particular activation *option*.

When carrying out activation according to *the first option*, it can be assumed that the course of the main reactions of hydroxide reduction and carbon oxidation with the release of H<sub>2</sub>, CO и CO<sub>2</sub>, i.e. the reaction of potassium in the mixture and, accordingly, the formation of a high specific surface area and porosity (the active phase of activation) occurs most intensively at a temperature of about 750 °C, which is also confirmed by a number of works [28–31, 35–37]. At the same time, a decrease in the specific surface area of  $S_{\text{BET}}$  was observed at temperatures from 800 °C and more, which is associated with the onset of destruction of the porous structure of the material with an increase in temperature and an increase in the pore volume in the mesometric range, respectively (Fig. 2, 3).

A similar effect is also observed when implementing the second option, and the most effective temperature value is already 600 °C, when using an additional activator – vapor, which acts as a mild oxidizer. At this temperature, the strongest synergistic effect of temperature and vapor on the formation of specific surface area and pore volume is observed. Also, from the obtained results it can be seen that with increasing temperature, there is a decrease in the specific volume of pores with a shift



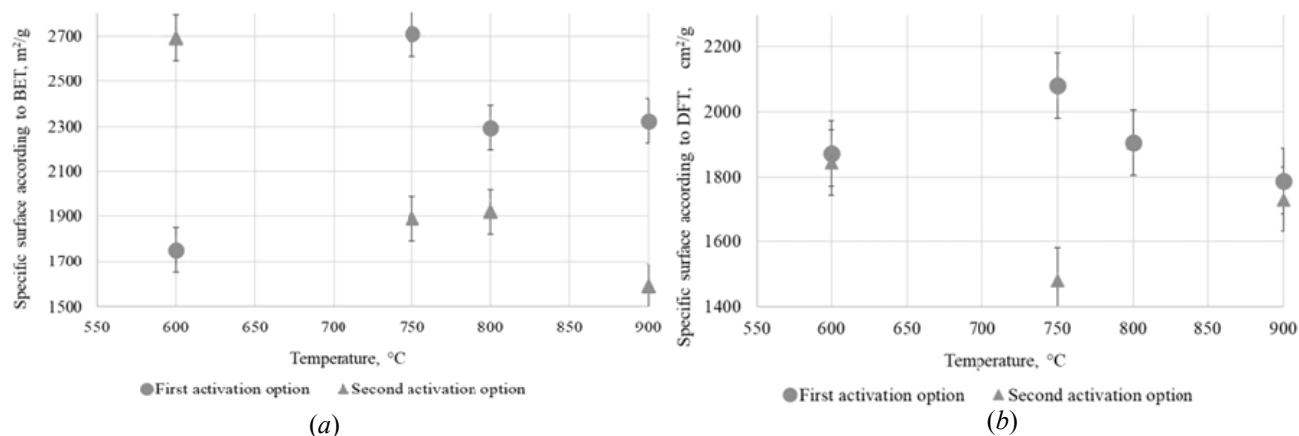


Fig. 2. Dependence of specific surface area on temperature:

*a* – specific surface parameters according to the BET model; *b* – specific surface parameters according to the DFT model

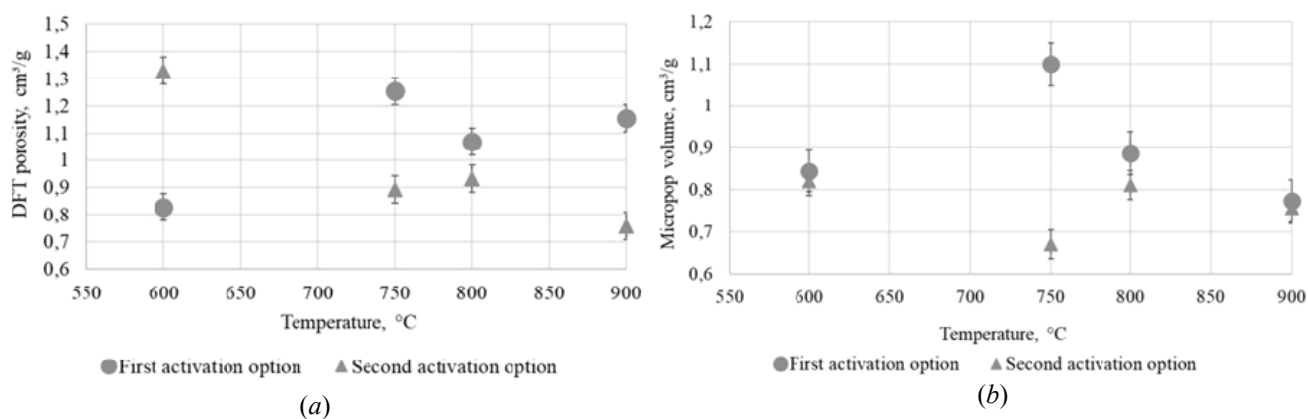


Fig. 3. Temperature dependence of porosity:

*a* – specific porosity parameters; *b* – volume of micropores

in their distribution mainly to the microrange. This can be explained by the intensification of the combined effect of high temperature, potassium compounds and the presence of vapor. The results obtained are in good agreement with the chemical processes that occur during activation, as well as those considered in [38, 39], and indicate a shift in equilibrium with changing conditions compared to the first option of activation.

Analyzing the *activation options* discussed above, their features, it can be noted, in relation to the practical implementation of these technologies, that both *options*, in general, are equivalent. The difference between these *options*, first of all, is that in the *first option*, the activation process is carried out at a high temperature with one activator – alkali. One of the main features of which is the high loading of the process, i.e. flowing at a significant temperature and aggressive environment, which contributes to the emergence of various corrosion processes [44].

The *second option* is the activation process using an additional activator – water vapor, carried out at much lower temperatures, which is an essential factor

for the industrial implementation of the process (reducing the activation temperature from 750 to 600 °C is a transition to simpler technological processes and equipment). The features of this option also include sensitivity to temperature changes, occurrence in a medium saturated with water vapor, the need for a metal catalyst (potassium), and the most efficient process at the phase separation boundary [40, 42].

At the same time, the development and improvement of the technological process and equipment, taking into account the specifics of high-temperature activation to obtain an activated material with a specific surface area of more than 2700 m<sup>2</sup>·g<sup>-1</sup> and a porosity of more than 1.3 cm<sup>3</sup>·g<sup>-1</sup>, is a typical task that can be successfully solved in real conditions of industrial production in relation to both options for the implementation of the process.

#### 4. Conclusion

The conducted studies have shown that at the moment there is a plurality of approaches to the implementation of the activation process, which

indicates a lack of unambiguity in understanding the ongoing processes, their mutual influence, and is also characterized mainly by the accumulation of experimental data in the laboratory.

At the same time, the possibility of implementing the activation process in two options was experimentally established and the key parameters affecting its implementation were identified, as well as the similarity and dependence of the characteristics of the obtained carbon materials, i.e. specific surface area and porosity, on temperature.

An analysis of activation options from the point of the industrial implementation of these technologies showed that in the first option, as more loaded (the equipment will be affected by the complex effects of adverse factors: temperature and aggressive environment), but simple in implementation, special attention should be paid to the selection of structural materials and design of the equipment. In the second option, which has more complex execution (selection of regime parameters related to the size of the activation reactor, the amount of activated material, activators, etc.), more attention will be required to develop specific technological regimes, but at a lower temperature.

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## 7. Conflict of interests

The authors declare no conflicts of interest.

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