



STUDY OF BENTONITE-SORBENT ISOTHERM

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Abstract:

In this study, the retention substance bentonite was used in the preparation of a mesoporous silica nanocatalyst. The analysis of sorbents from the sorption isotherm study and the results obtained were summarized. Also discussed are the chemical composition of bentonite from the Navbahor deposit, the types of sorbent isotherms obtained by the adsorption of various gases on sorbents, descriptions of sorbent isotherms in mesoporous sorbents, the phase composition of sorbent samples synthesized from textures, and the physic-chemical characteristics of the X-ray diffractometric catalyst, and the dependence of the characteristics of textures of the characteristics of nanocomposite sorbents made from bentonite

Keywords: mesoporous, silica nanocatalyst, bentonite, zeolite with a high silicon content, catalyst, texture, sorbent, sorption, adsorption.

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Introduction

In the world, silica materials and composite sorbents obtained on their basis are widely used in various fields of industry due to their unique properties, including high chemical, thermal, and mechanical stability, low toxicity, sorption properties, and the manifestation of catalytic inertia. It is distinguished by its structure and surface morphology characteristics, which include high comparative surface porosity with nanometer size and specific physico-chemical properties.[1,2.]. Silica nanomaterials, whose typical size has a porosity of 0.4 to 50 nm and has a large number of hydroxyl groups in their composition, are of great importance for the media and pharmacology [3]. This type of porous nanomaterial is widely used in the transportation of medicines of various functional groups (contingents), as well as in their chromatographic analysis, in the immobilization of enzymes, and as

a binder for some viruses. Modified organic/inorganic hybrid nanosorbents with micro- and mesoporous substances of various functional groups in their composition are of practical importance for the separation and analysis of samples used in chromatography of liquids and ion exchange. It is known that the synthesis of silica materials is important not only for practical purposes but also for the development of important theoretical studies [4].

The geometric shape and pore sizes determine the mechanism of the adsorption process and the shape of the sorption isotherms. Cylindrical pores are usually found in aluminum and magnesium oxides, prismatic pores -in fibrous zeolites, ellipsoid pores -in soil and activated carbon, and pores between ordered spherical particles in silica gel [5].

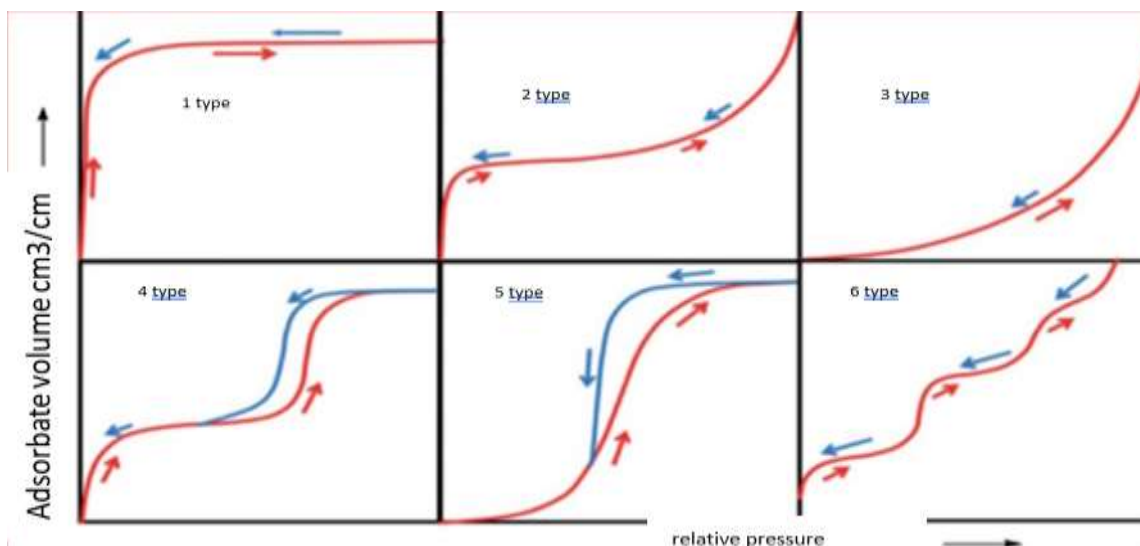


Figure 1. Types of sorption isotherms obtained by adsorption of various gases onto sorbents

According to the size of adsorbents, according to the IUPAC classification, the porosity size is divided into the following classes: macroporous $d > 50$ nm, mesoporous $d = 2 \div 50$ nm, microporous $d = 200 \div 400$ nm, mesoporous $200 \div 400 > d > 3 \div 3,2$ nm, microporous $d < 1.2 \div 1.4$ nm, supermicroporous $3 \div 3.2 > d > 1.2 \div 1.4$ [6].

Sorption isotherms obtained as a result of the adsorption of various gases onto adsorbents are divided into six types according to the Brunauer classification (Brunauer-Deming and Deming-Tellurium classification) [7]. The classification of sorption isotherms is shown in Figure 1:

Type I sorption isotherm-at low relative pressures ($p/p_0=1$), the greatest adsorption occurs (the so-called Langmuir isotherms).

Type II - This sorption isotherm is characteristic of macroporous adsorbents and is a free mono- or polymolecular adsorption.

Type III-This type of isotherm is usually rare and represents the absence of isotherm points associated with increased interaction between the adsorbate and the adsorbent.

This isotherm is formed from adsorption occurring due to capillary condensation in mesoporous bodies, which is single-layer adsorption. Due to residual pressure ($p/p_0=0,4$), the adsorption and desorption isotherms overlap and form a hysteresis ring.

Type V-double characteristic type III has no such turning points as an isotherm. Meso, like the type IV isotherm, forms hysteresis rings due to capillary condensation in the pores.

Type VI-sorption isotherm, however, arises due to gradual polymolecular adsorption due to the weak interaction between the adsorbate and adsorbent molecules [8].

Therefore, the analysis of the sorption isotherm of bentonite, a sorbent used in the preparation of mesoporous silica nanocatalyst, is relevant for the study and determination of its main characteristics.

Methods

Dilatometry is a device that allows you to measure the change in gel volume depending on time, the results obtained allow you to study the kinetics of gel formation.

X-ray diffractometry (XRD). The phase composition of sorbent samples synthesized at different temperatures has been studied by X-ray diffractometry. The X-ray diffractometer (XRD) of Empeyan, Malvern Panalytical (Germany) was used for the X-ray analysis of the samples. The Emugeap Malvern panalytic X-ray diffractometer performs complex functions, such as performing quantitative phase analysis in phase transfer mode using a wide dynamic range of hybrid architectures Pixel3D and Galipix3D, evaluating amorphous components, determining clusters and nanocomposite sizes.

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Quantitative and qualitative phase analysis: to study the phase composition of sorbents, CuK α radiation was used (β -filter, Cu, current mode 1.5406 A ° and voltage supplied to the pipe, respectively, 30 mA and 30 kV), with a diode step of 0.02° in 4 °/min, with a constant rotation speed (corresponding to $\omega/2 \theta$). And the scanning angle was changed from 0° to 90°. When recording experiments, a rotating camera was used, the rotation speed of which was 30 rpm. The phase composition of the sorbent samples was analyzed using X-ray diffractograms by a semi-quantitative method in which the intensity coefficients based on calibration standards were performed in accordance with the Cu radiation based on the I/Ic ratio.

Scanning electron microscopy. The surface morphology and pore sizes of mesoporous nanocomposite sorbents were studied by scanning electron microscopy. The analysis was carried out using a scanning electron microscope (SEM) EVO MA 10 (Carl Zeiss, Germany) equipped with an energy dispersed X-ray spectrometer device (EDS Aztek Energy Advantag X-Act, Oxford Instruments). The samples were first coated with a 14 nm thick carbon powder and kept for 2 hours under vacuum conditions. The accelerating voltage on the device is 20 kV, the current of the electron probe is 1.4 na, the spectrum summation time is 100 seconds, and the intensity is 27 thousand pulses. The process was carried out through the detector (SE2) and the reverse electron flow (ESB).

Microrentgen (EDS) analysis. Elemental analysis of sorbents was carried out using the SEM EVO MA 10 detector (Carl Zeiss, Germany), additionally connected to a scanning electron microscope (EDS Aztek Energy Advantag X-Act, Oxford Instruments). During the analysis, the accelerating voltage on the device is 20 kV, the current of the electronic probe is 1.4 na, the time of summation of the spectrum is 100 seconds, and the intensity is 27 thousand pulses per sec. Through the reflecting detector (SE2) and the reverse electron flow (ESB), the process was carried out in 10 seconds. IR spectroscopy.

The IR spectra of the samples were obtained using Fourier transform IR spectroscopy in the range of 400–4000 cm⁻¹ on the Brooker Tensor 27 instrument. At the same time, the

accuracy of the wavenumber was 0.01%, the scanning speed was 1.4-12.7 mm/s, and the sample quantum size was 25.5 x 27x16 cm. During the analysis, the composite mixture was prepared as follows: it consists of 95% CBD and 5% of the sample mixture. The samples were studied by diffuse reflection and the obtained spectra were processed using the OMNIC program.

Mesoporous for the study of sorption isotherms in sorbents. Benzene for sorbent samples taken at different temperatures (saturated steam at 298 K $p_0 = 74.7$ mmHg), n-hexane (saturated steam $p_0 = 150$ mmHg). Toluene (saturated steam $p_0 = 25.5$ mm. Mercury column) and water vapor (saturated vapor $p_0 = 23.75$ mmHg) adsorption was studied using a sensitive quartz spiral device, the Mc-Bain-Bakr.

Processing of information about adsorption. Using the adsorption data of various adsorbents on sorbents, their textural characteristics were calculated. The sample surface (SBET, m²/g), average porosity diameter (D, nm), porosity size (Vs), monolayer throughput (G_∞) and saturation adsorption (Gs) were calculated using the following methods.

Results and Discussion

Some physical dimensions of substances obtained as adsorbates are measured by various methods. [9] when determining the adsorption data of the adsorbate: benzene was initially purified under vacuum conditions, its vapors were frozen until they became identical to the vapor data for pure benzene. Then the benzene was dissolved, and the gases dissolved in it were isolated. Before measuring the adsorption of benzene vapors in adsorbents in a sensitive quartz spiral McBain-Bakr device under static conditions, the residual pressure in each adsorption system was evacuated to 1.33×10^{-3} Pa and adsorption isotherms were obtained at 298 K. At the same time, the measurement error did not exceed 0.05 mmol/g. The adsorption of toluene and n-hexane on sorbent samples was also carried out in the above order.

Using the adsorption data of various adsorbents on sorbents, their textural characteristics were calculated using the following methods: comparative surface surface (SBET,

m²/g), average porosity diameter (D,nm), porosity size (V_s), monolayer capacity (G_∞) and saturation adsorption (G_s) [10]/

Calculation of the comparative surface area of sorbent samples. Adsorption isotherm models are mainly used to quantify the adsorption of gases on solid adsorbents. In most cases, when determining the comparative surface area of sorbents based on the results of adsorption, the value of the monolayer capacity is used. [11].

$$S = \Gamma_{\infty} \cdot NA \cdot \omega$$

Here, S-comparative surface area of adsorbents (m² /g), - Avogadro Constant, Γ_∞- monolayer adsorbent capacity, ω- the area occupied by one adsorbate molecule in a monolayer (nm²).

For a nitrogen molecule, this number is 0.162 nm², and for benzene, - 0.49 nm². The best isotherm model. The BET method (Brunauer, Emmet, and Teller) is a standard method for measuring the comparative surface purity of porous materials. The linear form of the equation was used to determine the comparative surface area of sorbent samples based on the adsorption isomer. [12].

$$p/p_s / (G (1 - p/p_s)) = 1/G_{\infty} C + C - 1/G_{\infty} C \cdot p/p_s$$

$$\text{from this } 1/(G (1 - p/p_s)) = 1/G_{\infty} C + C - 1/G_{\infty} C \cdot p/p_s$$

Here p/p_s – relative vapor pressure of adsorbate, G_∞ – monolayer capacity,

G -the amount of adsorbed substance at the same pressure (mmol/g)C – a constant value associated with the heat of adsorption and condensation and temperature.

On the basis of experimental data obtained for p/p_s/G (1-p/p_s) and p/p_s connection, a graph of the value of the linear coordinates is constructed. From the graph obtained using linear coupling, the value of 1/G_∞S from the ordinate axis of the cross-section is obtained, while the value of C-1/Γ_∞C is obtained on the straight line of the tangent angle (Figure 8).

Based on these data, the comparative surface size (SBET) is determined by the values of G_∞ and S. Usually, the linear dependence in the BET equation gives a good result in the equation when the relative pressure is 0,5÷1. Consequently, the exact results of the BET equation are observed in type II and type IV isotherms, while deviations from the exact values are observed in type III and V isotherms [13].

Bentonite in Navoi region of Navbahor district is a light gray powder, odorless, practically insoluble in water and organic solvents. The pH value of the suspension is 7.1-8.7. The weak alkalinity of the suspension is explained by the presence of hydroxide soil and hydroxide metals in the clay. According to the adsorption properties, it is a combined meso-macro-micro porous adsorbent. Its specific surface area is 54.5 m² / g, the volume of porosity is 0.065 cm³/g, the average porosity is 4.8 nm. The adsorption activity for blue methylene is 62.0 bentonite/g. This is confirmed by a number of literature data. According to its technological properties, it is a fine powder of medium weight with an average yield index. For use in the chemical industry, standardization of bentonite clay is carried out. Standardization of bentonite was carried out according to the following indicators: description, pH of the aqueous suspension, adsorption activity during drying, cation exchange ability, heavy metals (meringue), comparative surface area, size, and average porosity. Technological and adsorption standardized characteristics of bentonite clay "Navbahor" are presented in Table 1.

It is known that the Langmuir adsorption isotope model is mainly used to quantify the adsorption of gases in solid adsorbents. Polymolecular adsorption on microporous monomolecular adsorption, mesh, and macroporous polymolecular adsorption is used to estimate the monolayer capacity (G_∞) and the specific surface size of sorbents. Based on experimental data, the value of the inverse sign of the Langmuir equation was used to determine the dependence of adsorbents on the pressure of the mono-chain (GM).

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Table 1
Technological and adsorption characteristics of bentonite clay "Navbahor"

No n/n	Quality indicators of bentonite clay	Features of bentonite clay
1	Description	light gray powder, odorless, almost insoluble in water and organic solvents
2	pH suspension (5 v 100) in water	7,1-8,7
3	Weight loss during drying,%	No more than 8%
4	Adsorbtion activity, bentonite / g	62,0±0,2
5	Cation exchange capacity of bentonite	19,4
6	Meringue	No
7	The ratio of Si ⁺⁴ and Al ⁺³ elements	3:1
8	Specificsurfaceareaaccordingtothefive-pointBETmethod, m ² /g	54,5±2,0
9	R/R ₀ = 0.98, the volume of the porous at a pressure of cm ³ /g	0,065±0,005
10	Average pore size, in nm	4,8
11	Moisture content in bentonite clay, %	26 -28
12	moisture content in dry bentonite clay, %	2-3

Table 2
Crimnezem sorbents dependence of texture characteristics on TEOS concentration (50° C)

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TEOS, mol/l	S _{bet} M ² /g	V, cm ³ /g	D, nm	Γ _s , mol /kg	p,g/cm ³
0,04	316,5±45,3	0,44±0,05	10,4±0,8	3,52±0,6	0,45±0,02
0,06	532,6±51,6	0,40±0,11	8,5±0,4	3,98±0,7	0,31±0,03
0,08	952,5±32,6	0,32±0,11	2,4±0,3	4,85±0,3	0,12±0,06
0,1	886,3±20,6	0,36±0,09	6,5±0,5	4,76±0,2	0,19±0,06
0,2	943,2±52,0	0,34±0,02	2,2±0,6	5,12±0,8	0,15±0,02

From the information presented in Table 2, it can be seen that when texturing silica sorbents, with an increase in the concentration of TEOS (at a temperature of 50°C) from 0.04 to 0.2, the porosity surface decreases by 3 times, and the average diameter by 5.

During the Zol-gel synthesis of silica sorbents, an increase in the concentration of reagents in solution was observed, which led to a change in the size of the comparative surface, the size of pores, and their average diameter, which were considered important textural characteristics of sorbents. It was found that the increased concentrations of Teos and SFM led to an increase in the porosity of the synthesized sorbent samples. This, in turn, led to an increase in sorption capacity in the studied systems.

As is known, the adsorption and desorption networks of adsorption isomers of the vapor adsorption equilibrium of various substances in mesoporous silica materials, such as MSM-41 and SBA-15, go together with the formation of a parallel hysteresis ring. Usually, the adsorption of water vapor on mesoporous silica materials depends on the number of silanol and siloxane groups on the surface. In addition, due to capillary condensation, a long stay of adsorbed water vapor inside the pores of the mesh leads to the formation of a large relative pressure from the hysteresis rings. The distribution of pores on the surface of nanocomposite sorbents at 50°C and nanocomposites with a load of 2.0 obtained from Navbahor bentonite obtained in a medium of 10.2 and 1 solution can be seen from images obtained using medium-diameter SEM (Figure 4).

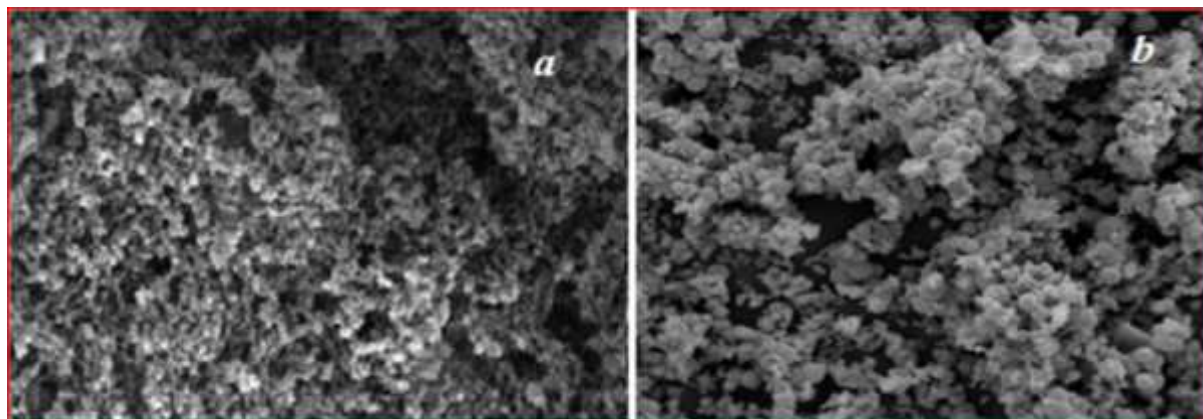
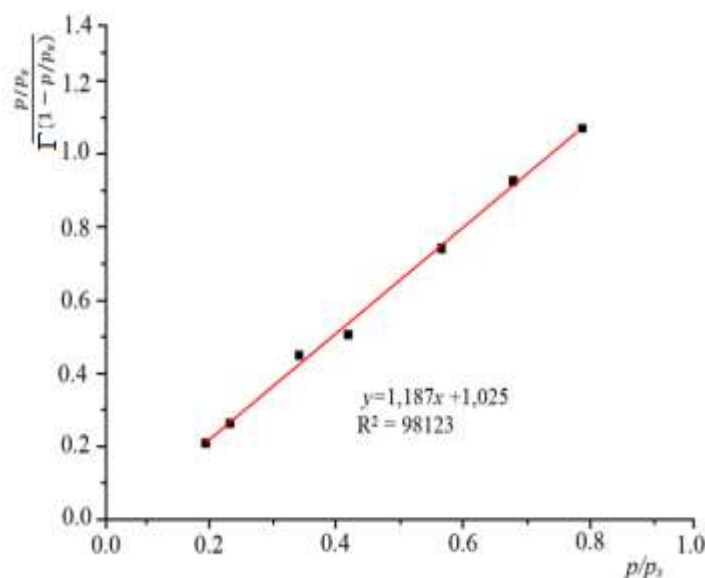


Figure - 4. At 50°C, PH=2.0 on the PEG-400 template (a) and pH=10.2 on SF ni on ni (b), the image of the surface of sorbents with applied loads is ЮКЦ-1

It can be seen from the image that the sorbent pores obtained in the presence of templant PEG-400 are evenly distributed over the surface and have the same dimensions, while the surface of

the sorbents obtained in the presence of GMDA consists of nanoparticles with a monodisperse spherical morphology.



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Figure 6. Values obtained from the sorption of benzene vapors in mesogovacs $p/p_s/(\Gamma(1-p/p_s)) = f(p/p_s)$ correspondence to the linear equation Bet based on the graph $f(p/p_s)$.

In adsorbents obtained in alkaline media, it was observed that the porosity size was 2.25 times smaller than in a porous sorbent obtained in acidic media, while the surface surface comparable to the aging surface was 1.15 times larger. The compatibility of isotherms obtained as a result of adsorption of benzene vapors at different relative pressures with the linear form of the BET equation was studied.

$$p/p_s/(\Gamma(1 - p/p_s)) = 1/\Gamma_m \cdot C + C - 1/\Gamma_m \cdot C \cdot (p/p_s)$$

Using different relative pressure (p/p_s), the sorbed amount of adsorbate $p/p_s/(\Gamma(1-p/p_s)) = f(p/p_s)$ graph $f(p/p_s)$ has been drawn up (Fig.6). The figures show that the sorbent samples taken at 50 °C correspond to the linear form of the beta model of the benzene vapor adsorption Isotherms at different relative pressures. Calculations were performed on the basis of the adsorbed amount of benzene within the relative pressure $p/p_s=0.2 \div 0.8$. It was found that the isotherm of the sorbent

sample obtained at pH = 2.0 has a degree of compatibility $R^2 = 98.12$, and in the sorbent obtained in an alkaline medium - 92.7%, which corresponds to the isotherm model. Also, based on the data obtained as a result of the experiment,

sorbents were determined by saturation adsorption volumes (V_s), microgravity volume (W_0), mesogravity volume (W_{mez}) and saturation volume (V_s) by benzene vapor (Table 3).

Table 3
“Navbahor” bentonite loads obtained at 50°C and at different pH ЮКҚ - 1 nanocomposite pore size in sorbents.

pH	Pore size per BDХ, cm ³ /g		
	Micro pores	Mesoese pores	Common porous
	$W_0 \cdot 10^3, \text{cm}^3/\text{g}$	$W_{mez} \cdot 10^3, \text{cm}^3/\text{g}$	$W_s \cdot 10^3, \text{cm}^3/\text{g}$
2.0	0.324±0,05	1.052±0,06	1.376±0,10
5.2	0.302±0,01	0.911±0,04	1.213±0,24
10.2	0.182±0,04	0.872±0,08	1.054±0,15

As can be seen from the table, the volume of the mesoporous nanocomposite sorbent obtained under acidic conditions was 1.2 times higher than the volume of the porous sorbent obtained in a slightly alkaline medium, and the saturation volume was 1.3 times higher. From the

image obtained at 50°C and the Sem of ЮКҚ -2 composites from sequential bentonite synthesized at different r_n values, it was found that they represent the complete expression of polydispersities in the crystalline and Tarq phases of the components (Fig. 7).

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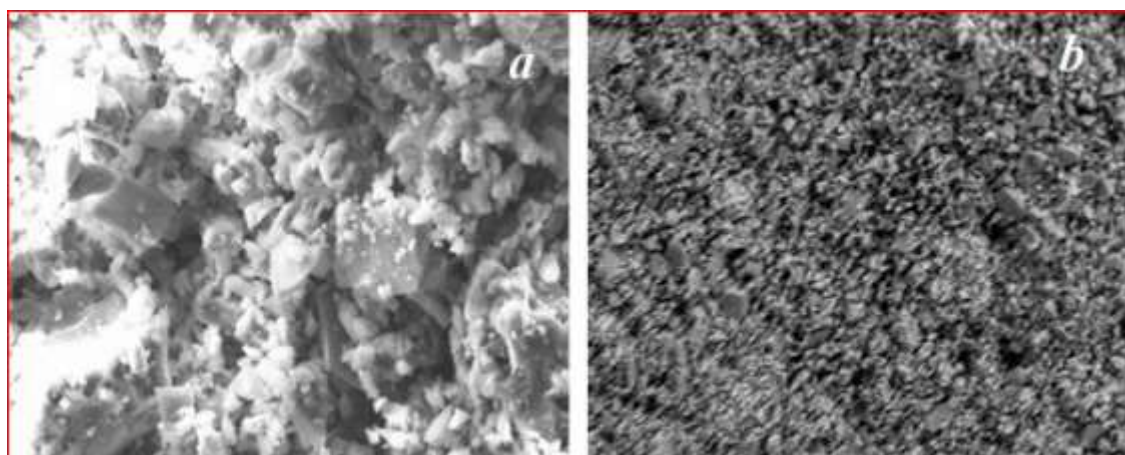


Figure - 7. The load of Navbahor bentonite obtained at 50°C pH = 2.0 and pH =10.2 is an ЮКҚ 2 composites in SEM.

From the Sam image of ЮКҚ -2 composites made of Navbahor bentonite, it can be seen that the degree of porosity is very low, and there are large fragments of crystals on the surface. From the sorbent samples obtained at 70°C and 90°C, it was found that they consist of denser mesoporous isomers (Fig. 8). From the figure, it can be seen

that the volume of the hysteresis rings formed due to capillary condensation breaks, and they sway towards a large relative pressure. Relative pressure $p/p_s=0.6-0.9$ hysteresis rings in samples taken at 70°C; It has been observed that sorbents obtained at 90 °C are formed in the range $p/p_s= 0.65\div 0.95$.

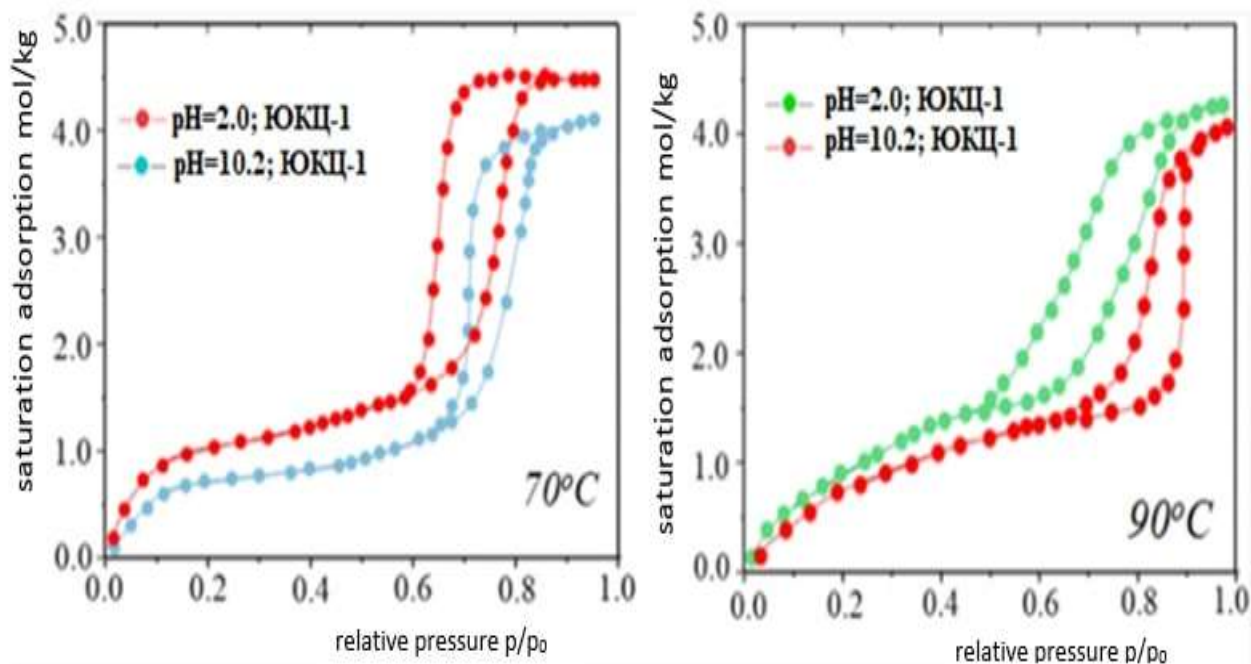


Figure 8. Isotherms of benzene vapor sorption in the absorbent IOKЦ-1 obtained from Navbahor bentonite at different temperatures (70° C and 90° C).

Loading from bentonite Navbahor IOKЦ-1[14-22] with a qualitative analysis of the sorption isotherms in the sorbent makes it possible to determine the types of water compounds in bentonite with the material and the stages of its drying temperature.

Colloidal capillary-porous material made of bentonite clay is a complex system that has both nature and structure, character, and plays an important role in the binding energy of a particular type of water at different stages of flour dehydration, if it has different moisture compositions.

P.A. The classification of forms of moisture binding in colloidal capillary-porous materials, proposed by Rebinder [23-25], takes into account the formation of various forms and the energy of their binding to the material. According to him, all its forms of moisture are divided into three large groups: chemical (molecular), physico-chemical

(absorption, osmotic humidity during storage (swelling and structural humidity), and physico-mechanical (humidity in macro- and microcapillaries).

The quantitative description of the contact energy with the material is of great practical importance. To determine this, the only criterion used in the classification of contact forms with the material is P. A. Rebinder; - this is the value of the free energy of isothermal dehydration. Here we come to the conclusion that due to the connection of water with the material, the pressure of water vapor on its surface decreases, so the free energy decreases accordingly. This decrease in free energy - work (in erg/mol), which is spent on separating 1 mole of water from the material, more precisely, on its disconnection, is considered binding energy, which at a constant temperature releases:

$$-\Delta F = L = RT \ln \frac{P_u}{P_H} = -RT \ln \varphi$$

here: R –gas constant.

P_u –the pressure of steam saturated with free water..

P_H – upper material moisture balance partial pressure of water vapor, in an inert gas or vacuum $\varphi = \frac{P_u}{P_H}$; upper material balance the lower the partial pressure of water vapor (P_H) the stronger the strong bond of water with the material, the more it is less, for free water (P_u) the pressure of saturated steam of free water is equalized. The binding energy is 0 when the binding energy is $\phi=1$:

$$-\Delta F = L = -RT \ln 1 = 0..$$

In bentonite, bentonite isotherms of sorption are mainly contained in three types of bound water (moisture): physico-mechanical (moisture in macro-and microcapillaries), physico-chemical (due to absorption, osmotic storage), and

chemical (molecular). At the initial initial initial stage (at a relative pressure of 0-0.6) in the emergency bentonite obtained from the sorption line (at a temperature of 70°C and 90°C), physico-mechanical (humidity of macro- and microcapillaries), that is, in the polymolecular bond and at the second stage (0.6-0.9), physico-chemical (due to absorption, osmotic storage) indicates the presence of monomolecular In our opinion, the formation of hysteresis rings in the second phase of sorption the isotherm indicates both polymolecular and monomolecular energy bonds, i.e., the formation of layers.

The textural characteristics of ЮКЦ-1 nanocomposite sorbents obtained from Navajor bentonite obtained at 90 °C and 70 °C are presented in Table 4.

Table 4.

Textural characteristics of ЮКЦ -1 nanocomposite sorbents from Navbahor bentonite obtained at various temperatures

t^0, C	pH	$S_{bet} M^2/g$	$\Gamma_s, mol/kg$	D, nm
70	2,0	450.8±35.0	3.5±0.3	25.3±2.2
	5,2	456.2±36.1	3.2±0.3	25.2±2.1
	10,2	436.2±34.8	2.8±0.2	48.6±3.8
90	2,0	400.3±32.6	1.6±0.1	52.2±4.8
	5,2	350.6±29.8	1.6±0.1	60.5±5.2
	10,2	356.5±30.2	0.8±0.1	72.8±6.4

Textural descriptions of ЮКЦ -1 nanocomposite sorbents obtained from Navbahor bentonite obtained at different temperatures also confirm the above points: with an increase in pH, the porosity surface decreases, with an increase in temperature, the decrease also continues, and the porosity diameter increases.

Conclusion

Mesoporous silica is a preservative used in the preparation of bentonite sorbent nanocatalysts, the analysis of isotherms sorption and the results obtained are of particular scientific and practical importance, and the descriptions of its structure and surface morphology: a high comparative surface, is porous, and has a size in the nanometer range, and its specific physico-chemical properties confirm that the bulk of the usual porous on the surface

In the SEM image of navajor bentonite composites, it can be seen that the porosity is very

low, and there are large fragments of crystals on the surface. From the sorbent samples obtained at 70°C and 90°C, it was found that they consist of a more voluminous mesogovac

Bentonite of the curve of the resulting sorption isotherms contains mainly three types of bound water (moisture): physico-mechanical (moisture in macro- and microcapillaries), physico-chemical (due to absorption, osmotic storage), chemical (molecular),. In Navajor bentonite obtained from the sorption line (at 70 °C and 90 °C), the initial start indicates the presence of a physically mechanized polymolecular bond in the first barrel (at a relative pressure of 0-0.6) and a physico-chemical monomolecular bond in the second barrel (0.6-0.9). The ligation autopsy of people with hysteresis in the second row of sorption isotherms indicates the dependence of both polymolecular and monomolecular energy.

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