STUDY OF ADSORPTION OF HIGH-SILICA ADSORBENTS OBTAINED FROM NAVBAHOR BENTONITE

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Abstract. Silicon-based adsorbents (SBA) were synthesized from Navbahor bentonite using sol-gel technology with the participation of various organic compounds (templates). For this, crystals were formed by adding hexamethylenediamine and alcohol fraction as templates to the liquid glass (29% SiO_2, 9% Na_2O, 62% H_2O) and $Al(NO_3)_3 \cdot 9H_2O$. After the crystallization process was completed, the resulting solid phase was separated from the solution using a Büchner funnel, dried in a SShU-M1 dryer at 120 °C, and calcined for 8 hours in a SNOL 30/1100 muffle furnace at 550 °C to remove the template. The crystallization of Navbahor bentonite lasted 8–9 hours. The element and oxide composition (mass %) of the obtained SBA samples was studied by X-ray fluorescence, and the thermal behavior of bentonite was studied by derivatographic analysis.

Keywords: Sol-gel, template, bentonite, derivatography, silicate module, hexamethylenediamine, crystallization, X-ray fluorescence.

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Аннотация. Синтезированы кремнийсодержащие адсорбенты (СКА) из навбахорского бентонита с использованием золь-гель технологии с участием различных органических соединений (темплатов). Для этого формировались кристаллы путем добавления в жидкое стекло (29% SiO_2, 9% Na_2O, 62% H_2O) и $Al(NO_3)_3{\cdot}9H_2O$) в качестве темплатов гексаметилендиамина и спиртовой фракции. После завершения процесса кристаллизации образовавшуюся твердую фазу отделяли от раствора на воронке Бюхнера, высушивали в сушилке СШУ-М1 при 120 °С и прокаливали в течение 8 часов в муфельной печи SNOL 30/1100 при 550 °С для удаления темплата. Кристаллизация навбахорского бентонита длилась 8–9 часов. Элементный и оксидный состав (мас. %) полученных образцов SBA.

Ключевые слова: Золь-гель, темплат, бентонит, дериватография, силикатный модуль, гексаметилендиамин, кристаллизация, рентгенофлуоресцентный анализ.

Introduction. In the chemical industry, adsorption methods are the most common in the purification of hydrocarbons from various mixtures and sulfur compounds, and the use of these methods allows to return to production a number of valuable compounds. Adsorbents are widely used in petrochemicals as sorbents and catalysts in the processing of oil, natural gas, and petroleum-associated gases, in the separation and purification of liquid and gaseous media[1,2]. In recent years, natural and artificial adsorbents have been widely used in the treatment of hydrocarbon raw materials [3].

Currently, one of the most important topical directions is the creation of environmentally safe sorbents, retainers and catalysts based on local raw materials [4].

Today, the world is paying great attention to the creation of waste-free or low-waste, energy and resource-efficient technologies. In successfully solving these problems , the level of purity of the substances used and produced for the technological process is important. The most important requirements for adsorbent materials are: high specific surface area, selectivity and easy regeneration. It is also necessary for the adsorbent to be cheap and harmless, to be able to maintain its adsorption properties for a long time, and to have high mechanical strength [6].

Among the sorbents used in adsorption processes and catalysis, adsorbents have a special place with acid tolerance, thermostability and acidity properties[7]. Today, the main problem in the production of adsorbents is to reduce their cost and simplify the synthesis technology, and extensive scientific research is being conducted in this priority direction[8-10]. Today, among the technologies for purifying oil and natural gas from water vapors and sulfur compounds, adsorption processes of drying and purification using adsorbents with high absorption property at low partial pressure of mixtures, selectivity of adsorption of polar substances, etc. occupy an important place [11].

Experimental part High silica adsorbents (YuKA) were synthesized in a stainless steel autoclave at 175-200 ° C for 6-8 days according to the following method.

was prepared by adding hexamethylenediamine and alcohol fraction as a template to liquid glass (29% SiO_2, 9% Na_2O, 62% H_2O) with rapid mixing. The value of the $Al(NO_3)_3 \cdot 9H_2O$reaction mixture was monitored by adding a pH0.1 N HNO_3 solution to it. Navbahor bentonite was added to the resulting mixture. After the completion of the crystallization process, the solid phase was separated from the solution using a Buchner funnel and dried for 12 hours in a ShSU-m1 drying cabinet at 120 ° C and fired in a SNOL 30/1100 muffle furnace at 500-550 ° C for 8 hours to remove the template.

Colloidal sol consists of macroparticles (solid dispersed phase particles).

100 g of 25% ammonium chloride was added to 10 g of adsorbents for decationization of the obtained high silicon adsorbent. The solution was kept in a water bath at 90-100 ° C with constant stirring for 2 hours, then the precipitated NH_4^+ /adsorbent was filtered, washed with distilled water, dried and calcined at 550 ° C for 8 hours. The decationized sorbent powder was then pressed into tablets and obtained as granules. Catalysts with modified adsorbents are prepared by absorbing certain salts or acids into the adsorbents. 50 hours at 115 ° C to form powdered YuKA with 100% crystallinity , at 150 ° C and about 10 hours is enough time.

In Figure 2

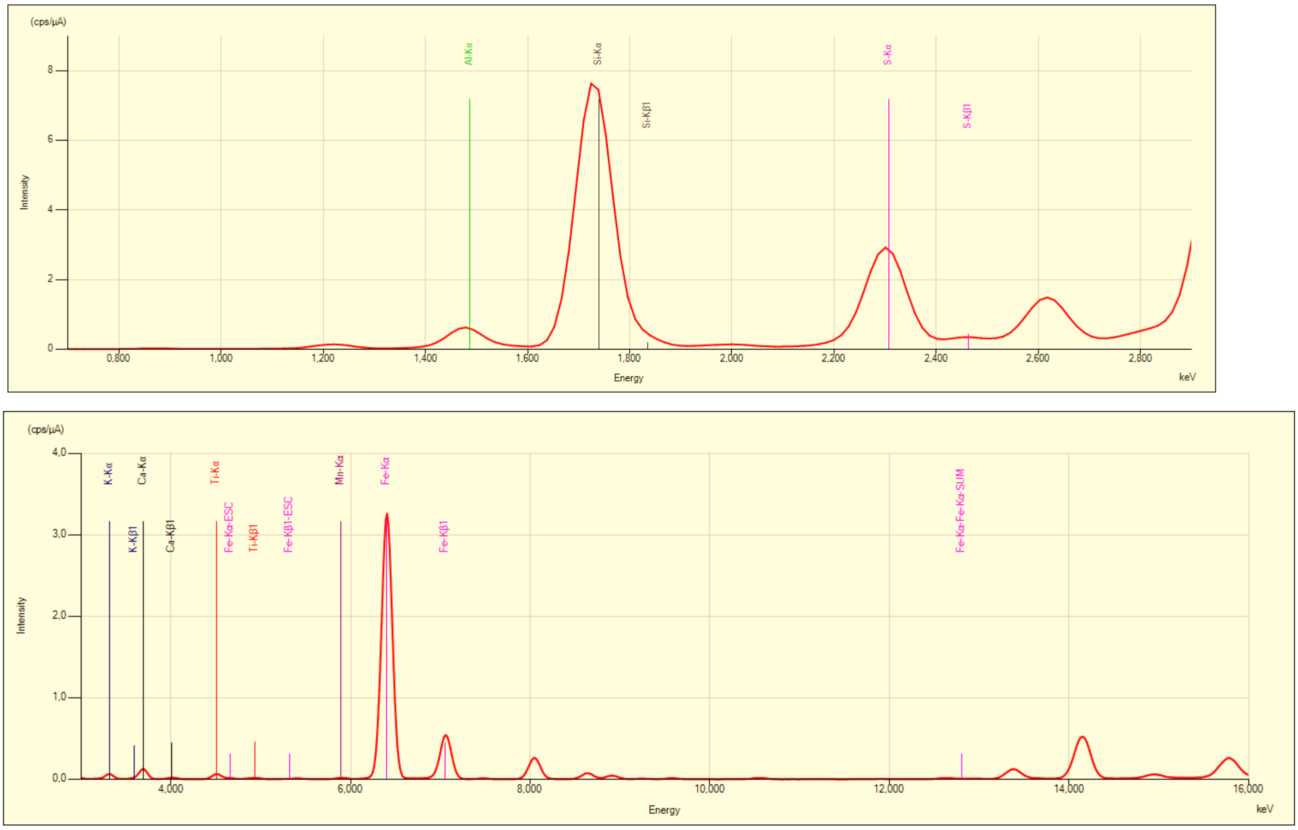

Figure 2. a) 6; b) 7 ; X-ray fluorescence results of YuKA samples obtained after crystallization for 9 hours

Figure 2 shows X-ray images of the synthesized samples after crystallization for 6.7 hours. It can be seen from the radiographs that when crystallization is carried out for 6, 7 hours, the amount of adsorbents in them is 50, 96 (respectively).

Navbahor district of bentonite chemical composition and structural characteristics are listed in Table 1.

Table 1

Name	SiO_2	TiO_2	Al_2O_3	Fe_2O_3	MgO	CaO	Na_2O	K_2O	P_2O_5	SO_3
Alkaline bentonite soil wt.%	57.91	0.35	13.69	5.10	1.84	0.48	1.53	1.75	0.43	0.75
Alkaline-earth bentonite soil wt.%	56.23	0.61	13.56	6.50	3.76	0.69	0.98	2.20	0.92	0.49

In order to study the chemical, physico-chemical and structural characteristics of bentonite, we placed 100 g of bentonite granules in a 250 cm ^{3} glass flask and added 150 cm ^{3} of distilled water. Flask **PE-6410** for 24 hours mixed in the device at a speed of 120. We added heated 40 ml to ground soil $H_2SO_4$and heated with stirring in a water bath. After the treatment, the soil was filtered with a paper filter in a Buchner funnel and washed again with distilled water until pH=5.4-5.7. Bentonite together with filter paper was dried in a drying cabinet at 120°C for 5 hours.

Table 2

Acidic from activation then , sample mass %:

Name	**SiO_2**	**TiO_2**	**A l_2O_3**	**F e_2O_3**	**MgO**	**Ca O**	**Na_2O**	**K $_2O$**	**Mn O**
Alkaline, Alkaline-earth bentonite soil mass.%	70.17	1.63	9.49	1.39	0.64	0.20	0.17	1.27	0.01

2 - As can be seen from the table, the silicate modulus of the bentonite sample by acid treatment is 7:1 increased to go to fight can _

The analysis of bentonite derivatographic curves was carried out (Fig. 3) shows that TG thermal logarithmic gravimeter lines are depicted, it is noted that the decrease in the mass of bentonite in the initial definite time interval over a certain period of time, DTG - differential thermal gravimeter temperature curves are depicted.

The resulting derivativeogram is presented in the figure, which consists of 4 curves. Analysis of the dynamic thermogravimetric analysis (DTGA) curve (Curve 2) shows that the DTGA curve mainly occurs in the 2 intensive decomposition temperature ranges. The 1st decomposing interval corresponds to the temperature of 106-294 ^{0} C , and the 2nd decomposing interval corresponds to the temperature of 302-950 ^{0}C.

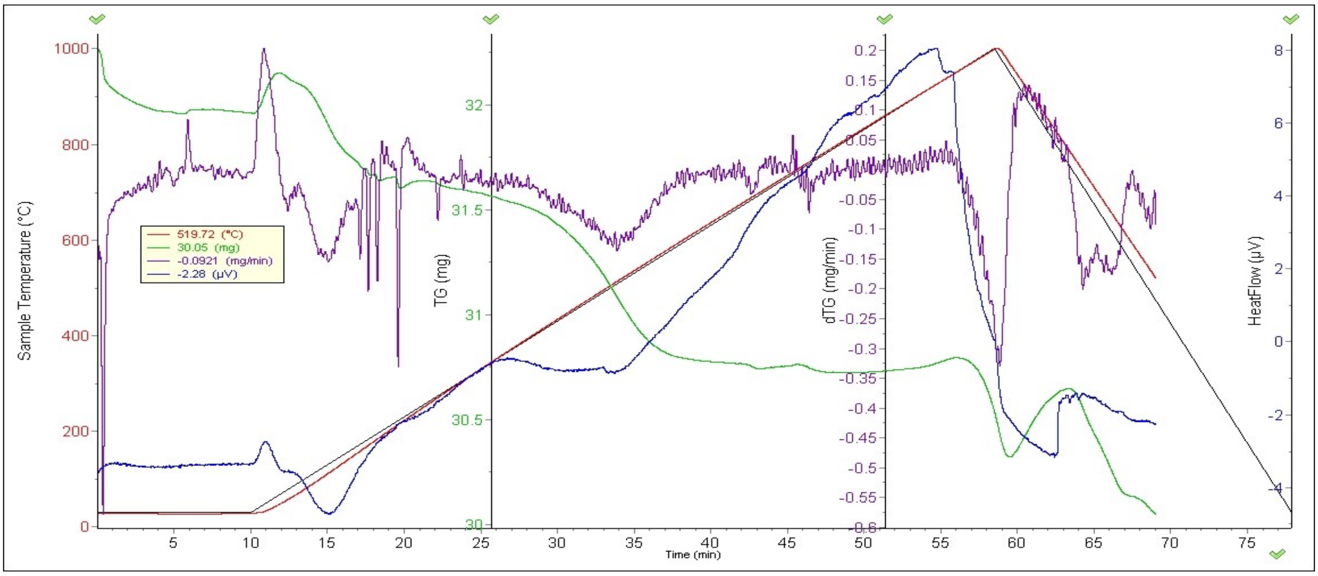

Figure 3. Activated bentonite derivativeogram

1-Temperature curve; 2- dynamic thermogravimetric analysis curve line (DTGA); 3- dynamic thermogravimetric analysis curve derivative (DTGP); 4- DSK curve.

Analysis shows that intensive decay occurs in the 2nd decay interval. In this interval, the amount of decomposition, i.e. 5.8% of the decomposition takes place.

detailed analysis of the dynamic thermogravimetric analysis curve and DSK curve is given in Table 3 below.

Table 3

Bentonite is active Analysis of DTGA and DSK curve results

No	Temperature , °C	Mass loss, %	of substance decay speed , mg/min	Amount of energy consumed (µ V * s / mg))
1	50	0.225	0.136	1.46
2	100	0.885	0.555	2.54
3	200	1,055	0.653	2.21
4	300	1,135	0.487	3.22
5	400	1,685	1,147	2.02
6	500	2,069	0.455	3.03
7	600	1,815	1,499	1.65
8	700	1,521	2,156	1.21

9	800	1,812	1,244	1.77
10	900	1,958	2,622	2.02
11	1000	2,041	1,235	2.25

This derivatograph As a result of these efforts, it appears that the main mass loss takes place in the range of 160-912 o ^{C} , in which 7.25% of the main mass, i.e. 3.21 mg of the mass, is lost.

Bentonites are small crystals of medium weight and powder with a scattering index. According to the adsorbent description, bentonite is an adsorbent consisting of a combination of meso-macro-microporosity, where mesopores make up the majority. Since the specific surface area is an average description of the internal pore size, its high value is determined by the average pore size, which is 4.9 nm for bentonite clays. [176; 24. - p. 57-61] . In conclusion, the swelling, colloidal, sorption ability of Navbahor bentonite to water and petroleum gases was studied.

Conclusions. Navbahor bentonite was obtained from clay soil, chemically treated, and high silicon adsorbents (YuKA) were prepared. A number of technological processes were implemented for the preparation of YuKA.

High silicon adsorbents were synthesized in a stainless steel autoclave at 175-200°C for 6 days according to the following method.

was prepared by adding hexamethylenediamine and alcohol fraction as a template to liquid glass (29% SiO_2, 9% Na_2O, 62% H_2O) with rapid mixing. $Al(NO_3)_3 \cdot 9H_2O$After the process was completed, the solid phase was separated from the solution using a Buchner funnel and dried at 120°C for 12 hours and calcined at 500-550°C for 8 hours to remove the template.

For decationization of the high silica adsorbent obtained, it was treated by adding 25% ammonium chloride and washed several times in distilled water, then calcined at 550-600°C for 8 hours. The decationized adsorbent powder was then pressed into tablets and granulated.

The chemical composition of synthesized YuKA samples, mass %, changes upon heating and structural characteristics of adsorbents (total specific surface area and volume of pores, surface-surface area corresponding to micro- and mesopores, size, crystallization conditions and physico-chemical properties of the samples) were studied based on the table information is provided.

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