ISSN 2181-8622

Manufacturing technology problems

Scientific and Technical Journal Namangan Institute of Engineering and Technology

INDEX COPERNICUS INTERNATIONAL

Volume 8
Issue 4
2023

Google Scholar
Crossref doi
SLIB.UZ Scientific library of Uzbekistan

STUDY OF ADSORPTION ISOTHERMS OF POLAR AND NON-POLAR MOLECULES ON SILICA ADSORBENTS

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Abstract:

Objective. The purpose of this research work is to study the adsorption isotherms of mesoporous silica adsorbents, their sorption-structural indicators and their relative surface area.

Methods. Adsorption isotherms of mesoporous adsorbents were carried out using the quartz spring balance method in the Mac-Ben-Bakra device. Using this method, adsorption isotherms of silica adsorbents synthesized at different temperatures are obtained. Based on this, information is given about their sorption structure characteristics.

Results. During the research, the adsorption isotherms of the samples (230/22, 234/22 and 235/22) showed a steep appearance due to the high amount of adsorption P/P_s=0.2 at low relative pressures. The curves of the adsorption isotherm graph of adsorbents showed that they correspond to type IV of the classification proposed by Brunauer. The main absorptions of adsorption of benzene molecules on silica adsorbents: 24.1% in 230/22, 19.7% in 213/22, 24.2% in 234/22, -27.9% in 235/22, -23.9% in 244/22, -21.6% in 245/22. corresponds to the amount of monolayer capacity of adsorbents.

Conclusion. Adsorption isotherms were studied in the presence of polar molecule water and nonpolar molecule benzene as adsorbate on silica adsorbents . Based on the isotherm curves of synthesized silica adsorbents, it was determined that they belong to type IV according to Based on the isotherm curves of synthesized silica adsorbents, it was determined that they belong to type IV according to Brunaeer's classification. The sorption-structural characteristics studied during the research and the data obtained on the volumetric pores gave results on their important dimensions such as micro- and meso-porosity, radius size of the pores, as well as the relative surface area.

Keywords: isotherm, benzene, water, Mac-Ben-Bakra, micropore, mesopore, specific surface area, volume of pores, adsorption.

Introduction. Today, adsorbents are widely used in various fields of production. The main participant of the adsorption process is the adsorbent product, which enters our country as an imported product. In the synthesis of sorbents, their production on the basis of local raw materials, especially with the help of waste, plays an important role in solving the problem of environment and ecology, as well as in solving the problem of cost and economy. To date, adsorbents are based on various raw materials, including lignite and hard coal [1-3], activated wood of various trees [4-7], waste from the food industry [8-10], as well as zeolites [11-12] and bentonite. with the help of adsorbents obtained [13-15] are widely used in various industries. In the production of adsorbents, issues of ecology and environmental protection are provided based on the use of waste.

The raw material is important when obtaining the sorbent, so it is important to select it taking into account its chemical composition and adsorbing properties. For this reason, in this research work, to obtain mesoporous adsorbents, it is important to select waste raw materials containing silica - rice husk [15].

From an analysis of the literature, it is clear that the authors [16] proposed a technology for extracting silicon dioxide by burning rice husks (by pyrolysis method), processing residual ash, recycling with acid or alkali. The main criterion was the use of a simple and cheap technology for extracting SiO_2 in a chemically pure state. In a previous research work [15], chemically pure silicon oxide was obtained, and in this work its adsorption properties were studied, including the sorption-structural index using an isotherm, as well as the volumetric theories of its pores.

Methodology. Adsorption isotherms of mesoporous adsorbents based on silica and chitosan synthesized at different temperatures were determined using a Mac-Ben-Bakra device using a quartz spring balance method. The device is equipped with a highly sensitive quartz spiral. Its sensitivity level is $1.78\text{-}10^{-3}$ kg/m. Samples of adsorbents in an adsorption flask (test tube) were kept in a water thermostat at a temperature of 20°C with an accuracy of 0.1°C. The structure of the device and the main working parts of the working system are structured as follows: - columns with quartz springs (equipped with cups in which the studied samples of adsorbents are placed, measured with an accuracy of 1 g), - a fore vacuum pump (brand VN - 461M, - a diffusion pump (residual the pressure in the system is 1.33 • 10-3 Pa until a vacuum is created, the pressure in the system is controlled using a screw thermo-vacuum gauge (brand VIT2), U-shaped pressure gauges - a trap (it is designed to trap various gases and vapors water in a system with liquid nitrogen), ampoules in which adsorbents are placed, and taps for separating parts of the device from each other. Fore vacuum and diffusion pumps produce up to $1\text{-}10^{-5}$ mm Hg. vacuum in the adsorption device. The pressure difference in U-shaped pressure gauges is measured using a cathetometer type B630. The cathetometer has a resolution of 0.05 mm. The samples prepared for the study were ground in an agate mortar to a powder and, after thorough mixing, were weighed and placed in a cup. The pressure in the system is stabilized by evacuation for 6-8 hours. Benzene and water obtained as adsorbate were purified and dried under vacuum conditions before being used in adsorption, its vapor pressure was first frozen and then heated to release dissolved gases from it until it became the same as the pressure data vapors indicated in the tables for pure benzene and water, and studied its adsorption.

Results and discussion. The device was cleaned and dried under vacuum

before adsorption of the samples with selected organic benzene and non-polar water vapor molecules as adsorbate. After freezing and then heating until the vapor pressure inside the device equals the vapor pressure data for pure benzene and water given in the tables, the adsorption process was studied after the release of dissolved gases.

It can be seen from the adsorption isotherms in the mentioned systems that the amount of adsorption rises sharply from the zero value of the relative specific pressure to the value P/P_s ≈0.4, and then the adsorption slowly increases and approaches the saturation state.

The sharp appearance of the adsorption isotherms at such a low relative pressure (P/P_s ≈0.4) is a reason to conclude that benzene vapors are adsorbed on surfaces with a high adsorption potential in the initial fillings.

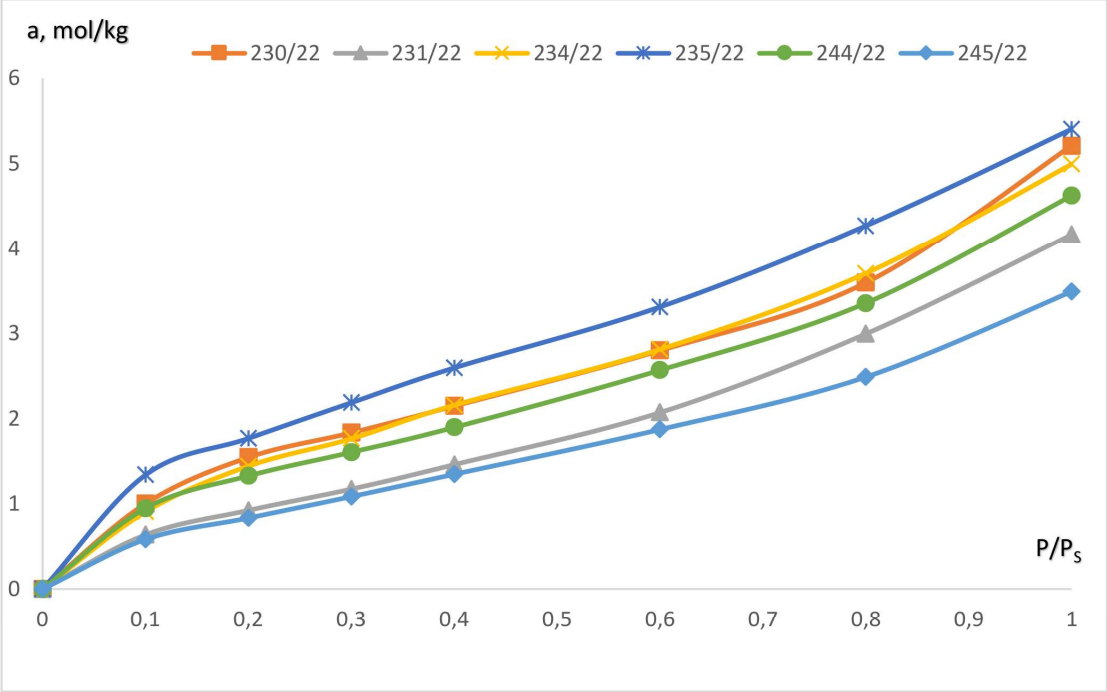

Figure 1. Benzene vapor adsorption isotherm on silica adsorbents

In the course of research, it is observed that the isotherm is steep due to the high adsorption amount of P/P_s =0.2 in samples synthesized from silicon oxide and chitosan (230/22, 234/22 and 235/22) at low relative pressures. This shows that the curves of the adsorption isotherm of all adsorbents obtained during the experimental preparation belong to type IV of the classification proposed by Brunauer.

The appearance of adsorption isotherms depends on the properties of the adsorbent and the absorbed substance adsorbate and the forces of interaction between them. First, it is related to the size, nature and charge of the exchangeable cations in the samples, and secondly, it is related to the specificity of the interaction of non-polar benzene molecules with modified adsorbents, i.e., the change in the hydrophilic and lyophilic nature of the adsorbents. In the synthesized sample 235/22, the adsorption amount of benzene vapors is higher compared to other adsorbents, due to the interaction of non-polar benzene molecules with cations between the adsorbent layers. During the research, it can be seen that the relative

pressure of the adsorption isotherm increases again in the range P/P_s =0.6-1.0. Absorption of adsorbate molecules into adsorbent pores indicates that adsorption of benzene vapors in these samples occurred as a result of capillary condensation in secondary pores.

From the structural adsorption parameters of adsorbents, the specific surface area (S) was determined using the Brunauer-Emmett-Teller (BET) theory equation. If P/P_s /a(1 - P/P_s) is placed on the ordinate axis and P/P_s values on the abscissa axis, it is observed that straight line coordinates are generated. The specific surface area of adsorbents was calculated using the following formula:

$$S = a_m \cdot N \cdot \omega_0$$

Where: S-relative specific surface (m^2/g);

a_m- monomolecular layer (mol/kg);

N_A- Avagadro's number;

ω- surface area occupied by one molecule (nm^2)

Based on the isotherms of benzene vapor adsorption on synthesized silica adsorbents, the monolayer capacity am, saturation volume Vs (or adsorption as) and their relative surfaces S were calculated from the important indicators of adsorbents. The obtained results are presented in Table 1.

Table 1

Results of sorption-structural indicators of benzene vapor adsorption on cermene earth adsorbent samples

Adsorbent samples	Monolayer capacity, a_m, mol/kg	Relative surface area, $S \cdot 10^{-3}$, m^2/kg	Saturation adsorption a_s, mol/kg
230/22	1.257	302.76	5.21
231/22	0.820	197.51	4.16
234/22	1.207	286.78	4.99
235/22	1.510	363.61	5.40
244/22	1.108	266.73	4.62
245/22	0.754	181.60	3.49

The main part of the absorption of benzene molecules in the synthesized adsorbent samples is 24.1% in the 230/22 adsorbent sample, 19.7% in the 213/22 adsorbent sample, 24.2% in the 234/22 sample and -27.9% in the adsorbent sample 213/22. In the adsorbent sample 235/22, in the adsorbent sample 244/22 - 23.9% was observed, and in the 245/22 sample -21.6%, which corresponds to the monolayer capacity. The observed increase in the relative surface area and the size of the absorbent adsorption in the following samples of synthesized adsorbents: 245/22-231/22-244/22-234/22-230/22-235/22. Such a change in specific surface area and saturation adsorption in silica adsorbent samples depends on their chemical composition and the conditions during the synthesis process. Adsorption isotherms of benzene vapor in the samples and the volume of micropores (W_0) of adsorbents, adsorption volumes for saturated states (V_s) and the volume of mesopores were determined by the formula $W_{me} = V_s - W_0$ using the equation of the theory of volumetric filling of micropores (VTFM). The average radius of the pores was calculated according to the

formula . $r_{av} = \frac{2 \cdot V_s \cdot 10^4}{S}$. The obtained results are presented in Table 2.

Table 2

Pore volume indicators based on the results obtained from the adsorption of benzene vapors on adsorbents

Samples	Micropore size, $W_0 \cdot 10^3$, m^3/kg	Saturation volume, $V_s \cdot 10^3$, m^3/kg	Mesopore size, $W_{me} \cdot 10^3$, m^3/kg	Average pore radius, r_{av}, nm
230/22	0.297	0.461	0.164	3.05
231/22	0.222	0.369	0.147	3.74
234/22	0.284	0.442	0.158	3.08
235/22	0.336	0.479	0.143	2.63
244/22	0.265	0.410	0.145	3.07
245/22	0.195	0.309	0.11	3.41

From the analysis of the obtained results, it can be seen that the size of micropores in adsorbent samples 230/22, 234/22, 235/22 and 244/22 is close to each other, but in sample 230/22, the amount of mesopores is relatively large, and in sample 235/22, the saturation adsorption volume is relatively high. It was found that the remaining two adsorbents have small micropores and saturation adsorption capacity. All adsorbents are among mesoporous (2<r<50 nm) adsorbents according to the classification proposed by M.M.Dubinin according to the average radius of their pores.

In addition to the benzene vapor molecule, the adsorption isotherm of water vapor, a polar molecule, was selected as an adsorbate in the synthesized adsorbent samples. The obtained results are presented in Figure 2.

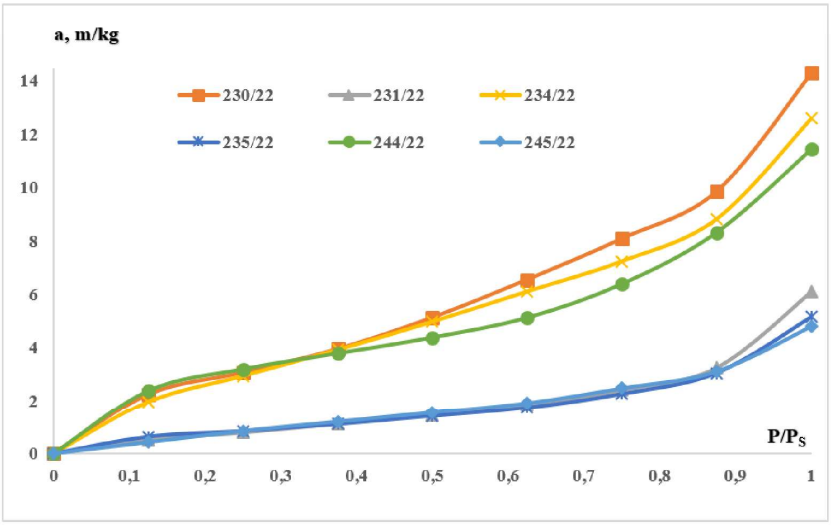

Figure 2. Adsorption isotherms of water vapor on silica adsorbent samples

In all systems studied during experimental studies, the isotherms first increase sharply to a relative pressure P/P_s=0.3, and then slowly increase in the range P/P_s = 0.4-0.7 and in the saturation state (P/P_s = 0, 7) P_s =0.8-1.0) increased sharply again. The initial stages of adsorption proceed when water molecules

form complexes with cations contained in adsorbents in the form of $K^+(H_2O)_n$. In the next stages, adsorption takes place between centers and pores with a high adsorption potential, and the last stages take place in the case of capillary condensation. The results of studying the pore structure of mesoporous dispersed solids are often associated with the interpretation of type IV adsorption isotherms. This type of isotherm is mainly characterized by absorption in mesoporous areas. At low pressure, the initial parts of type IV and type II isotherms become similar. But type IV isotherms shift upward from a certain point of pressure, and its slope decreases at higher pressures during the study. When approaching the saturated vapor pressure ($P/P_s=1$), it can be observed that the value of adsorption increases significantly. Dipole moment values of adsorbates (polar and non-polar), the nature and structure of pores in adsorbents, the interlayer distance formed between these pores and their volumes, and the size of the adsorbent radius have a great influence on the amount of adsorption. It was observed that the dipole moment of polar water vapor molecules is higher than the dipole moment of non-polar benzene molecules in all synthesized adsorbent samples. In particular, it was determined that the adsorption amount of the 230/22 adsorbent sample against benzene vapor is equal to 5.21 mmol/g, and against water vapor is equal to 14.30 mmol/g.

Therefore, the effect of the electronic nature of the adsorbent is considered significant when using adsorbents in adsorption processes.

Table 3

Structure - sorption indicators of water vapor adsorption on silica adsorbents

Samples	Monolayer capacity, a_m, mol/kg	Relative surface area, $S \cdot 10^{-3}$, m^2/kg	Saturation adsorption a_s, mol/kg
230/22	2.38	154.71	14.30
231/22	0.645	41.91	6.11
234/22	2.304	149.79	12.61
235/22	0.645	41.91	5.16
244/22	2.347	152.60	11.45
245/22	0.669	43.52	4.81

The basis of the results of isothermal polar molecular water and silica adsorbents by the studied sorption-structural properties, the obtained results are presented in the table. 3. In the presence of isotherms of water vapor adsorption on silica adsorbents, the monolayer capacity am, saturation volume V_s (or adsorption a_s), and their relative surface areas S_{sp} were calculated using important parameters of the sorbents.

Among the obtained silica adsorbents, the specific surface area (S_{sp}) and saturation volume (a_s) of sample 230/22 with a mass of 200 kDa, synthesized at a temperature of 400°C, turned out to be the largest. It can also be seen that the specific surface area (S_{sp}) and saturation volume (a_s) are high in the 244/22,500 kDa sample at 400°C. It can be seen that the specific surface area (S_{sp}) and saturation volume (a_s) of sample 230/22 are the highest among the obtained silica adsorbents.

Table 4

Indicators of pore volume during adsorption of water vapor on silica adsorbents

Samples	Micropore size, $W_0 \cdot 10^3$, m^3/kg	Saturation volume, $V_s \cdot 10^3$, m^3/kg	Mesopore size, $W_{me} \cdot 10^3$, m^3/kg	Average pore radius, r_{av}, nm
230/22	0.145	0.257	0.11	3.33
231/22	0.050	0.110	0.06	5.25
234/22	0.134	0.227	0.09	3.03
235/22	0.043	0.093	0.05	4.43
244/22	0.119	0.206	0.09	2.70
245/22	0.047	0.087	0.04	3.99

During the research, the adsorption isotherms of the samples (230/22, 234/22 and 235/22) showed a steep appearance due to the high amount of adsorption r/rs=0.2 at low relative pressures. The curves of the adsorption isotherm graph of adsorbents showed that they correspond to type IV of the classification proposed by Brunauer. The main absorptions of adsorption of benzene molecules on silica adsorbents: 24.1% in 230/22, 19.7% in 213/22, 24.2% in 234/22, -27.9% in 235/22, -23.9% in 244/22, -21.6% in 245/22. corresponds to the amount of monolayer capacity of adsorbents.

In this case, the amount of benzene vapor adsorption does not differ greatly due to the fact that the composition, structure, and nature of the synthesized adsorbents are almost similar to each other. The synthesized 235/22 sample adsorbent is characterized by a higher adsorption amount of benzene vapors compared to other adsorbents, higher active centers between adsorbent layers and higher pore size compared to other adsorbents.

Conclusions. Adsorption isotherms were studied in the presence of polar molecule water and nonpolar molecule benzene as adsorbate on silica adsorbents . Based on the isotherm curves of synthesized silica adsorbents, it was determined that they belong to type IV according to Based on the isotherm curves of synthesized silica adsorbents, it was determined that they belong to type IV according to Brunaeer's classification. The sorption-structural characteristics studied during the research and the data obtained on the volumetric pores gave results on their important dimensions such as micro- and meso-porosity, radius size of the pores, as well as the relative surface area.

It was found that the volume of micropores (W_0) of samples of silica adsorbent synthesized at a temperature of 400°C, as well as the adsorption volumes (V_s) for their saturated states, increased.

According to the results of adsorption of vapors of water molecules, the resulting adsorbents can be used as adsorbents for the purpose of purifying industrial products in various industries from polar compounds.

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