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Study of the Activation Process of Ti-Carbon-Containing Minerals Based on Rice Husk

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ABSTRACT: The article discusses the methods and conditions of the activation processes. The results of carbonization carried out due to thermal oxidative destruction are presented. The results of differential thermal analysis of rice husks are discussed. To study the effect of chemical activation on the composition of coal adsorbents, we chose mass amounts of modifiers in the mixture equal to 3, 6, 10, 13, 18%, temperatures - 700, 800, 850, 900 and 950°C for experiments, heating of the material was carried out in an argon atmosphere with exposure equal to 1, 2 and 3 hours. The mass ratio of the components TiO_2:KOH in the composition of the modifying additive is 1:1.

KEYWORDS:rice husk, activated carbon, titanium, steam activation.

I. INTRODUCTION

Highly porous carbon adsorbents in industry are obtained as a result of thermal and chemical activation. As a result of changing the conditions of the processes, it is possible to purposefully change the porous structure of the adsorbent. However, in many cases, complex activation methods are used, which differ among themselves in the duration of the process and the index of the finished product yield. During chemical activation, in contrast to steam-gas, the activating agent is uniformly distributed throughout the entire mass of the material and its effect on the processes occurring in the initial carbon-containing material is manifested at all stages of its transformation. In the presence of an inorganic activator, the process of formation of a porous structure of a carbon material occurs within the space occupied by decomposition products of organic matter and an activating agent, therefore, the formation of structural elements of a carbon material and their combination with each other depend on the number, mobility and chemical activity of molecules and ions of the activating agent. The role of the activating agent in this method of obtaining activated carbon materials is decisive and consists in the formation of a homogeneous microporous structure, the pore size of which can be controlled by the amount of inorganic activator introduced [1]. During the heat treatment of any carbon-containing material at the initial stage of decomposition, a large number of crystallization centers arise in it, the simultaneous appearance of which, the low rate of their growth and the release of gaseous substances lead to the formation of a porous carbon skeleton. The development of the volume of micropores can be provided only by those inorganic activators that remain mobile from the moment the structure begins to form until completion. In most of the methods described in the literature, solutions of inorganic substances are used as activators, which are impregnated with the starting unactivated carbon material. The quantitative characteristic of the activation process is the impregnation coefficient, defined as the ratio of the mass of the inorganic activator to the mass of dry ash-free carbon-containing raw materials. Optimal activation conditions are achieved at values of the impregnation coefficient for metal chlorides in the range of 0,6-0,8 g/g (60-80% activator), for sulfur compounds – 0,30-0,35 g/g (30-35% activator) [1], for phosphoric acid – 0,022–0,060 g/g (2,2–6,0% activator) [2]. It can be seen from the above data that the consumption of activators for carrying out the processes is quite large and, from an economic point of view, is not always justified. Activation with alkaline solutions has its own peculiarity: carbon dioxide released at the initial stages of decomposition interacts with hydroxides, forming carbonates, and the activating effect of alkalis will be determined by the properties of their carbonate salts. The disadvantages of these activation methods include an additional stage of dissolution of inorganic substances, as well as a higher cost of the chlorides used in comparison with carbonates. It is generally known that the activation process is carried out with the aim of developing a porous structure of a carbon material. This

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work uses the methods of steam-gas and chemical activation. Steam-gas activation is carried out using water vapor (H_2O) and argon as activators. For chemical modification, TiO_2 and potassium carbonate were used. Steam-gas activation is based on the reactions between carbon and an activating agent:

$$C + H_2O \rightarrow CO$$

When each carbon atom is removed during activation, a free space is formed in the carbon matrix. The set of emerging vacancies forms a porous structure in coal, changing the pore size (most often, there is a tendency to the formation of a large number of micropores), increasing the free volume, this leads to an increase in the specific surface area of the material.

II. SIGNIFICANCE OF THE SYSTEM

The article discusses the methods and conditions of the activation processes. The results of carbonization carried out due to thermal oxidative destruction are presented. The study of literature survey is presented in section III, methodology is explained in section IV, section V covers the experimental results of the study, and section VI discusses the future study and conclusion.

III. METHODOLOGY

The dried rice husk samples were subjected to the carbonization process, which was carried out due to thermal oxidative destruction (TOD) and pyrolysis. Usually, thermal oxidative destruction is understood as the process of thermal activation with air access. The process of thermo-oxidative destruction and activation was carried out in an experimental laboratory setup consisting of an electrically heated reactor with an argon and water vapor supply system, a gas analyzer and a thermocouple. The activation temperature was provided by an external electric heater, which is located on the outer wall of the furnace. To maintain a constant set temperature of the process, an automatic thermostat was used [3]. In order to study the processes occurring during the heat treatment of rice husks (RH), differential thermal analysis was carried out in air and in an argon atmosphere, which simulates the pyrolysis process without air access. The results are shown in Figure 1. The course of the TG and DSC curves as a whole characterizes the processes of destruction of organic components of RS: hemicellulose, cellulose and lignin, which have been studied in a large number of works [4-6]. To study the process of chemical activation, the samples for experiments were prepared by mixing titanium oxide, potassium hydroxide with carbon material in ratios that ensure the required content of the additive in the mixture. During the process, we varied: the content of the additive in the mixture, the activation temperature, and the holding time. The temperature of the activation process when using solid reagents is determined by their melting point. For the experiments, the mass quantities of modifiers in the mixture were chosen, equal to 3, 6, 10, 13, 18%, temperatures - 700, 800, 850, 900 and 950 ^oC, the heating of the material was carried out with an exposure equal to 1, 2 and 3 hours ... The mass ratio of the components TiO_2:KOH in the composition of the modifying additive is 1:1.

IV. EXPERIMENTAL RESULTS

The resulting carbonated product is black and retains its original morphological form.After carbonization, the material was subjected to grinding in a ball mill, followed by particle size classification (Table 1).

Table 1

Partial size, mm	>1	1-0,63	0,63-0,43	0,43-0,32	0,32-0,25	0,25-0,16	0,16-0,10	<0,1	total
Content,% wt.	0,11	0,91	0,29	2,44	0,04	6,36	5,87	83,98	100

Table 1 shows that the highest content (83,98% wt.) In the carbonizate after grinding is characterized by a fraction of particles with a size of <0,1 mm.The need for grinding is due to the further operation of leaching the mineral components, the rate of which depends on the active surface of the solid phase obtained during the activation process.The resulting solid product in the leaching step is a carbon material with a carbon content greater than 70%.

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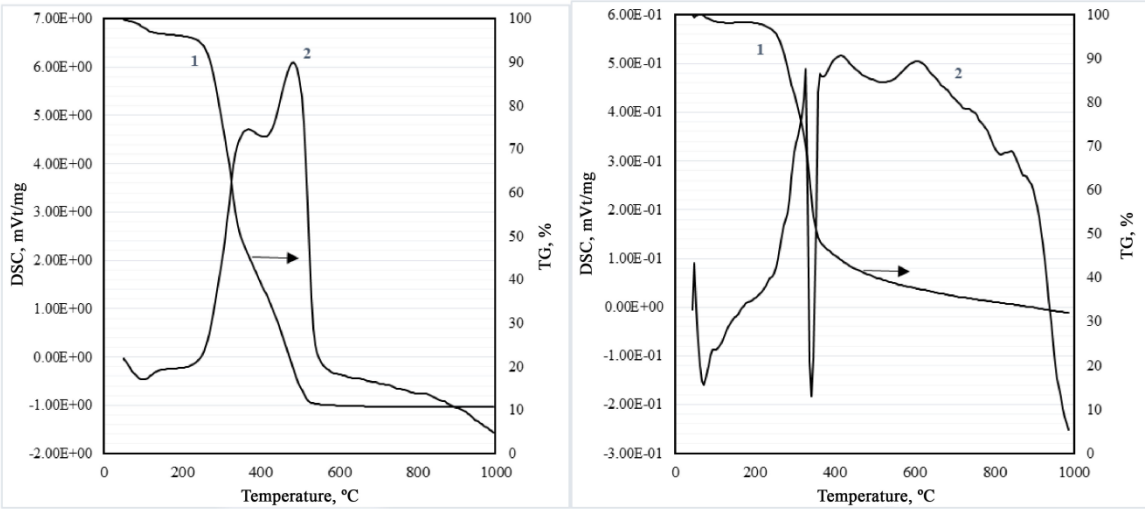

a *b*

Fig. 1.Thermograms of RS in air (a) and argon (b): 1) TG; 2) DSK

The data obtained from Fig. 1 (a) indicate that during the combustion of RS in a stream of air, the material reduces its mass during processing to a temperature of 520 °C by 87,70%, and up to 600 °C by 88,95%. The main weight loss is associated with the destruction of the structures of cellulose and lignin under the action of thermal oxidative destruction in the temperature range 250–520 °C with the release of mainly water, carbon monoxide, and carbon dioxide. A slight decrease in the mass of the material in the range of 520–600 °C (1,67%) is associated with the burnout of carbon formed after the destruction of cellulose and lignin and the release of a small amount of CO and CO_2. Table 2 shows the values of the decrease in the mass of the original RS when calcined in an air flow and an argon flow.

Table 2.
Dependence of the decrease in the mass of the feedstock on the calcination temperature in an air flow and an argon flow

Warm-up temperature,ºC	200	300	400	500	600	700	800	900	1000
Reducing the mass of rice husks when heated in a stream of air,%	3,93	22,28	60,39	84,19	88,95	89,23	89,32	89,27	89,33
Reducing the mass of rice husks when heated in a flow of argon, %	1,98	18,85	54,82	59,83	62,31	64,04	65,49	66,78	67,16

The DSC dependence is characterized by two intense exothermic effects with maxima at 370 and 485 °C. In this temperature range (220–570°C), cellulose and lignin undergo thermal oxidative decomposition. Lignin decomposes at a higher temperature due to its "more aromatic structure", although there is no clear sequence of decomposition, since lignin and cellulose are linked in RS by chemical bonds of different strengths. The yield of the solid residue - carbonizate - during thermal oxidative destruction is insignificant, and it is economically and technologically ineffective to obtain carbon materials by burning most of the sawdust. The study of the dependence of the product yield and the carbon content in the material on the activation temperature and the mass amount of the activator in the mixture showed that the product yield with the amount of addition of the activator equal to 10% by weight and the holding time of 1, 2, 3 hours varies in the range from 65 to 92%. There is a tendency for the yield of the product to decrease with increasing temperature, so the maximum yields are achieved at 700 °C and holding for 1 hour and is 90%. The minimum yields correspond to a temperature of 950 °C, which is naturally associated with the removal of carbon due to the reactions of CO formation. The best indicators of the product yield and carbon content in it are achieved at a

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temperature of 700 °C, since a further increase in temperature leads to the loss of carbon due to its more intensive removal in the form of CO_2 and CO.

V. CONCLUSION AND FUTURE WORK

The pyrolysis process meets the requirements for the production of activated carbon material (AC) most fully.In this case, heating the starting material to a temperature of 550 °C leads to a weight loss of 60,02%, and up to 600 °C - by 62,31%, which suggests that this method is economically feasible for obtaining AC.

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