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PRODUCTION OF COAL ADSORBENTS BY THERMOCHEMICAL METHOD BASED ON COTTON STALKS AND COTTON SHELLS AND THEIR PHYSICAL PROPERTIES

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Abstract: This article talks about adsorbents, in particular, activated carbons, which can be a solution to environmental and water pollution, which is the cause of today's urgent problems. Studies on the sources of raw materials for obtaining activated carbon, methods of their activation and advantages of the method are presented based on scientific literature. Methods of obtaining thermochemically activated adsorbents based on cotton stalks and cotton shells, the effect of pyrolysis temperature on the yield of coal formation, chemical activation of the obtained coal samples with alkali, and the physical properties of thermochemically activated adsorbents were studied. Methods of studying dust density and particle sizes and conclusions based on them are presented. The results of the studies show that the increase of the pyrolysis temperature from 300°C to 500°C led to a decrease in the yield of the produced coal. Due to the low adsorption property of the formed coal, it was thermochemically activated by chemical activation using alkalis such as NaOH and KOH. During thermochemical activation, its adsorption properties increased.

Keywords: Adsorbent, cotton stalk, cotton shells, raw material, activation, thermal activation, activated carbon, thermochemical activation, pyrolysis, carbonization, density, particle size.

Introduction. Today, due to the rapid development of production, the pollution of the environment, water and air is increasing. Various heavy metals, surfactants, petroleum products, paints, etc., pollute the water and the air with toxic gases released from them. The role of adsorbents is incomparable in reducing the quantity of such toxic substances. Thermally stable, chemically resistant and mechanically strong adsorbents are widely used in various industries due to their high sorption properties. Accordingly, it is important to obtain environmentally safe, cheap, easily synthesized, easy-to-use adsorbents and to use them in cleaning industrial wastewater from heavy metal ions, organic compounds, and dyes.

Recently, green adsorbents, such as cellulose-based materials developed through low-energy routes, have received much attention due to their non-toxicity, low cost and renewable nature. Much of the available cellulose is biodegradable, renewable, flexible and easy to modify, making it an ideal raw material for adsorbent materials [1]. Activated

carbon refers to materials based on amorphous carbon with large surface areas and high porosity. The starting raw materials and activation methods used affect the properties of each finished activated carbon. [2]. Activated coal is a porous material with 87-97% of its elemental composition being carbon, the remaining 3-13% being hydrogen, oxygen, nitrogen, and sulfur [3]. The type of raw material used in the production of activated carbons is also important. Because the type of raw material has a significant impact on the quality indicators of the produced adsorbent. The main technological requirements for raw materials are the stability of the chemical composition and structure of raw materials [4]. Stems of various fruit trees, fruit pods, and secondary food waste are cheap raw materials for obtaining activated carbons, which are not widely used in industry [5]. Therefore, many studies have been conducted to obtain suitable adsorbents for adsorption using many raw materials, including; rice husk [6], corn stalks [7], bamboo [8] and others.

For the production of activated carbon, various raw materials with high carbon, low inorganic content, and relatively cheap are usually selected [9].

Recently, cellulose-based adsorbents, which are developed by using low energy, are receiving great attention due to their non-toxicity, low cost, renewable properties. Most cellulose-based materials are biodegradable, renewable, flexible, and easy to modify, making them ideal raw materials for adsorbent materials [10].

As a result of studies from the literature, the composition of various components of the cotton stem was studied. According to their results, the content of α -cellulose in cotton stalk was 42.25%, which is higher than other lignocellulosic resources, and the contents of lignin and ash were 20.62% and 2.71%, respectively.

The process of obtaining FC is carried out on the basis of the following 2 stages [12]:

1. Carbonization
2. Activation

Carbonization is a commonly used thermal decomposition method that is used to create a rigid, cross-linked carbon framework. This is achieved by using pyrolysis methods, which cause the combustion of volatiles and other non-carbon heteroatoms in the form of carbon monoxide, oxygen, hydrogen, nitrogen, methane and water [13].

Activation: It is carried out in order to obtain activated carbon with a pore structure and high adsorption properties from the coal product that has passed through the carbonization process. Physical and chemical types of activation differ [14].

In the climatic conditions of Uzbekistan, cotton is one of the most cultivated types of plants, in particular, for the 2024 cotton harvest, fertile cotton varieties obtained by various genetic methods were planted on nearly 1 million hectares of land. After the cotton crop is harvested, some of the bolls produced in the fields are harvested as fuel, while the rest remains in the fields and becomes a problem for replanting. Above ground parts of the cotton stem (cotton stem and cotton boll), considered as a secondary product of agriculture, were selected as the object of research. Method: Activation methods for obtaining charcoal adsorbent from cotton stalks and cotton shells.

Methods. The cotton stem (P.P) was crushed in 2-3 cm sizes, and the cotton stalk (P.Ch) was separated and crushed (picture: 1).



Picture 1. Cotton stem and cotton boll.

It was dried in a drying cabinet for 24 hours at a temperature of 105°C to get rid of moisture. 200g (accurate to 0.1g) was taken from the dried cotton boll. The raw material was sent to the pyrolysis unit for pyrolysis. The part of the pyrolysis device where raw materials are placed is made of stainless steel. In the production of activated carbon, the starting material was first pyrolyzed for 1.5 hours at different temperatures in an airless environment. As a result, volatile substances (moisture and partial resins) were removed from it (figure 2). The obtained coal product was crushed using a porcelain mortar. The production yield of cotton-based charcoal adsorbent was calculated.

$$A_U = \frac{M_{AC}}{M_B} * 100\% \quad [1]$$

A_U – Product of activated adsorbent, %

M_{AC} – Activated adsorbent mass, g

M_B – Initial raw material mass, g



Picture 2. After thermal treatment of cotton stalk.

The obtained coal product was crushed using a porcelain mortar. Then it was transferred to the chemical activation process. A chemical method was chosen for activation. Solutions of different concentrations of KOH and NaOH were used as chemical activators. Chemically activated at 400°C for 1.5 hours. After the activation, it was boiled and washed with distilled water in order to get rid of excess alkali in the coal. The boiling wash was carried out for 30 minutes, then filtered. This process was carried out 3 times (500 ml of water was used for each wash). Then it was treated with 0.1n HCl for de-ashing. It was washed again with distilled water. The change in pH was monitored

on an indicator piece of paper. It was dried at 105°C for 24 hours (to absolute dry mass). The mesh was passed through a sieve with a size of 0.1 mm. The mass was measured.

Density of activated carbon. The density of activated carbon is an important parameter because it affects the mass and volume of the carbon. The density of activated carbon can be divided into two types.

Bulk density (also called apparent density).

Actual density (also called skeletal or solid density).

The mass density of activated carbons was determined using the pycnometer method. A standardized 20 ml glass cylinder was used for this purpose.

First of all, the mass of the glass cylinder was measured. FK was put into the cylinder and it was hit for 1 minute and delivered to the specified part of the cylinder. The cylinder and coal samples were weighed together, and the mass of the cylinder was subtracted from their combined mass, the net mass of the coal was calculated, and their density was determined according to the following formula.

$$\rho = m/V$$

The results are presented in Table 2.

Particle size. Results were obtained using a Mastersizer 3000 laser particle size analyzer. Mastersizer 3000 (laser granulometer) allows determination of particle size distribution. The principle of operation is based on the laser diffraction method. This method is based on measuring the intensity of scattered light when a laser beam passes through a dispersed sample. The particle size distribution is calculated from the measured angle of light scattered by the dispersed sample particles. It is able to determine the particle size of suspensions, emulsions, dry powders from 10 nm to 3.5 mm. The results are presented in Table 2.

Determining the amount of ash. Determination of the amount of ash in coal was carried out on the basis of GOST 12596-67.

First of all, the porcelain crucibles used for the experiment are brought to a constant weight. For this, the porcelain crucible is fired at 850 °C for 3 hours and cooled to room temperature using a desiccator. It is necessary to pay special attention to the size of the porcelain crucible taken for the experiment (the selected porcelain crucible has a capacity of 15-20 cm³ and a height of no more than 25 mm).

According to the specified standard, a coal sample weighing about 1 g is taken for analysis on an analytical balance with an accuracy of 0.001 g, in the middle of a preheated muffle furnace (to a temperature not higher than 300 °C). will be placed. After the temperature reaches 700°C (analysis temperature), the sample is kept in the oven for 3 hours, after which the crucibles are removed and kept on the porcelain stand for 10 minutes for pre-cooling. After that, the crucible with ash is placed in a desiccator for 30 minutes and then weighed.

The mass fraction of ash in coal in a completely dry sample is calculated using the formula, %,

$$X = \frac{m \cdot 100 \cdot 100}{m_1 \cdot (100 - x)} \quad [3]$$

where m is the mass of the residue after burning.g;

m_1 - the mass of the product sample taken for testing, g;

X is the moisture in the test sample, %.

The difference in the amount of ash in the conducted tests should not exceed 2%.

The results are presented in Table 2.

Result and discussion. In order to obtain carbon adsorbents based on raw materials containing carbon, they are first thermally treated. For this, they are pyrolyzed at high temperatures.

It was found that the selected raw materials for obtaining activated carbon adsorbents turn into coal at a temperature of 300 °C. The pyrolysis process was carried out in the temperature range from 300 °C to 500 °C (in the process, the temperature change every 50 0C was studied). Duration of pyrolysis time was chosen as 1.5 hours.

Table 1. The effect of carbonization temperature on the productivity of carbon adsorbents based on cotton stalks and cotton shells.

Raw material type	Pyrolysis temperature				
	300 °C	350°C	400 °C	450 °C	500 °C
Cotton shells, %	34,5	33,0	32,0	30,5	29,5
Cotton stalk, %	43,0	41,5	40,0	38,5	36,5

From Table 1, it can be seen that increasing the pyrolysis temperature from 300 °C to 500 °C leads to a decrease in the yield of the product. The sorption properties of thermally activated adsorbents are low. In order to improve their adsorption properties, the obtained adsorbents are chemically activated. Different concentrations of alkalis KOH and NaOH were used for chemical activation.

In order to determine the physical properties of activated coal, the density and particle size of coal powder were determined.

Table 2. Coal powder density and particle size.

Raw material type	Activation type	Density	amount of ash	Particle size		
				max	middle	min
Cotton shells	Thermally activated	0,294	5,2%	627	250	20,1
	activated with KOH	0,235	3,7%	642	245	25,3
	Activated with NaOH	0,243	3,5%	564	171	27,8
Cotton stalk	Thermally activated	0,286	4,9%	655	239	55,8

activated KOH	with	0,242	3,5%	613	191	68,7
Activated NaOH	with	0,253	3,6%	614	215	28,3

Conclusion. adsorbents activated by the method of chemical activation were obtained on the basis of cotton stems and cotton bolls. In the process of obtaining adsorbents, the effect of increasing the pyrolysis temperature on the yield of coal was studied. Due to the low adsorption activity of the formed coal, it was activated using a thermochemical method. In order to study their physical properties, the scattering density and particle size were determined.

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