Methods of obtaining activated adsorbents based on gossypol tar and local bentonites and analysis of their physical parameters

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Abstract. This article provides information on the methods of obtaining activated adsorbents based on oil-oil waste gossypol tar and local benonites and their various physical properties. In addition, the effect of temperature change on the yield of activated adsorbents from gossypol resin was analyzed. As a result, when Logon bentonite is modified with activated gossypol resin, the hydrophilic properties decrease and the hydrophobic properties increase. Compared to coke obtained from gossypol, it was known that the density of adsorbents obtained as a result of its modification with local bentonite increases. At the end of the study, it was concluded that by adding bentonite as a binder to gossypol resin, its strength increases several times.

1 Introduction

World scientists have provided information on the methods of purification of additional harmful substances in wastewater by oxidation processes (AOP) [1], membrane filtration [2], coagulation [3] and adsorption [4]. AOP and membrane filtration methods are the most effective way to remove harmful substances from wastewater. However, these methods cause oxidation in water due to the formation of high concentrations of additives [5]. The adsorption method is considered the most optimal method for reducing the concentration of additives in wastewater.

Highly effective adsorbents were obtained on the basis of activated carbon, zeolites, clay minerals, non-structured carbon, mesoporous silicon compounds, industrial and local waste [6]. Composites obtained with the help of natural gill minerals and carbon materials are effective methods of cleaning wastewater from harmful additives [7]. carbon-mineral composites are mainly based on obtaining bentonite or kaolin and non-structured carbon [8]; however, when composites are made of activated carbon materials instead of non-structured carbon, its price will decrease several times. In these studies, methods of cleaning organic waste from aqueous solution are shown. Thus, in these studies, the effect of substances on adsorption was studied, but the information about the conditions of adsorption of the processes occurring on the surface was not fully covered. Therefore, there is not enough

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information on obtaining adsorbents by activating natural coal dust, oil-oil waste gossypol and other carbon materials in pilot-industrial works. According to the analysis of the studied literature data, it is possible to conduct research on obtaining activated carbon adsorbent from products containing carbon [9].

2 Method and materials

I chose Gossypol tar from the oil industry during the research. It is possible to obtain carbon adsorbents with high stability from oily waste gossypol. As a result of its additional treatment, i.e. modification with bentonite, adsorbents with the possibility of efficient and fast regeneration can be obtained from it.

Activation refers to increasing the porosity and surface area of carbon storage materials. Physical, chemical and physicochemical methods are used to increase the porosity of carbon storage materials [10].

Physical activation consists in removing additional substances from it using various physical methods (centrifugation, flotation and filtration) without changing the composition of the substance.

Acid, alkali and other substances are often used in chemical activation. In this, the coke is cleaned of various inorganic substances and the coke is activated, as a result of which the porosity and surface area of the coke increases.

Also, coal activation is done by heating to 700-800 ºC using steam or other gases (N_2,CO_2, Ar). For activation in this method, SO_2 or N_2O gases are often used. As a result, additional pores are created due to excess amorphous carbon in the coal structure [10, 11].

The waste oil was pyrolyzed at 400 ºC for 3 hours until degassing to activate gossypol. The yield of carbon adsorbent from gossypol resin was 40-50%. Pyrolyzed coke was activated with 0.1 N hydrochloric acid for 3 hours to remove various additives. After the activation, the coke was washed with distilled water. The activated coke was heated in airless conditions at different temperatures (60 ºC, 70 ºC, 80 ºC and 100 ºC) and the carbon adsorbents of gossypolly were conditionally G-1 at 600 C, G-2 at 700 C, G-3 at 800 C, 1000 C was called G-4.

Coal obtained on the basis of Gossypol tar, selected for modification with adsorbents, was further processed using various methods in order to remove additives from existing Logon and Shorsuv bentonites in Fergana region and increase the sorption properties. For this purpose, the required portion of bentonite is extracted from each soil during preparation for the experiment. Separated bentonite samples are finely ground using a laboratory grinder, passed through a 0.1 mm sieve to separate large sand and rock fragments and crushed residue samples. Then take 1000 g in 20-liter bottles, add distilled water and leave for 6 hours. In this case, bentonite swells under the influence of water, and the resulting suspension is thoroughly mixed. The suspension is cleaned of various water-insoluble additives that have fallen to the bottom. After this process is repeated three times, the bentonite suspension is separated from the water using a centrifuge. After that, it is first dried at room temperature to 30% humidity, and then dried in a drying oven at 100℃ for 4 hours.

Samples of coke obtained on the basis of Gossypol tar and cleaned of various additives were mixed with Logon bentonite (LB) and Shorsuv bentonite (in the ratio of 2/1, 1/1 and 1/2). Add water to the mixture and activate it for 3 hours using a mixer with a heater (Figure 1) adsorbents were obtained. After the activation process was completed, each sample was dried to a moisture content of 30%. The dried samples were converted into granules.

Each adsorbent in the granule state was activated using a pyrolysis device with water vapor at 750-8000C for 30 minutes in airless conditions in order to increase the sorption properties.

3 Results and discussions

The 1st stage of obtaining activated adsorbents from gossypol tar is to obtain carbon gossypol coke by pyrolyzing it at 400 0C and removing various additives in its composition. It consists in activating the obtained coke using various methods (thermal, chemical) and obtaining carbon-mineral adsorbents with double sorption properties in the case of using bentonite as a binder. In order to do this, the intermediate coke product from the gossypol tar to the process of obtaining coal-mineral adsorbents, it is important to determine moisture content % (WA), ash content % (As) and strength of adsorbents (MPa).

The moisture content of the obtained adsorbents was determined using the methodology developed on the basis of GOST 11014-2001, and the ash content was determined on the basis of GOST 11022-95 (Table 1).

From the table above, we can see that the moisture content of gossypol tar is 30%, and the coke obtained from its pyrolysis is in the range of 2-3% in samples activated in airless conditions at different temperatures (700, 800, 1000). Also, the yield of production is little different in adsorbents obtained at temperatures of 400 ^{o}C and 1000 ^{o}C.

Table 1. Physical properties of adsorbents obtained by heat treatment of gossypol.

Samples	The temperature is ^{0}C	Moisture content, %, W^{A}	Ash content, %, A^{s}	Productivity of formation of adsorbents (%)
Gossypol	400	30	12	40.0
Gossypol-based coke	700	3.0	4.2	25.0
	800	2.5	6.2	28.0
	1000	2.0	8.4	30.0

Another important indicator of adsorbents is their strength, as a result of the location of adsorbent and adsorbate molecules on the surface of the adsorbent after absorption of adsorbate molecules, the surface tension of adsorbents decreases and at the same time leads to a decrease in their strength. The strength of the adsorbents after their use is of great importance in separating them from the systems. Therefore, the strength of adsorbents obtained on the basis of Gossypol tar and local bentonitesGOST R 55873-2013was determined using the methodology developed on the basis of The obtained results are presented in Table 2.

Table 2. Strength of adsorbents based on gossypol and local bentonites (MPa).

The name of the sample taken as an object	Adsorbents	Strength of adsorbents (MPa)
Gossypol	G(coke)	4.3
	G-1	5.6
	G-2	5.8
	G-3	5.7
	G-4	5.8
Log bentonite (LB) and G (coke)	GLB-1	7.4
	GLB-2	8.6
	GLB-3	10.5
Absorbent bentonite (LB) and G (coke)	GSHB-1	6.4
	GSHB-2	7.6
	GSHB-3	8.4

It can be seen from the obtained results that compared to coke obtained on the basis of gossypol, the density of adsorbents obtained as a result of its modification with local bentonite increases. For example, the durability is the highest GLB-3 showed a 2.5-fold increase in strength compared to G(cox). From this it can be concluded that as a result of adding bentonite as a binder to gossypol resin, its strength increases several times.

It is important to study the change of water vapor adsorption in modified adsorbents in order to determine the decrease of its hydrophilic properties as a result of modification with log bentonite and industrial waste adsorbents. For this purpose, water vapor adsorption was studied in the obtained adsorbents.

Water vapor adsorption isotherms on carbon-mineral adsorbents obtained by modifying coal adsorbents with Navbahor alkaline bentonite are presented in Figure 1.

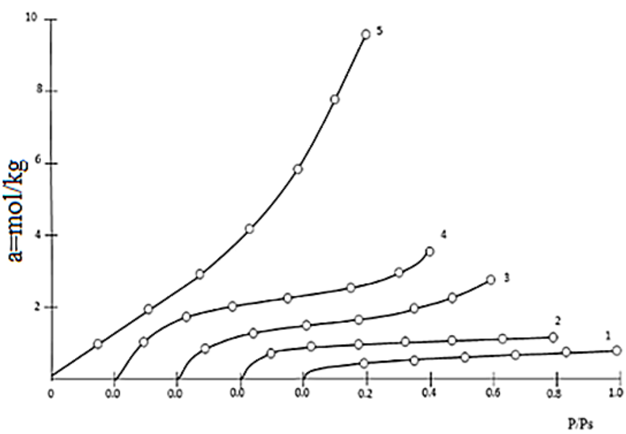

Fig. 1. GLB-1 (1), GLB-2 (2), GSHB-1 (3), GSHB-2 (4). LBK(5) water vapor adsorption isotherm.

In the studied systems, water vapor adsorption isotherms showed ~11 times more adsorption in LBK compared to GLB-1 and GLB-2. Compared to LB, we can see that the adsorption with water vapor is reduced up to 3 times in modified coal-mineral adsorbents GLB-1 and GLB-2. This means that when Logon bentonite is modified with activated gossypol resin, the hydrophilic property decreases and the hydrophobic property increases.

On the basis of water vapor adsorption isotherms in activated and modified adsorbents, the monolayer capacity of adsorbents am, saturation volume as and their relative surfaces S were calculated (Table 3).

Table 3. Structure-sorption indicators of water vapor adsorption on activated and modified adsorbents.

Adsorbents	Single floor capacity, a_m, mol/kg	Comparison surface, S m2/g	Saturation adsorption, a_s, mol/kg
LBK	1.22	79.80	10
GLB-1	0.52	33.96	0.8
GLB-2	0.64	47.70	1.0
GSHB-1	1.20	79.30	2.5
GSHB-1	1.15	76.11	3.5

It can be seen in the table that ~12% of the water vapor adsorption in LB corresponds to the monolayer capacity. In LBK, up to ~3.5 mol/kg of water vapor was absorbed by absorption into the pores, and the remaining part was absorbed by absorption into the adsorbent. The specific surface area (S) of carbon-mineral adsorbents GLB-1 and GLB-2 obtained on the basis of LBK is almost equal to each other. From this it can be concluded that as a result of modification by adding gossypol resin to LB, its hydrophilic properties decreased and hydrophobic properties increased once again.

4 Conclusion

Up to ~12% of water vapor adsorption on LB corresponds to the monolayer capacity. In LBK, up to ~3.5 mol/kg of water vapor was absorbed by absorption into the pores, and the remaining part was absorbed by absorption into the adsorbent. The specific surface area (S) of carbon-mineral adsorbents GLB-1 and GLB-2 obtained on the basis of LBK is almost equal to each other. Water vapor adsorption isotherms revealed ~11 times more adsorption on LBK than GLB-1 and GLB-2. Compared to LB, we can see that the adsorption with water vapor is reduced up to 3 times in modified coal-mineral adsorbents GLB-1 and GLB-2. This means that when Logon bentonite is modified with activated gossypol resin, the hydrophilic property decreases and the hydrophobic property increases. Compared to coke obtained on the basis of gossypol, the density of adsorbents obtained as a result of its modification with local bentonite increases. For example, GLB-3, which has the highest durability, was found to have a 2.5-fold increase in durability compared to G(coke). From this it can be concluded that as a result of adding bentonite as a binder to gossypol resin, its strength increases several times.

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