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QUANTITATIVE DETERMINATION OF ADSORPTION ACTIVITY OF ADSORBENTS OBTAINED ON THE BASIS OF COTTON STALK AND COTTON BOLL

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Abstract: This article discusses adsorbents used to reduce water and air pollution, which is one of the current problems of our time. The importance of adsorbents today has been studied. Methods of their production and sources of raw materials have been considered. Information is provided on the need to pay attention to the sufficiency of their sources, low cost, environmental safety, recyclability, and carbon content when choosing raw materials for obtaining adsorbents. Also, the raw material base of cotton stalks and cotton bolls selected for obtaining adsorbents in the republic has been studied. Methods for obtaining adsorbents by thermochemical activation based on cotton stalks and cotton bolls and determining the sorption activity of the obtained adsorbents are presented. The iodine number of adsorbents was used to determine the sorption properties of the obtained activated carbons. The iodine number of adsorbents obtained by thermochemical activation with solutions of NaOH and KOH of different concentrations was determined according to State Standard 33618-2015. Among them, adsorbents activated with 30% NaOH and KOH solutions showed good results in terms of sorption properties and economy. Based on these results, adsorbents with good results in terms of iodine number were selected for further analysis.

Keywords: Adsorbent, raw material, cellulose, carbon, cotton, cotton stalk, cotton stalk, pyrolysis, coal, NaOH, KOH, iodine number, titration, sorption property, thermal activation, thermochemical activation.

Introduction. Nowadays, as a result of the development of industrial sectors, gases and wastewater released from them are causing environmental and water pollution. In the world, one of the important tasks of chemistry is the purification and softening of wastewater in the chemical industry, pharmaceutical and other industries, and in household service enterprises. For these purposes, it is important to produce and widely use adsorbents with high sorption and selective properties. In particular, the role of activated carbon as a non-specific adsorbent in the purification of wastewater from organic pollutants: pesticides, antibiotics, dyes and preservatives is incomparable. Therefore, the use of activated carbons based on cheap and local raw materials in the purification of wastewater from organic substances in industrial enterprises is of great importance.

The development of production in the world, the increase in their capacity, and the increase in the population are causing an increase in the demand for modified adsorbents. As a result, the need for high-quality activated carbon (AC) with high adsorption properties in various industries is also increasing. According to the areas of use of carbon adsorbents, 42% are used in the food production and purification industry, 38% are used for cleaning waste generated in various technological processes, and about 10% of adsorbents are used for environmental safety purposes. The remaining 10% are used in various processes. AC is also used in the synthesis of medical drugs, tablets and antibiotics, and for deep adsorption purification at their final stages [1; 156-158 p.]

LITERATURE REVIEW.

Activated carbon is a carbon-based material with a high surface area and varying degrees of porosity. The raw materials used and the activation methods affect the textural properties of each activated carbon [2; 2062 p.].

From a chemical point of view, activated carbon is a type of carbon that does not have a perfect structure, which contains 87-97% carbon by weight and the remaining 3-13% hydrogen, oxygen, nitrogen, sulfur in various proportions [3]. The surface treatment of carbons or physical activation of raw materials is one of the important processes for obtaining activated carbons. The presence of micropores on the surface of the coal adsorbent increases its specific yield and ensures the effective adsorption of various pesticides. At the same time, the adsorption loss depends on a number of factors, such as the carbonization conditions and activation methods, the type of raw material, modification, particle size in the case of granular or dispersed adsorbents [4; 84-85 p].

In the production of porous carbon adsorbents, it is important to have low cost and high sorption properties of the resulting product. For this purpose, the use of waste materials as raw materials to reduce the cost of the product is considered effective from a practical point of view. In this regard, biomass can be considered as the best candidate [5].

The high quality of activated carbons largely depends on the type of raw material selected for production. The main technological requirements for raw materials are the stability of the chemical composition and structure of the raw material. Although different types of raw materials are used in the production of activated carbons, their elemental composition is similar, but the type of pore structure can differ significantly [6; p. 75-76]. Recently, adsorbents developed through low-energy routes based on green materials such as cellulose have received great attention due to their non-toxicity, low cost and renewability. The abundance of available cellulose is biodegradable, renewable, flexible and easy to modify, which makes it an ideal raw material for adsorbent materials. In addition, the presence of a large number of hydroxyl groups in cellulose can be used in the preparation of adsorbents. However, its low adsorption capacity and very poor solubility limit its application. To improve the adsorption performance of cellulose, it is necessary to increase the number of active sites by chemically modifying the hydroxyl groups [7; 1966-1967 p.]. The abundance of cellulose-containing materials in nature, as well as their biocompatibility and the presence of functional groups, make them a good choice for obtaining cellulose-based adsorbents for water and wastewater treatment. Unmodified cellulose molecules do not have the ability to adsorb metal ions, but they can be modified by chemical or physical processes, forming surface functional groups. As a result, they become environmentally friendly materials with high adsorption capacity for metal ions [8].

Cellulose is a natural polymer produced by plants and trees, and is a naturally biodegradable and environmentally friendly resource. Cellulose molecules have a large number of active hydroxyl groups, so it can be easily modified by introducing various functional groups to the hydroxyl [9; p.1-3]. Currently, cellulose is a widely distributed and renewable biopolymer on earth, and is attracting attention as a raw material for obtaining activated carbons. Also, due to several of the above-mentioned advantages of cellulose, it is used to obtain adsorbents for the purification of wastewater from pollutants [10].

"In Uzbekistan, in 2022, cotton was grown on 1 million 32 thousand hectares in 136 districts of 134 cotton-textile clusters and 29 thousand farms attached to them, with an average yield of 34 centners per hectare, and a total of 3 million 510 thousand tons of cotton were grown," the Ministry of Agriculture reported. Tons of cotton stalks were produced from cotton plants grown on 1 million 32 thousand hectares [11]. Some of them are used as fuel in agriculture, some can be used as feed for livestock, and some can be used as fertilizer. The excess biomass remaining after use is usually burned in the open air or crushed with the help of agricultural machinery. The practice of burning large amounts of cotton stalks in the field further increases the damage caused by the "greenhouse effect" due to the release of many toxic gases, especially CO_2, into the atmosphere. It takes about 6 months for 25-35% of the crushed cotton stalk to decompose in the soil, which causes difficulties in growing the next season's crop. Therefore, it is not recommended to compost the entire amount of cotton stalks produced. Also, it is impossible to feed animals directly with cotton stalks, since they contain a high lignin content and are poorly decomposed by gastrointestinal microorganisms. For biomass used as fodder to be more than 80% digestible, lignin should be less than 10%. Therefore, these three common methods of cotton stalk disposal have their own limitations. Therefore, it is very necessary to use some of these wastes directly as an energy source through gasification or pyrolysis, which leads to rapid processing and waste reduction [12].

While the large tonnage of cotton by-products (stems, leaves, husks and roots) is partly used as fuel, the other part is burned in the fields [13; pp. 90–92].

Cotton-based adsorbents are promising materials in combating the problem of heavy metal pollution of environmental waters. This is due to the low cost, abundance, biodegradability and efficiency of cotton-based adsorbents. Cotton-based adsorbents have been widely studied and many researchers have found them effective for the removal of heavy metals [14; p. 4223–4225].

More than 60% of the agricultural sector of our republic is specialized in cotton cultivation, in particular, in 2024, high-yielding cotton varieties obtained by various genetic methods were planted on about 1 million hectares of land for cotton harvest. After the cotton harvest, part of the cotton stalks, which are produced in large quantities in the fields, are collected as fuel, while the rest remain in the fields, which becomes a problem for replanting. Considering that the cotton stalk and cotton boll consist of cellulose and lignin and are produced in large quantities every year, the above-ground parts of the cotton stalk (cotton stalk and cotton boll), which are considered a secondary agricultural product, were selected as the object of research.

The iodine number in adsorbents, like activated carbon, is a measure of the adsorbent's ability to adsorb iodine. It's a widely used parameter for assessing the quality and surface area of adsorbents, particularly activated carbon. The higher the iodine number, the more iodine the adsorbent can take up, and thus, the more surface area and porosity it likely has. The iodine number provides an indication of the adsorbent's surface area and porosity, making it a useful tool for characterizing adsorbents like activated carbon.

Iodine number determination is usually carried out using a standard titration method based on State Standard 33618-2015. The iodine is absorbed into the adsorbent and the excess iodine is titrated with sodium thiosulfate solution.

METHOD.

Determination of iodine content is carried out according to State Standard 33618-2015 [15]. The standard titer of $Na_2S_2O_3$ is used.

Determination procedure: 1.0 g of coal sample is weighed to the nearest 0.0001 g, placed in a conical flask, 100 ml of iodine solution is added, the lid is closed and shaken in a mixer for 30 minutes. Then the contents of the flask are left until the coal particles settle.

First, the initial amount of iodine in the solution is determined. For this, 10 ml of iodine solution is taken and titrated with sodium thiosulfate solution. Using a pipette, 10 ml of the solution is taken, placed in a conical flask and titrated with sodium thiosulfate solution until it turns straw-yellow. At the end of the titration, 1 ml of starch is added and titrated until the blue color disappears.

The iodine number, X mg/g, is determined by the formula

$$X = \frac{(v_1 - v)*0{,}0127*50*100}{10*m} \qquad (2.5)$$

Where v_1- the volume of 0.1 N sodium thiosulfate solution used to titrate 10 ml of iodine solution, ml.

v- the volume of 0,1 N sodium thiosulfate solution used to titrate 10 ml of iodine solution after treatment with coal, ml

0,0127- the mass of iodine corresponding to 1 ml of 0.1 N sodium thiosulfate solution, g.

50 - the amount of iodine, ml

m - the volume of the coal sample, g

RESULT AND DISCUSSION.

To thermally activate the raw material, first, cotton stalk (G‘P) and cotton boll (PCh) were crushed to a size of 2-3 cm (Figure 1)

Figure 1. Cotton stalk and cotton boll

200 g of each crushed raw material were taken. The obtained samples were dried in a drying cabinet at a temperature of 105 °C to an absolute dry mass. Based on the results obtained, the moisture content of the samples was determined. The moisture content of the cotton stalk was 8,2 percent, and that of the cotton was 6,7 percent. This indicates that the moisture content of the cotton stalk was slightly higher. A certain amount of the dried samples was taken and sent to a pyrolysis device for carbonization. In this process, various non-carbon compounds in it were pyrolyzed. The pyrolysis process was carried out for 1.5 hours at a temperature of 300 °C to 500°C. The temperature change was monitored every 50°C times. An increase in temperature led to a decrease in the efficiency of charcoal formation. Complete carbonization of the raw material was carried out at 400. Based on this, the optimal carbonization temperature was taken as 400.

The samples obtained as a result of pyrolysis at 400 °C were activated in an alkaline (NaOH and KOH) environment for activation. Activation processes In the G'P sample, each reagent was activated in 10%, 20%, 30%, 40% alkaline solutions to determine the optimal conditions for activation in an alkaline environment. The adsorbent samples obtained as a result of activation with NaOH were named G'P-Na1, G'P-Na2, G'P-Na3, G'P-Na4, respectively, and as a result of activation with KOH, G'P-K1, G'P-K2, G'P-K3, G'P-K4, respectively. The adsorbents obtained on the basis of cotton were also activated by the same method and activating agents and were named as follows (PCh-Na1, PCh-Na2, PCh-Na3, PCh-Na4, PCh-K1, PCh-K2, PCh-K3, PCh-K4). The adsorption properties of iodine were studied to determine the most optimal of the resulting adsorbents.

In order to determine the adsorption properties of non-polar substances of adsorbents obtained as a result of all thermal and chemical treatments based on cotton stalk and cotton boll, the adsorption activity of the adsorbents was determined by the iodine number. The iodine number was determined according to State Standard 33618-2015.

To determine the iodine number, a 0,5 g sample of coal was taken, dried at 105 for 2 hours, and then the sample was placed in a conical flask. 50 ml of a standard solution of 0,1 N iodine was poured onto it and mixed with a magnetic stirrer for 30 minutes at a rotation speed of 500 RPM. After mixing, the coal particles adsorbing iodine in the mixture were allowed to settle until they fell to the bottom of the vessel.

First, 10 ml of a standard 0,1 N iodine solution was titrated with 0.1 N sodium thiosulfate. Then, using a pipette, 10 ml of the carbonized iodine solution was taken and titrated with sodium thiosulfate solution. The titration process was carried out until the iodine color turned yellow (straw color). 1 ml of starch was added to it (it is necessary to use a freshly prepared starch solution for the analysis) and the titration was completed when the blue color disappeared.

Accordingly, the adsorption activity of the adsorbents was calculated by the iodine number (based on the formula 2.5). The titration process was carried out 3 times for each adsorbent and their average values were obtained.

Table 1.1. Adsorption activity of adsorbents by iodine number

№	Raw material type	Name of the activated sample	Iodine number, %
1		PCh-T	27 %
2		PCh-Na1	35 %
3		PCh-Na2	41 %
4		**PCh-Na3**	**46 %**
5	Cotton boll	PCh-Na4	47 %
6		PCh-K1	37 %
7		PCh-K2	45 %
8		**PCh-K3**	**51 %**
9		PCh-K4	51 %
10		G‘P-T	29 %
11		G‘P-Na1	37 %
12		G‘P-Na2	44 %
13		**G‘P-Na3**	**49 %**
14	Cotton stalk	G‘P-Na4	49 %
15		G‘P-K1	39 %
16		G‘P-K2	47 %
17		**G‘P-K3**	**53 %**
18		G‘P-K4	54 %

According to the results of the study of the adsorption activity of iodine in the obtained samples, it was found that the samples obtained as a result of thermal and NaOH activation of cotton stalks have 29% in G'P-T, 37% in G'P-Na1, 44% in G'P-Na2, 49% in G'P-Na3, and 49% in G'P-Na4. Therefore, the adsorbent obtained as a result of activation of G'P with 30% NaOH (G'P-Na3) is considered to have the most optimal adsorption activity for iodine. Because the adsorption activity of iodine with the sample (G'P-K4) activated with a 40% solution is almost equal to the adsorbent obtained as a result of activation with 30% NaOH. Therefore, the cost of the adsorbent activated with 30% NaOH for the sample obtained by thermal activation of cotton stalks is slightly lower than that of the adsorbent activated with 40% solution.

CONCLUSION.

In conclusion, today adsorbents are of great importance in water and air purification. It is necessary to find new sources of raw materials and activation methods for the production of adsorbents.

The adsorption activity of adsorbents obtained by thermochemical activation based on cotton stalk and cotton boll was determined by the iodine number.

In the remaining results above (activation of GP with KOH, activation of PCh with NaOH and KOH), the adsorption activity for iodine in adsorbents activated with 30% alkali solutions was also obtained as the most optimal condition. It was also found that, due to the difference in raw materials, the adsorption activity for iodine in cotton stalks was slightly higher than in cotton bolls.

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