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THE OBTAINING AND INVESTIGATION OF PHYSICAL AND CHEMICAL PROPERTIES OF CARBON MATERIALS BASED ON POWER-GENERATING RAW MATERIALS RK

Abstract. The method of obtaining activated and impregnated carbon sorbent based on the special coke of the Shubarkol field is described. Elemental and X-ray fluorescence spectroscopic analysis of the raw material was carried out. Physical and chemical characteristics were studied, the specific surface area of the raw material and the obtained carbon materials were determined. It is shown that the specific surface area rises from 14.44 m^2/ g to 361.4 m^2/g when the coke is activated by water vapor, with additional impregnation up to 504.425 m^2/g. The use of coke as a raw material simplifies the technology of obtaining a carbon support, reducing energy consumption and increasing the environmental purity of the process by eliminating the carbonization stage of the coal. The possibility of using impregnated coke for deep cleaning of furnace gas of the phosphorous industry from toxic substances was also investigated.

Key words: active coals, impregnation, power-generating coals, specific surface, phosphine.

Introduction. The phosphorous industry is one of the sources of pollution of atmospheric air of the environment, as in gas emissions contain toxic gases such as phosphine, phosphorus anhydride, hydrogen sulfide, etc. Only in the Zhambyl branch of LLC «Kazphosphate» NDFZ, in the production of 110.0 thousand tons of yellow phosphorus, 4969.36 tons/year of gaseous substances are released into the atmosphere. Specific output of furnace gas at phosphorous plants is 2800-3000 m^3 per 1 ton of phosphorus [1].

The furnace gas of phosphorus production contains about 85-90% carbon monoxide [2], which can be used as a raw material in organic synthesis. However, the use of furnace gas is limited because the furnace gas the content of phosphine that is a potent catalyst poison [3]. Also currently an urgent problem for the phosphorous industry is an unsuccessful system for cleaning gas-dust emissions. The solution of these problems is the use of sorption and catalytic purification methods [4,5]. The advantages of these methods are the ability to remove contaminants to almost any residual concentrations. Moreover, lack of secondary pollution and the controllability of the process, the relatively low cost of construction of sewage treatment plants. Also high removal efficiency of low concentrated contaminants; the small footprint of the unit adsorption purification; the possibility of adsorption of substances in multicomponent mixtures.

Activated carbons are universal adsorbents and supports of catalysts due to their unique properties, high chemical and heat resistance, strength, high sorption capacity in relation to various substances, stability of its structure under the reaction conditions [6,7]. As is known, impregnation of activated carbons with oxides or chlorides of metals creates specific forces on their surface (hydrogen bonding, acid–base interactions or chemical reactions, complex formation, etc.) responsible for chemisorption. Based on the literature data, copper salts with additives of transition and rare-earth metals are the most

frequently used impregnating agents for purification from phosphine [8-12]. Therefore, copper, zinc and chromium salts were chosen as impregnates in this work.

The aim of this work is to obtain and study the physical and chemical properties of the carbon support and catalyst based on the special coke of the Shubarkol field for cleaning the furnace gas of a phosphorous plant from toxic substances such as phosphine, phosphoric anhydride, hydrogen sulphide, etc..

Materials and methods

In the present work, a special coke on the basis of coal "D" of the Shubarkol field was used as the carbon raw material.

Elemental and X-ray fluorescence spectral analysis of raw materials was carried out on the elemental analyzer "Vario Micro Cube", Germany and X-ray fluorescence spectrometer "Focus-2M", Russia, respectively.

Obtaining a carbon support on the basis of the special coke. Special coke on the basis of coal grade " D " Shubarkol field previously crushed to a fraction of 1.5-4 mm, then activated with water vapor at a temperature of 850-950°C. The activation process transforms the carbon material into a form that contains as many randomly distributed pores of various shapes and sizes as possible, thereby increasing the specific surface area of the sorbent [13].

Impregnation of carbon support with the metal salts. The impregnation of the sorbent was carried out with solutions of the following salts in a certain order: $Zn(CH_3COO)_2$, $(NH_4)_2[Cr(C_2O_4)_2]$, $Cu(NH_4)_2[Cu(C_2O_4)_2]$ to obtain the required concentrations of the oxides in the solid residue with further evaporation of the solution. The concentration of oxides in the solid residue was determined by atomic absorption spectroscopy on the spectrometer «AAnalyst 400», Perkin Elmer, Germany. Drying of impregnated sorbent was carried out at 120-140 ^{0}C for 20 minutes, then calcined at 260-295 ^{0}C for 10 hours with a heating rate of 10^{0}C/min in the air at Teflon and steel trays on the muffle furnace SNOL 7,2/1100. As a result, a carbon-metal system with the following content of metal oxides was obtained: CuO 8.3-9.8 mass.%, ZnO 0.4-0.6 of the masses.% , Cr_2O_3 0.9-1.1 mass.% [14].

Humidity was determined by the difference between the masses of the original sample (its mass is ~1 g) and dried sample at 110°C for 1 h in the weighing bottle. Ash was also found by weighing a sample of sorbent with a mass of 1 g, heating it for 2-2.5 hours at 800 ° C. In all cases, three parallel experiments were conducted [15].

When determining the sorption capacity for iodine, preliminary preparation of the sample was carried out, which consist in a 10-minute boiling of 20 g of sample in 200 cm^3 of 0.2N solution of HCl, followed washing it with distilled water and drying for 1 hour at 110 ° C. To determine 1 g of the sample shake 15-30 min with 100 cm^3 0.1N iodine solution in KI (25 g/dm^3), then aliquot (10 cm^3) titrated 0.1 N sodium thiosulfate solution (indicator – starch) [16].

The mass fraction of volatile substances and the total volume of pores is determined by RMG 6382-2001 and RMG 17219-71 [16, 17].

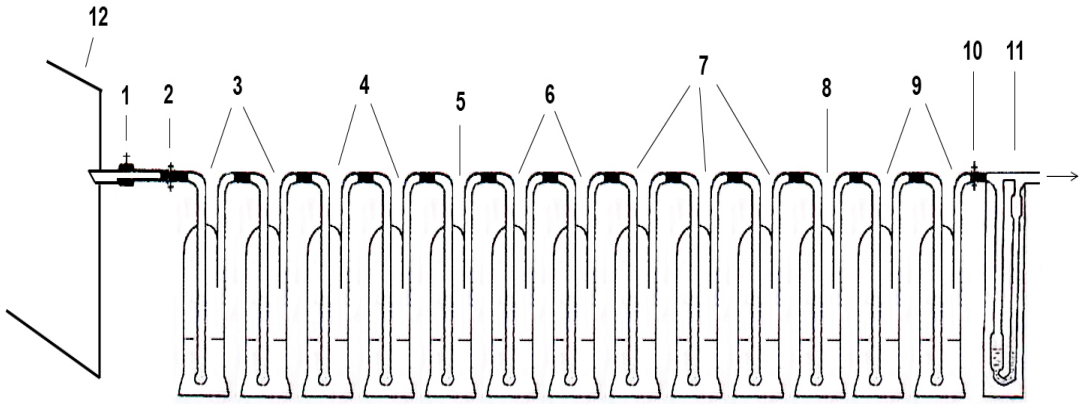
The pH of the aqueous extract was determined by the procedure of [18].

pH of the aqueous extract was determined at 3-minute boiling of 5 g of finely ground sorbent in 50 cm^3 of distilled water with a reverse refrigerator, followed by rapid filtration of the suspension through a paper filter and cooling it before pH measurement [19].

The sorption capacity by methylene blue is determined for a dry sample weighing 1 g, which is in contact with the solution in static mode. The contact time is 24 hours. The sorption capacity E_{mg} of sorbent is calculated from the difference between the concentrations of methylene blue solution before and after the experiment. Analysis of the concentration of methylene blue was carried out on a photoelectrocolorimeter AR-101, Japan [20].

The specific surface area and the total pore volume were determined by the Brunauer–Emmet–Teller (BET) method using the standard procedure based on the data on the measurement of adsorption – desorption isotherms at 77 K using the surface area and pore size analyzer NOVA 3200E (Quantachrome Instruments, USA).

Methodology of sorption purification of furnace gas. The laboratory installation for the purification of furnace gas consisted of a series of connected Drexel flasks, the volumes of which are 50 ml, filled with 5% soda Na_2CO_3 and 25% solution of copper sulfate, benzene to absorb yellow phosphorus, and the flask filled with 207 g of impregnated sorbent (figure 1).



11 - the valve; 2,10 - clamps; 3 - Na_2CO_3 soda solution 5%; 4 - $CuSO_4$ - 25%, 5-8 - absorbers are empty; 6 - absorber with benzene for P_4; 7 - absorber with NaOH solution for P_2O_5 and HF; 9 - impregnated adsorbent; 11 - rheometer; 12 - flue.
Figure 1- Scheme of deep cleaning of furnace gas from PH_3, as well as other associated gases

From the flue is supplied furnace gas, which is passing through the cleaning flasks Drexel undergoes deep cleaning. The flow rate of the furnace gas was controlled with the rheometer and was 1 dm^3/min. The purification tests were carried out for 2.5 hours. Gas samples were taken at the beginning of the experiment and 2 hours after the start of the experiment. The concentrations of P_2O_5 and PH_3 before and after purification were determined by the photocolorimetric method [20,21], and concentrations of the associated gases were determined by the methods of [22-24].

Results and discussion

Visually, the special coke of the Shubarkol field used as a feedstock, solid, has a grayish-black color and a characteristic specific smell (velvet-black color on the fracture of the pieces). Fraction size from 0,1 to 10 mm. Elemental and component composition of the initial coke is presented in tables 1,2. As can be seen from the table, coke is characterized by a high content of carbon - 80.501%, a small amount of sulfur. The oxide composition of coke is dominated by oxides of silicon, aluminum, calcium and iron.

Table 1-Elemental composition of the special coke

Element	Content, %
Carbon	80.501
Hydrogen	3.971
Sulfur	0.054
Nitrogen	not found
Unidentified elements	15.474

Table 2 - Component (oxide) quantitative composition and total sulfur of the original special coke

Component	Content, %
Na_2O	0.01
MgO	0.03
Al_2O_3	1.09
SiO_2	2.14
P_2O_5	0.03
K_2O	0.06
CaO	0.63
TiO_2	0.06
MnO	<0.01
Fe_2O_3	0.14
п.п.п.	95.81
Total	100
S_{total}	0.03

As a result of activation of the special coke with water vapor, the specific surface area increases from 14,443 to 361, 377 m2 / g, and the iodine number increases from 1.78 to 40.47%, this improves the sorption properties due to the burnout of unstructured amorphous carbon (Table 3). The mass fraction of volatile substances and moisture is significantly reduced.

Table 3-Physico-chemical characteristics of the initial and activated coke

№	Name of the indicator	Initial coke	Activated coke
1	Mass fraction of moisture, %	22.24	2.24
2	Mass fraction of ash, %	6.61	6.24
3	Mass fraction of volatile substances,%	9.98	0.1
4	Adsorption activity by iodine, %	1.78	40.47
5	Specific surface area, m^2/g	14.443	361.377

The total pore volume of activated coke is determined, which is equal to 0.59 cm^3/g, and the sorption capacity for methylene blue is 114 mg/g and pH of aqueous extract 7.1. The data obtained indicate that the obtained carbon material is comparable to the known commercial BAU-A sorbent by sorption properties [25].

Impregnation of activated coke with solutions of salts $Zn(CH_3COO)_2$, $(NH_4)_2[Cr(C_2O_4)_2]$, $Cu(NH_4)_2[Cu(C_2O_4)_2]$ increases the sorbent ash content to 19.5-20.0%. Figure 2 shows the isotherm of adsorption and desorption of nitrogen in impregnated coke. The adsorption isotherm is of type I or Langmuir isotherm, inherent for microporous samples with a relatively small outer surface, where the limiting amount of adsorbate depends more on the available volume of micropores [26].

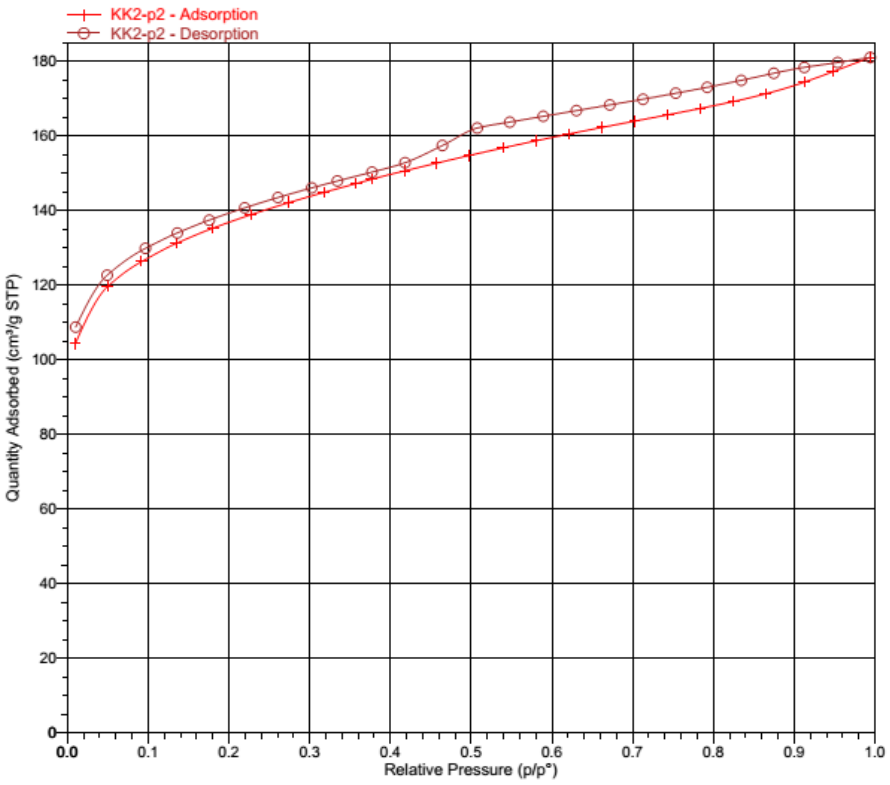


Figure 2 – The isotherm of adsorption and desorption of nitrogen on impregnated coke

The specific surface area determined by the BET method for the nitrogen adsorption isotherm is 504.425 m^2/g, which is 1.4 times greater than the activated coke.

According to the analysis of the pore volume distribution, the sorbent obtained relates to fine-porous sorbents, the volume of mesopores (15-50 nm) is not more than 0.0118 cm^3/g, the volume of micropores (0-15 nm) is 0.1380 cm^3/g, macropores are absent.

In the central factory laboratory of LLC «Kazphosphate» NDFZ, in February 2018, laboratory tests were carried out on the technology of deep cleaning of furnace gas adopted at the plant using coke impregnated with salts of active metals. The purpose of the tests was to establish the possibility of using the developed adsorbent for deep purification of the furnace gas from phosphine and other associated gases. The average composition of the furnace gas is shown in Table 4.

Table 4 - Average composition of furnace gas of LLC «Kazphosphate» NDFZ

The composition of furnace gas										
P_4, мг/м^3	P_2O_5, мг/м^3	PH_3, мг/м^3	F, мг/м^3	S_{total}, мг/м^3	CO_2,% (об.)	PH_3,% (об.)	O_2, % (об.)	CO,% (об.)	H_2, % (об.)	CH_4, % (об.)
180	180	770	5,2	430	0,6	0,2	2,0	65,5	1,3	0,4

As a result of the studies, it was found that impregnated coke exhibits a high degree of purification with respect to phosphine, phosphoric anhydride and concomitant gases (HF, H_2S) throughout the experiment, and poorly adsorbs SO_2 (Table 5).

Table 5 – test Results for cleaning of furnace gas LLC «Kazphosphate» NDFZ

Date	Sampling point	Defined components	Before cleaning mg/m^3	After cleaning mg/m^3	the Purification efficiency,%	Temperature of sampling
16.02.18 1 sampling (at the beginning of the experiment)	the furnace №6 SUPG	P_4 P_2O_5 PH_3 HF H_2S SO_2	351,522 804,985 1497,415 Следы 488,225 917,863	traces traces 3,475 traces traces 734,291	100 100 99,8 100 100 20,0	27^{o}C
2 sampling (after 2 hours)	the furnace №6 SUPG	P_4 P_2O_5 PH_3 HF H_2S SO_2	277,066 634,481 886,787 Следы 494,761 930,151	traces traces 15,687 traces traces 930,151	10 0 100 98,2 100 100 –	31 ^{o}C

Conclusion. As a result of the work, the sorbent activated and impregnated with salts of metals was obtained on the basis of the special coke of the Shubarkol field and their physical and chemical properties were determined. It is noted that the impregnated sorbent obtained has a fine-porous structure and a high specific surface area. The results of the study showed that the impregnated adsorbent exhibits high sorption characteristics in the purification of furnace gas LLC «Kazphosphate» NDFZ. The resulting carbon catalyst is a promising adsorbent for deep purification of furnace gases of the phosphorous industry and is recommended for research in semi-industrial and industrial conditions.

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