

Study Of Texture And Sorbtion Properties Of High Silicone Zeolites Synthesed From Bentonite

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Annotation: The article proposes a method of synthesizing high-silicon zeolite on the basis of "zol-gel" technology by activating bentonite in Navbahor district of Uzbekistan by physical and chemical methods, and studied the physicochemical, texture and sorption characteristics of the samples.

According to the X-ray phase analysis, the samples had a zeolite-like crystalline phase with a ZSM-40 structure, and the relative crystallinity of the samples was calculated from a reflection area of 22–25 ° relative to the most crystallized sample in the series.

As a result of studying the effect of the temperature of "Zol-gel" synthesis on the size, texture and structure of the crystals of the synthesized sorbents, it was proved that the increase in temperature leads to an increase in the average size, texture and structure and crystallinity of samples .

In the adsorption isotherms of water: with the increase of the ratio of silicon, a gradual disappearance of hysteresis is observed, as a result of a decrease in the volume of mesocytes, the total volume of pores decreases with increasing crystal size to the total volume of pores between packaged crystals. water: as the silicon ratio increases, the outer surface area of zeolites first increases, water: reaches a maximum for a sample synthesized from a mixture of tetraethyl orthosilicate, bentonite, sodium hydroxide, ethyl alcohol, hexamethylenediamine and citric acid with silicon = 50, then decreases to a minimum . The transition of zeolites to the H-shape leads to the formation of strong acid fields and promotes the formation of high-temperature peaks in the thermo-programmed desorption curves of ammonia in the range of 425 - 440 ° C. For all zeolites, the concentration of strong acid fields is close and lies in the range of 0.05 - 0.1 mmol / g.

As a result of the research, methods of synthesis conditions were developed to find optimal conditions for the synthesis of a type of high-silicon zeolite sample of the required size and structure, as well as with improved texture characteristics .

Based on the values of the adsorption capacity of the samples and the approximation of the phenol molecule to occupy an area of 0.57 nm², the surface occupied by phenol in the single-layer coating of the samples was evaluated.

Keywords: Bentonite, High Silicon Zeolite, "Zol-Gel" Technology, Water: Silicon Ratio, Texture, Sorption.

INTRODUCTION

Although bentonite is a cheap raw material, it is an effective [1] sorbent and is widely used in the purification of substances [2-8], in technology and in various sectors of the economy. However, in order to improve the adsorption properties of bentonite, additional mechanical, thermal and chemical treatment must be applied to it. The structures of bentonites allow modification in order to

improve their surface properties and adsorption characteristics [9]. When synthesizing porous materials, an attempt is made to increase the specific surface area, because as the specific surface area increases, the sorption properties of sorbents increase as the average size of the pores decreases [10]. Several methods of activation modification of natural clay bentonites are known; their selection should take into account the nature of the absorbed substance and the environment (liquid or gas) in which sorption occurs. It is necessary to reduce the cost of sorbents using inexpensive and detectable activators that provide high adsorption capacity of extractable materials [11].

Today, the oil and gas industry is developing rapidly. As a result, large amounts of toxic gases are released into the environment. This leads to the deterioration of the environment and ecology. Based on the above, today's catalytic and sorption processes require the creation of sorbents and catalysts with controllable properties in all ranges from micro-porous to macro-porous, which improves the mobility of molecules and the use of catalysts and sorbent surfaces. allows you to increase efficiency. Due to the presence of orderly micro-pores of molecular size, zeolites have a very large surface area (350–480 m²/g), however, large molecules cannot enter most active areas within micro-pores [12–16].

Zeolite adsorbent and catalyst samples for a multitude of solving the problem of efficient use of the more porous the streets of the city to keep the crystal structure of the system. Zeolite synthesis of two radically different approaches, the first micro-pore include: Zeolite crystals grate or partially through the re-establishment of the porous and the other directly formed during crystallization of zeolite more porous. Direct zeolite formed during the crystallization of additional porosity to the advantage of having a high crystallinity and improved features of the textile surface of the meso overlearning and the size and size distribution deals sure. The second approach can be two directions: ZOL-gel synthesis phase of meso groveling made use of the structure of large crystals and standards nanoplastinlarni or through zeolite nanocrystals to make sure meso / macro groveling [17 19].

Due to the orderly structure of micro-pores, zeolites have a surface area of 350-480 m²/g. However, the size of the zeolite pores is in the range of 0.2 to 1 nm. Therefore, it is difficult for gas and liquid molecules to enter the crystal pores. This does not allow full use of the surface of zeolite catalysts and adsorbents. Therefore, in recent years, scientific work has been carried out to improve the methods of synthesis of nanoseolites on the basis of high-silicon zeolites derived from bentonite and to study the possibility of their application [20-27].

EXPERIMENTAL PART

High-silicon zeolite was synthesized on the basis of "zol-ge" technology through physical and chemical processing of Navbahor bentonite. Methods of synthesis conditions have been developed to find optimal conditions for the synthesis of a type of high-silicon zeolite sample of the required size and structure as well as with improved texture characteristics .

For this purpose, we changed the water: silicon ratio and synthesis temperature. "For the synthesis of Zol-Gel we used the following substances: hexamethylenediamine and citric acid, sources of silicon - SiO_2 and tetraethylorthosilicate, bentonite, sodium hydroxide, ethyl alcohol, hexamethylenediamine and citric acid.

For the synthesis of high-silicon zeolite at different "zol-gel" synthesis temperatures and the standard synthesis of high-silicon zeolite nanocrystals, the gel was prepared by the following methods: 15.0 ml of tetraethylorthosilicate, bentonite, sodium hydroxide, ethyl alcohol, hexamethylenediamine and citric acid and 15.0 ml of ethanol solution was added in a thin stream to 15.0 ml of distilled water and 15.0 ml of hexamethylenediamine solution at 100 ° C and the mixture was stirred vigorously for 20 min.

For the two-step synthesis of high-silicon zeolite, the gel was prepared according to the following method: 50.0 ml of H_2O solution was added in a thin stream to 50.0 ml of tetraethylorthosilicate, bentonite, sodium hydroxide, ethyl alcohol, hexamethylenediamine and citric acid and 50.0 ml of ethanol solution. 12.0 ml of 0.1 M NaOH and 50.0 ml of hexamethylenediamine and the mixture were left under strong stirring for 10 min.

Preparation of gels from the core solution (iz matochnyx rastvorov) was carried out as follows. 15.0 ml of tetraethylorthosilicate, bentonite, sodium hydroxide, ethyl alcohol, hexamethylenediamine and citric acid were added in 38 ml separated from high-silicon zeolite crystals synthesized at 100 ° C with a thin stream .

For the synthesis of high-silicon zeolite microcrystals, the gel was obtained at room temperature as follows: 15 g of bentonite and 7.6 g of SiO_2 were slowly added to an aqueous solution of 0.72 g of NaOH and 3.18 g of citric acid and allowed to stand for 25 min.

The synthesis products, depending on the size of the particles mother liquid is separated by filtration and washed with distilled water at pH = 7. The nanocrystals were washed in three purification processes consisting of the addition of water, dispersion in an ultrasonic bath, centering, and separation of the liquid. After the synthesis of the product was dried on 120 ° C for 12 hours. One part of the high-silicon zeolite nanocrystals formed a suspension of nanocrystals, the other in the form of monoliths obtained after centralization, forming a set of correspondingly loose and dense particles.

The dried samples were continuously heated at 550 ° C for 6 h, respectively.

Half of the samples of nanocrystals and microcrystals of high-silicon zeolite were activated in a thermostatic reactor equipped with a magnetic stirrer. High silicon zeolite powder was added to a 1 M solution of hydrochloric acid and then mixed at 60 ° C for 40 min. The powder was then separated by filtration and washed with copious amounts of water, dried at 120 ° C for 10 h, and then potassiumized at 550 ° C for 4 h. The resulting powder was ground with lime and a section of <0.25 mm was separated using a sieve.

The elemental composition of the obtained samples was analyzed on an energy-dispersed X-ray fluorescent spectrometer .

Sample porous structure parameters were performed in a fast-reacting gaseous adsorption analyzer on low-temperature nitrogen adsorption.

The Brunauer-Emmett-Taylor (BET) method was used to measure the specific surface area of solid samples. This method uses the BET equation:

$$\frac{1}{W \cdot \left(\frac{P_0}{P} - 1\right)} = \frac{1}{W_m \cdot C} + \frac{C - 1}{W_m \cdot C} \cdot \frac{P}{P_0}$$

where W is the adsorbed mass of the gas when the pressure ratio is R / R_0 . W_m is the mass of monolayer adsorbed bodies completely covering the surface of the sample, S-BET constant, adsorption energy corresponding to the first adsorption layer.

The Barrett-Joyner-Halend (BJH) method was used to find the volume of the pores and to find the laws of distribution by size . The calculations used values of the pressure of desorption and adsorption isotherms in the range 0.97-0.4 R / R_0 .

The inspection of particle size was determined by the method of laser beam diffraction in a diffraction analyzer with lasers. For spectroscopic examination, all samples were scaled down and dried at 80 °C until the mass was constant .

EXPERIMENTAL RESULTS AND THEIR DISCUSSION

According to the X-ray phase analysis data (Fig. 1), the samples have a crystalline phase corresponding to zeolite with a ZSM-40 structure.

The size of the average crystal was measured using the dynamic light scattering method, and the image of the particles was obtained by the scanning electron microscopy method. The characteristics of the synthesized high silicon zeolite are given in Table 1.

Table 1 Properties of synthesized zeolites

For example	Average crystal size, nm	Relative crystallinity,%	Product efficiency,%
HIGH SILICONE ZEOLITE Water: silicon = 10	180 ^b	99	92
HIGH SILICONE ZEOLITE	250 ^b	100	88

Water: silicon = 25			
HIGH SILICONE ZEOLITE Water: silicon = 50	500 ^b	71	86
HIGH SILICONE ZEOLITE Water: silicon = 100	750 ^b	90	84
HIGH SILICONE ZEOLITE T synthesis = 80 ° C	120 ^b	67	83
HIGH SILICONE ZEOLITE T synthesis = 100 ° C	150 ^b	70	80
HIGH SILICONE ZEOLITE T synthesis = 120 ° C	220 ^b	80	85
HIGH SILICONE ZEOLITE T synthesis = 140 ° C	250 ^b	93	88
HIGH SILICONE ZEOLITE Water: silicon = 20	130 ^a	76	-
HIGH SILICONE ZEOLITE T synthesis = 90 ° C	180 ^a	80	65
HIGH SILICONE ZEOLITE T synthesis = 150 ° C	450 ^b	100	79

^a according to **dynamic light scattering data**; ^b on ^a **scanning electron microscope**

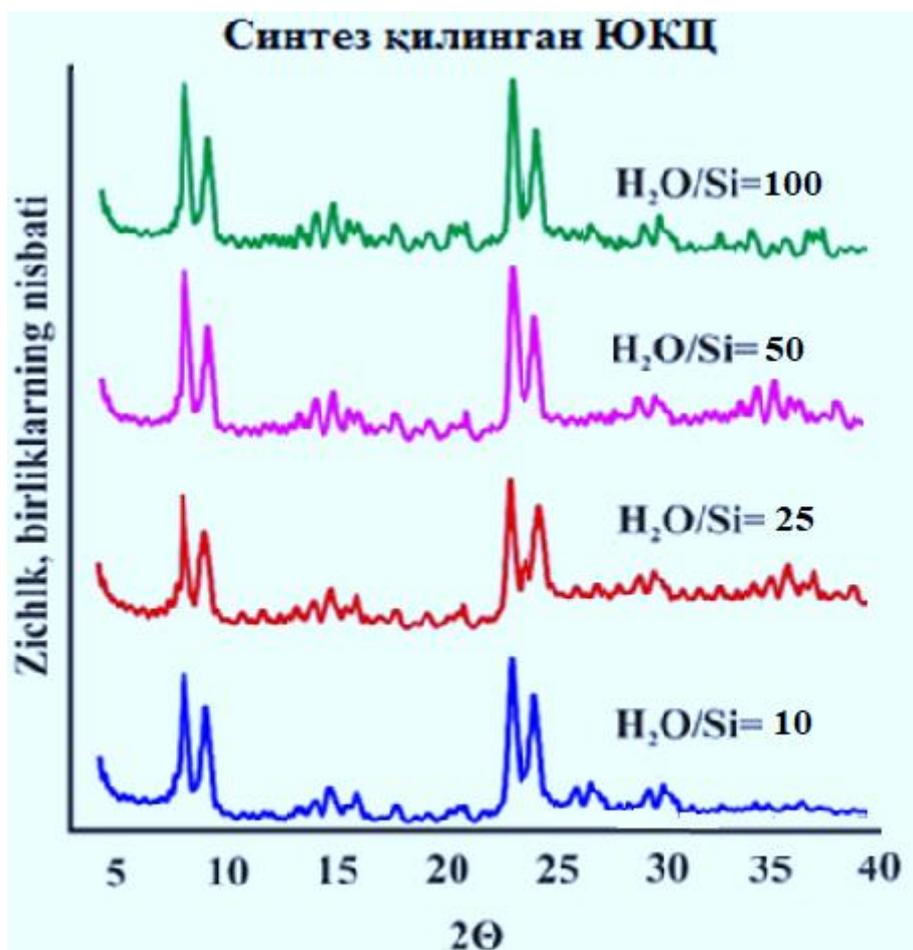


Figure 1. X-rays of samples of high-silicon zeolite Water: silicon synthesized from a mixture of tetraethylorthosilicate, bentonite, sodium hydroxide, ethyl alcohol, hexamethylenediamine and citric acid in various ratios

We obtained nano-zeolites of 10–30 nm in size by removing ethanol formed by the hydrolysis of a mixture of tetraethylorthosilicate, bentonite, sodium hydroxide, ethyl alcohol, hexamethylenediamine, and citric acid (Fig. 2).

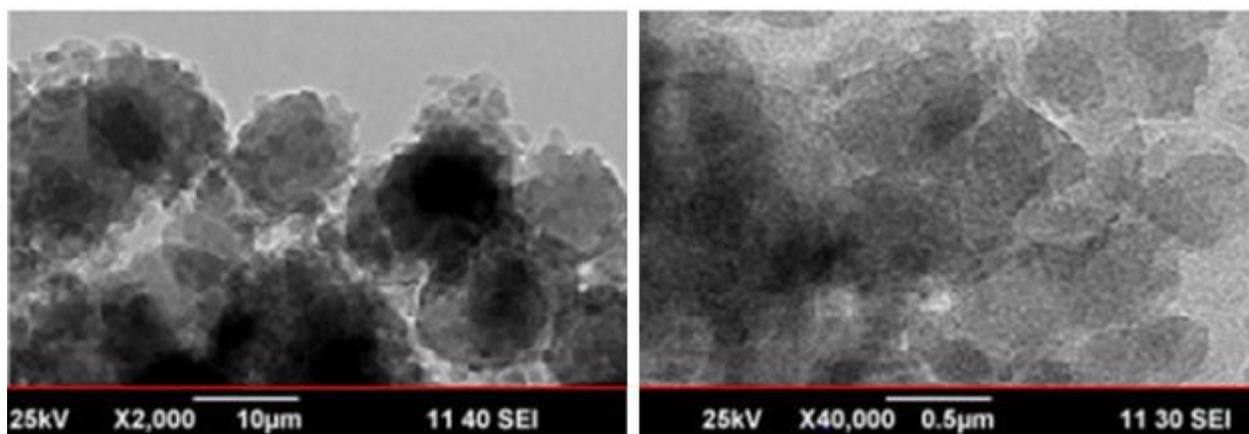


Figure 2. Water in the absence of ethanol: scanning electron microscope image of a sample synthesized from a mixture of tetraethylorthosilicate, bentonite, sodium hydroxide, ethyl alcohol, hexamethylenediamine and citric acid in the ratio of silicon = 10

Synthesis of a type of high-silicon zeolite sample with the required size and structure, as well as improved texture characteristics, as well as water in a mixture of tetraethylorthosilicate, bentonite, sodium hydroxide, ethyl alcohol, hexamethylenediamine and citric acid: depends on the ratio of silicon (Figures 3 and 4). $25 < \text{water} : \text{silicon} < 100$ aggregate-like oval crystals are formed. According to the scanning electron microscope data, aggregate-like crystals are single crystals. Crystal morphology is related to different mechanisms of crystal growth, depending on the state of the mixture consisting of tetraethylorthosilicate, bentonite, sodium hydroxide, ethyl alcohol, hexamethylenediamine, and citric acid. The growth of crystals at the level of $25 < \text{Water} : \text{Silicon} < 100$ is carried out by the mechanism of nanoparticle fusion.

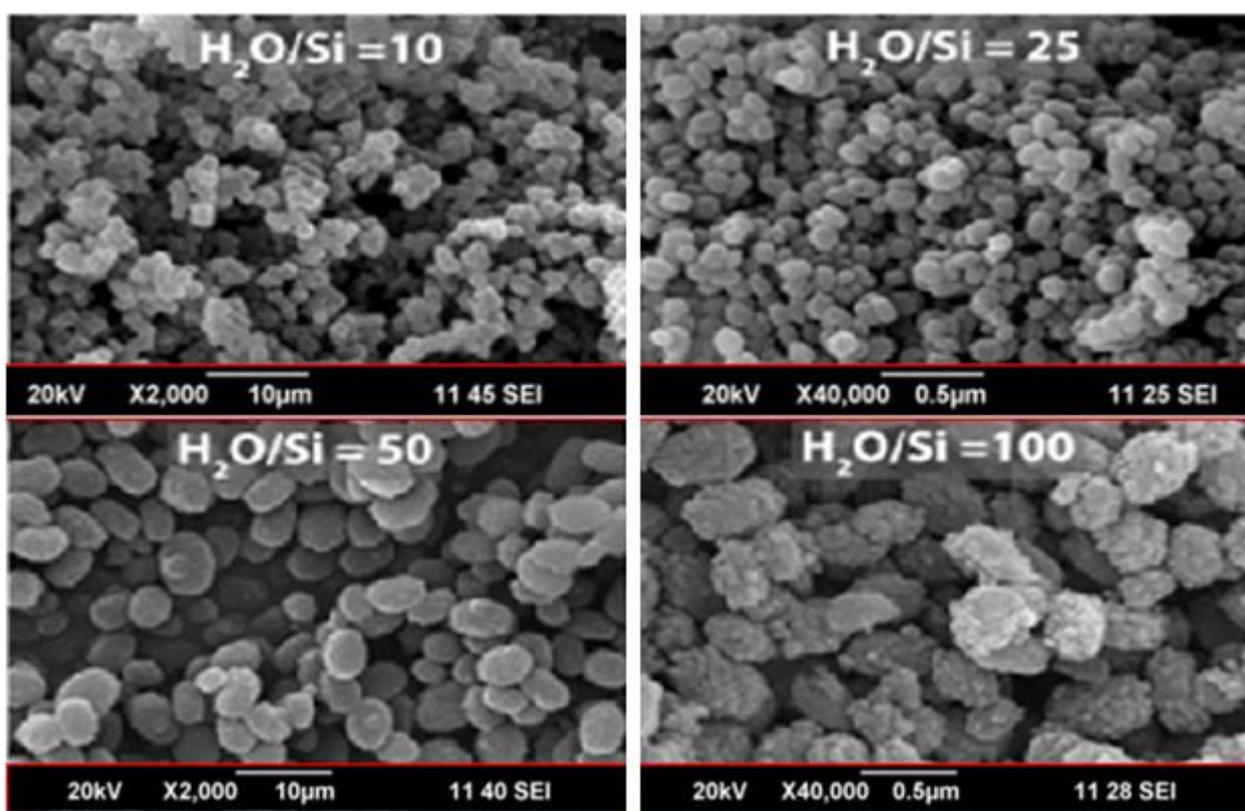


Figure 3. Variety Water: Scanning electron microscope images of high-silicon zeolite synthesized from a mixture of tetraethylorthosilicate, bentonite, sodium hydroxide, ethyl alcohol, hexamethylenediamine, and citric acid with silicon ratios.

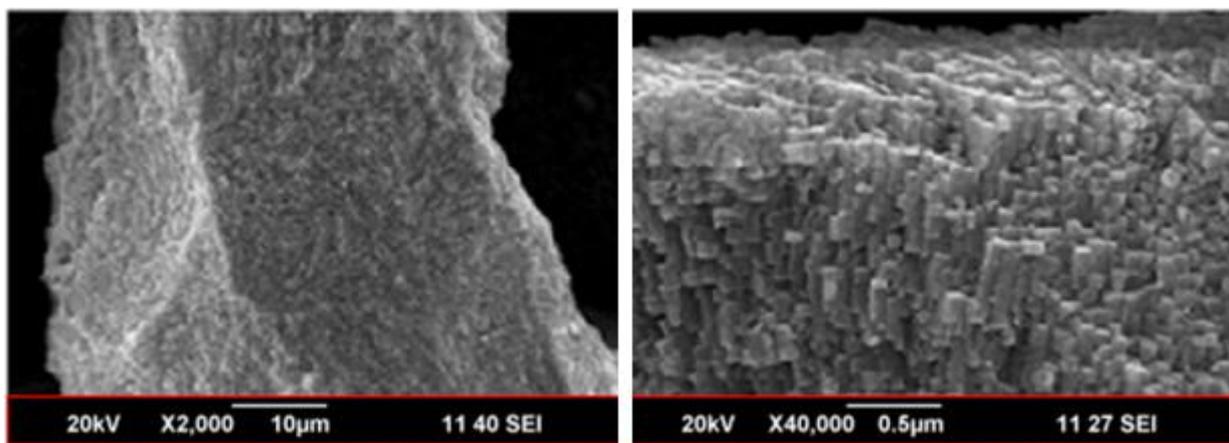


Figure 4. Water: Scanning electron microscope images of a sample synthesized from a mixture of tetraethylorthosilicate, bentonite, sodium hydroxide, ethyl alcohol, hexamethylenediamine, and citric acid at a ratio of silicon = 100.

Monodisperse crystals of high-silicon zeolite were synthesized at a temperature of 100–130 ° C to study the effect of the size and structure of the pores of the high-silicon zeolite sample obtained from 'Zol-gel' and the improved texture characteristics on the synthesis conditions . An increase in the synthesis temperature of 'Zol-gel' leads to an increase in the size and crystallinity of the average crystalline volume of the samples (Table 1, Fig. 5).

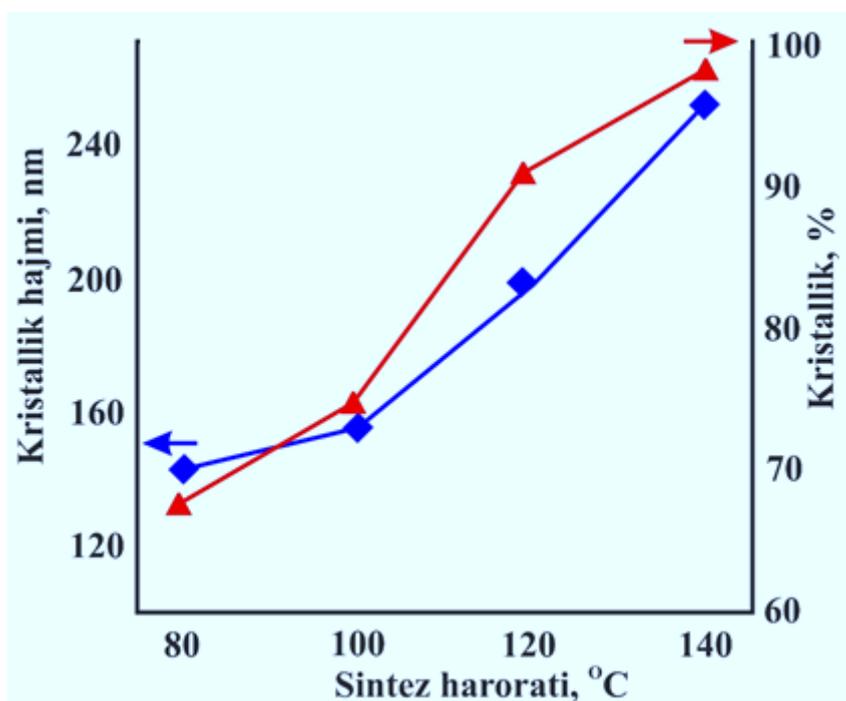


Figure 5. The size of the crystalline volume of the synthesized high-silicon zeolite and the dependence of the degree of crystallinity on the temperature of the "zol-gel" synthesis

High temperature of 120 ° C and 140 ° C, the silica tselotning two-step synthesis of an average of 320 nm, which is monodispers crystals. The two-stage synthesis of the desired size of crystals, can

significantly shorten the cooking time. The temperature of the first phase, all the time qo'sg'atish remained suspended, on the condition that the concentration of the nuclei and determines the size of the crystals in the final. The first step is to extend the period of temperature decrease qo'sg'atish increase in concentration in the core. The second phase of the temperature of the crystal growth of linear speed and reduces its synthesis increased.

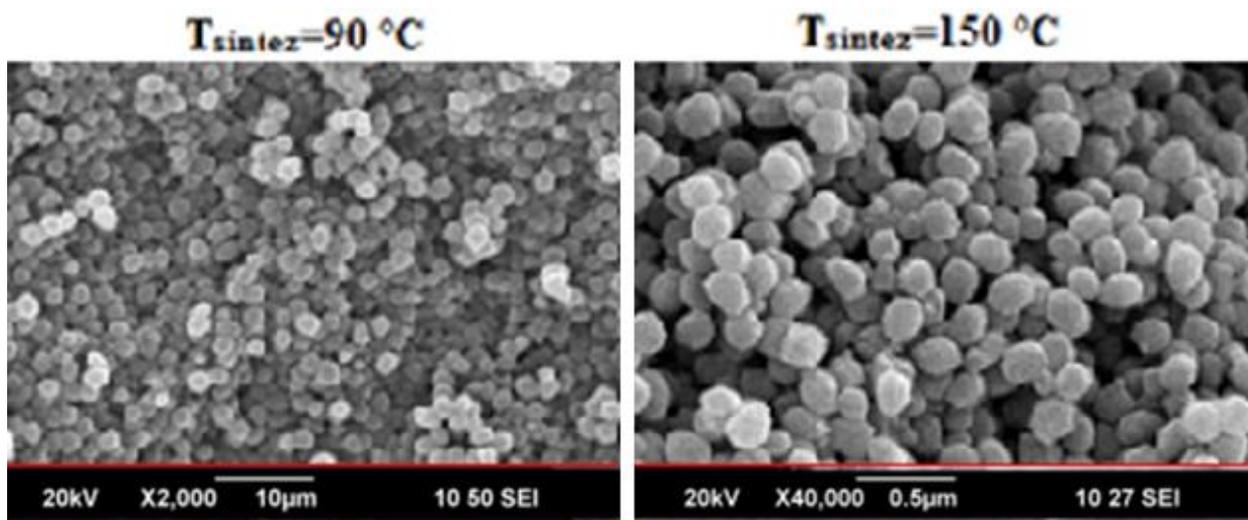


Figure 6. Scanning electron microscope images of high silicon zeolite samples synthesized at different temperatures .

For the economical consumption of reagents, especially the molecular template - hexamethylenediamine, parent solutions separated from high-silicon zeolite crystals were reused. As a result, the product efficiency of well-crystallized monodispers crystals of high-silicon zeolite was > 88% and averaged 265 and 360 nm at 90 and 150 ° C, respectively, at synthesis temperatures (Table 1, Fig. 6).

Water: silicon with a variety of tetraetilortosilikat, bentonite, sodium hydroxide, ethyl alcohol, geksametilendiamin and citric acid mixes synthesized seoliltarning textile properties are listed in Table 2. Izotermalarida adsorbed water: silicon ratio gisterezning gradually have been observed (Figure 7), as a result of a decrease in the volume of mezog`ovak packed up to the total amount of crystalline porous crystals, reducing the total size of the enlarged pore (Figure 8). The surface of the water: silicon ratio seoliltarning surface area increases, water: silicon = 50 tetraetilortosilikat, bentonite, sodium hydroxide, ethyl alcohol, geksametilendiamin and a mixture of citric acid synthesis, for example, then the minimum to the maximum value falls (Figure 8).

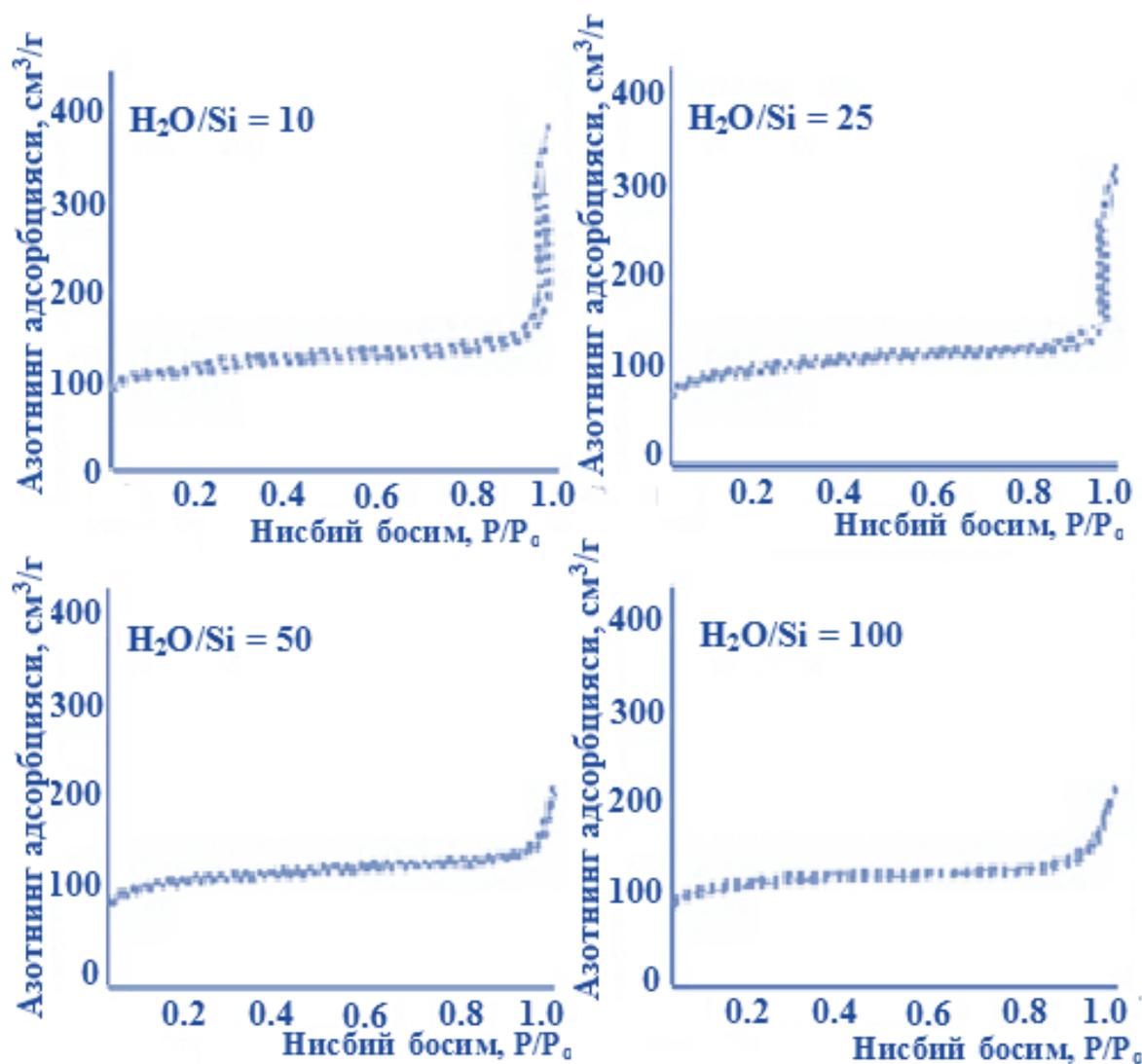


Figure 7. Water: Isotherms of nitrogen adsorption in zeolites synthesized from a mixture of tetraethylorthosilicate, bentonite, sodium hydroxide, ethyl alcohol, hexamethylenediamine and citric acid with different ratios of silicon.

Table 2. Texture properties of high-silicon zeolite zeolites.

For example	S _{BET} , m ² /g	S _{mesogovak} , m ² /g	V _{total} , cm ³ /g	V _{mikrog'ovak} , cm ³ /g
Water: silicon = 10	418	112	0.68	0.16
Water: silicon = 25	408	147	0.56	0.14
Water: silicon = 50	465	144	0.36	0.14
Water: silicon = 100	458	139	0.38	0.16

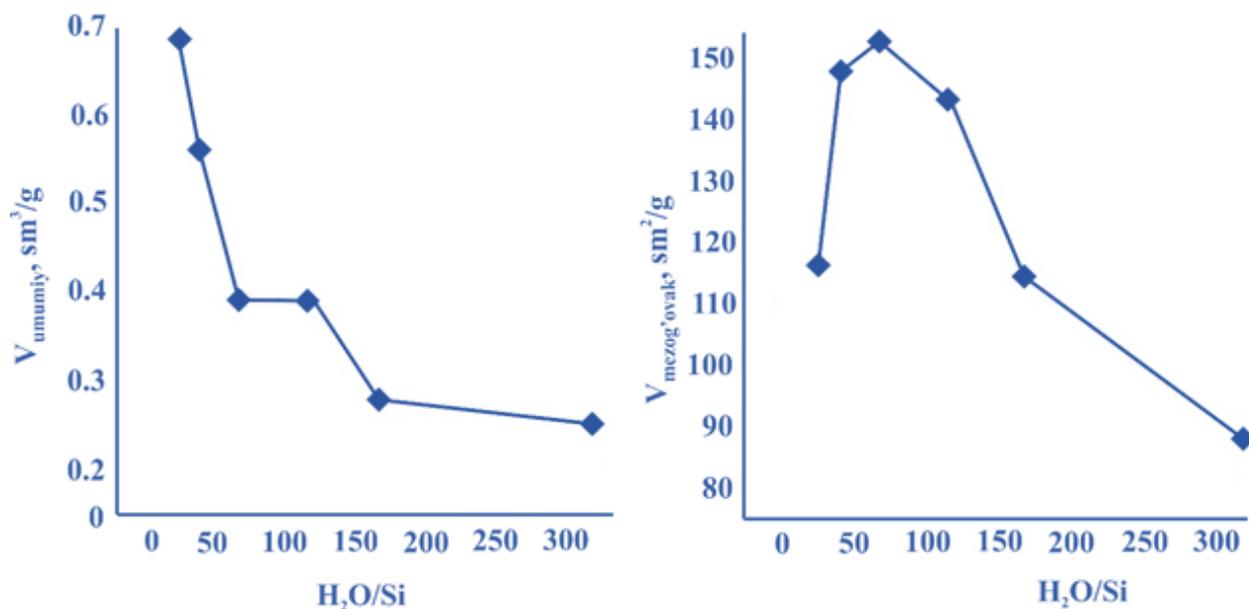


Figure 8. The total volume of the pores and the outer surface of the zeolites depend on the ratio of water: silicon in a mixture of tetraethylorthosilicate, bentonite, sodium hydroxide, ethyl alcohol, hexamethylenediamine and citric acid.

The transition of zeolites to the H-shape leads to the formation of strong acid fields and promotes the formation of high-temperature peaks in the thermo-programmed desorption curves of ammonia in the range of 425 - 440 ° C. For all zeolites, the concentration of strong acid fields is close and lies in the range of 0.05 - 0.1 mmol / g.

Thus, methods for the synthesis of high-silicon zeolite crystals in the range of 30 to 5000 nm and morphology of the desired size have been developed.

To study the stability of the texture and structure of the catalysts, the samples were tested using "Zol-gel" treatment at 170 and 200 ° C and activation with 1 M hydrochloric acid at 70 ° C. The properties of the catalysts before and after the tests are given in Table 3.

Table 3. Properties of catalysts before and after stability tests .

For example	S _{BET} , m ² /g	S _{mesogovak} , m ² /g	V _{total} , cm ³ /g	V _{mikrog'ovak} , cm ³ /g	Relative crystallinity,%
"Zol-gel" processing -200 ° C, 10 hours					
HIGH SILICONE ZEOLITE microcrystals BEFORE	351	56	0.25	0.17	95
HIGH SILICONE ZEOLITE	438	87	0.29	0.19	100

microcrystals AFTER					
"Zol-gel" processing - 200 ° C, 15 hours					
HIGH SILICONE ZEOLITE microcrystals BEFORE	421	75	0.25	0.17	100
Nanocrystals dense packaging BEFORE	557	207	0.77	0.15	94
Nanocrystals dense packaging AFTER	285	65	0.77	0.13	96
Activation with 1 M hydrochloric acid at 70 ° C for 30 min					
HIGH SILICONE ZEOLITE microcrystals BEFORE	400	110	0.22	0.15	100
HIGH SILICONE ZEOLITE microcrystals AFTER	439	119	0.27	0.15	94
Nanocrystals tight wrapping BEFORE	529	239	0.70	0.18	98
Nanocrystals tight wrapping AFTER	485	210	0.79	0.19	97

After "Zol-gel" treatment at 200 ° C, the specific surface area of high-silicon zeolite decreases, but the crystallinity level of the samples remains unchanged (Table 3). The decrease in the specific surface area of the sample is due to the synthesis of microporous particles of the amorphous silicate phase, which after the processing of "Zol-gel" formed porous globules up to 1 micron in size. Iadi (Fig. 9).

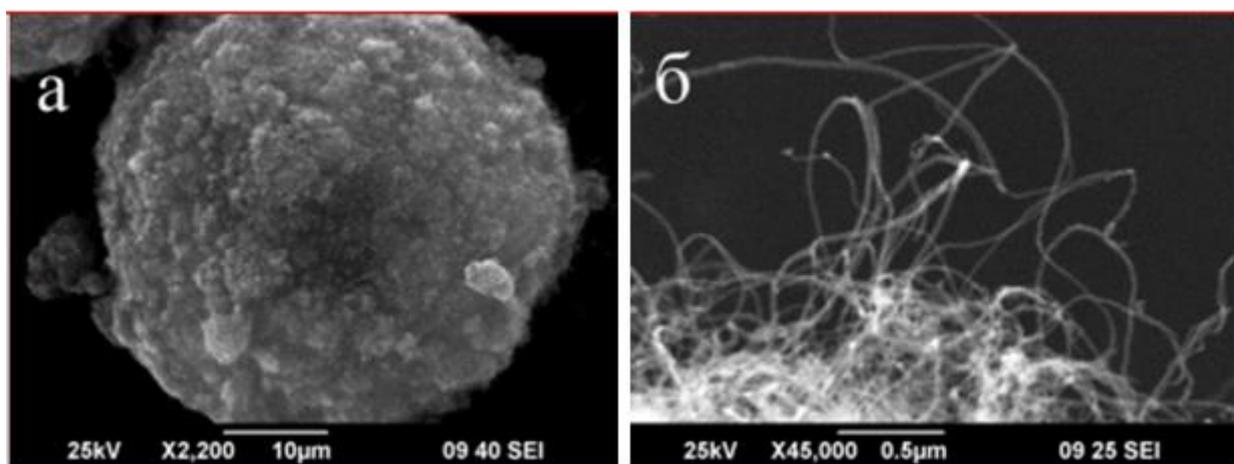


Figure 9. Scanning electron microscope images of high-silicon zeolite were performed for 10 hours at 200 ° C in an argon atmosphere before "Zol-gel" treatment (a) and after (b).

For high-silicon zeolite microcrystals, "Zol-gel" after treatment at 200 ° C simultaneously reduces the specific surface area and surfaces of mesocyttes, the total volume of pores, the volume of pores and crystallinity, as well as in the sample. an increase is observed as a result of washing out the amorphous phase, which is

During the 20 hours of high-silica tseolitning crystallized at a temperature of 200 ° C Zel micro-gel processing g`ovaklarning does not affect the overall size and the size of the mikrog`ovaklar, at the same time, the specific surface of the sample and the surface of the mezog`ovaklar decreases.

After "Zol-gel" treatment at a temperature of 200 ° C for 20 hours, the specific surface area of porous nanocarbons made of high-silicon zeolite nanocrystals is reduced by 20-30%, as the volume of micro-pores is reduced, the total size of the samples the size of the hollow is preserved (Fig. 10).

Activation of high-silicon zeolite microcrystals leads to a slight decrease in crystallinity due to the formation of lattice defects, an increase in specific surface area, mesocellular surface and the total volume of pores. The total volume of the pores and the distribution of meso-pores by size are maintained after the acid activation of porous nanocarbons made of high-silicon zeolite nanocrystals (Figure 10).

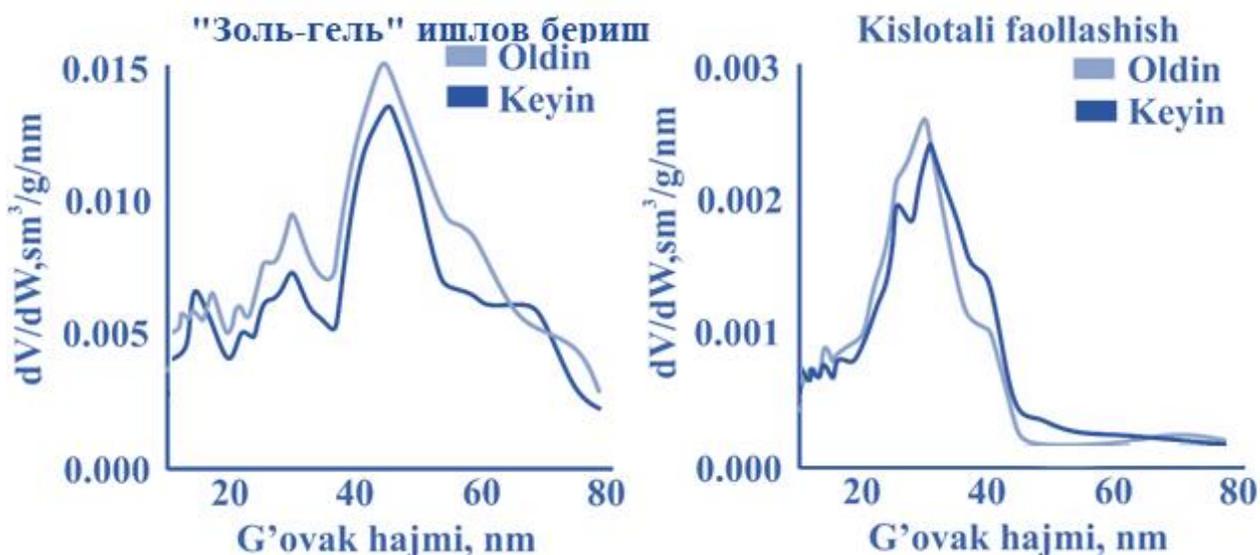


Figure 10. 30 min at 70 ° C with 1 M hydrochloric acid and 200 ° C for 15 hours to enable the 'Zel-gel processing before and after the high silica tseolitning tightly wrapped nanocrystals built g`ovakli nanouglerodlarda mezog`ovaklarning resolution distribution on.

Thus, the state of the pores of nanocarbons, consisting of high-silicon zeolite nanocrystals, is maintained after much stronger effects - "Zol-gel" treatment and acid activation.

Phenol adsorption isotherms in high silicon zeolite (Fig. 11) can be described by the Langmuir equation:

$$\Gamma = \Gamma_m \frac{K \cdot C}{1 + K \cdot C}$$

Where G is the adsorption, C is the equilibrium concentration of the adsorbed substance in the suspension, K is the adsorption constant, and G_m is the limiting adsorption capacity of the catalyst.

Adsorption constants and adsorption capacity of samples with respect to phenol according to Langmuir's equation were found (Table 4).

For nanoglerodes containing an amorphous phase, higher adsorption capacity is observed compared to high-silicon zeolite samples in relation to phenol .

Microporous samples are characterized by high phenol adsorption constants, the highest adsorption constant (7.2 l / g) is observed for high-silicon zeolite nanocrystals (Table 4).

Based on the values of the adsorption capacity of the samples and the approximation of the phenol molecule to an area of 0.57 nm², the phenol-occupied surface in the single-layer coating of the samples was evaluated (Table 4).

Table 4. Adsorption parameters of samples in aqueous solution of phenol, T = 30 ° C.

For example	K, l / g	G _m , mg / g	S _{PHOH} , m ² / g	S _{PHOH} / S _{BET}
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Microcrystals	2.9 ± 0.8	45 ± 7	160	0.36
Nanocrystals	7.0 ± 1.0	54 ± 2	188	0.38
HIGH SILICONE ZEOLITE	0.16 ± 0.04	127 ± 37	445	1.86

Phenol is adsorbed on the outer surface of a multilayer high-silicon zeolite sample (Table 4).

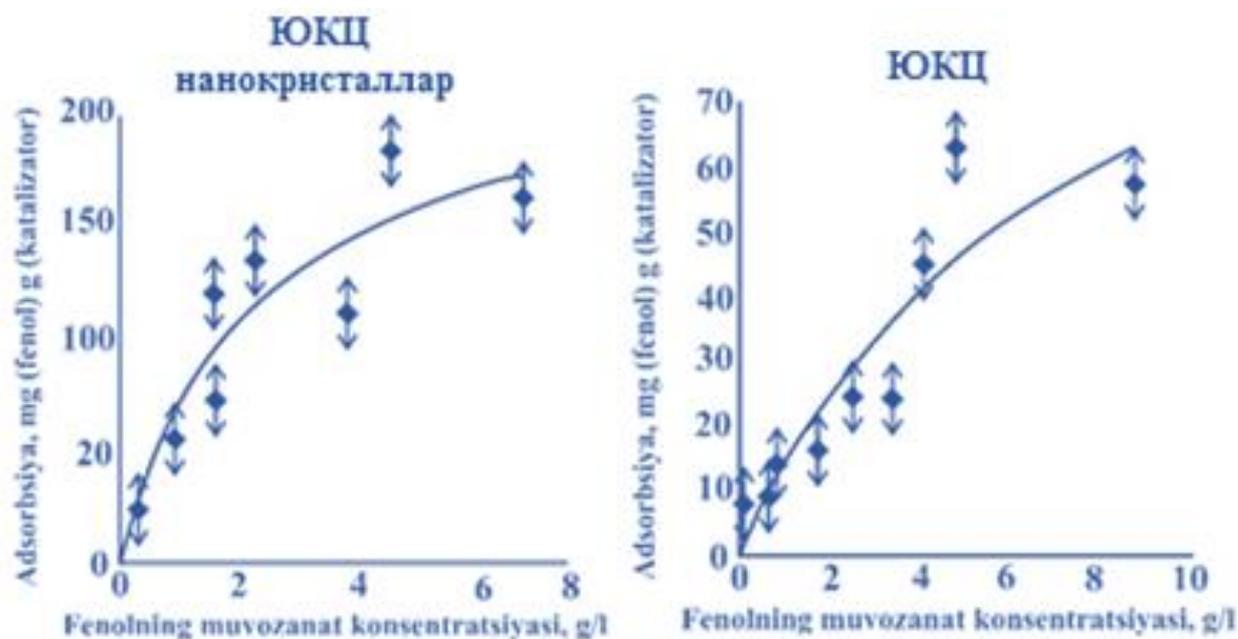


Figure 11. Isotherms of phenol adsorption from aqueous solutions in high silicon zeolite samples. T = 20–25 ° C, t = 60 min.

Figure 12 shows the electron diffuse reflection spectra of high-silicon zeolite crystals of various sizes, potassium-plated and activated with hydrochloric acid .

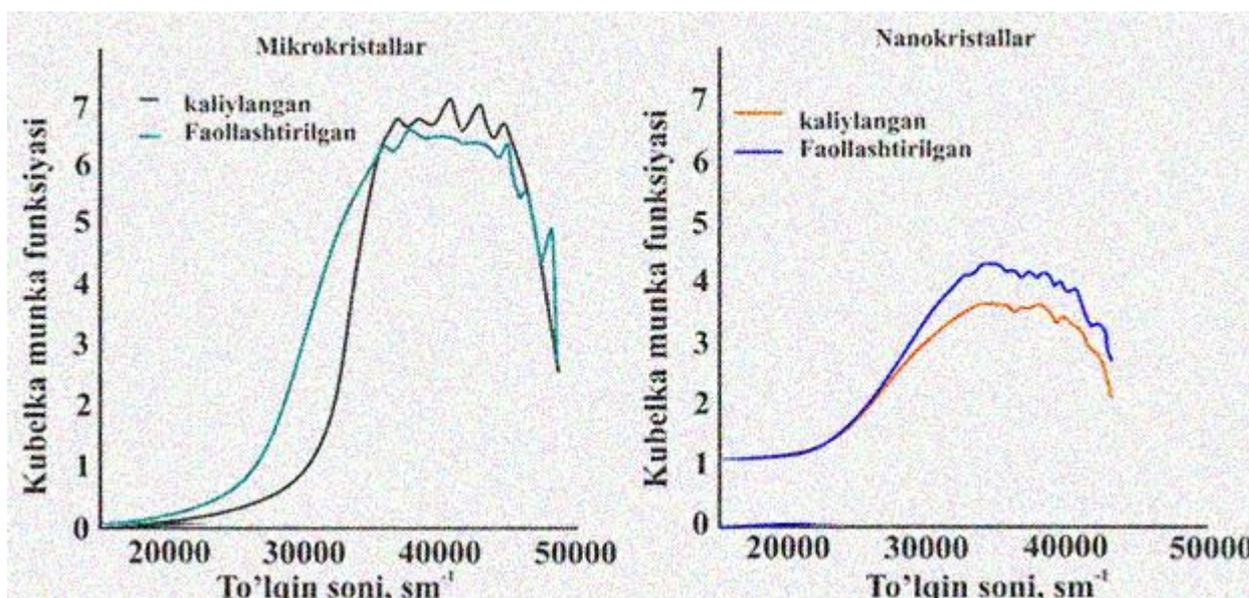


Figure 12. Electron diffuse reflection spectra of high silicon zeolite crystals of various sizes calcined and activated with hydrochloric acid .

Activation of microcrystals by treatment with hydrochloric acid leads to the subsequent migration of particles and their subsequent fusion.

CONCLUSION

- 1) Activation of bentonite in Navbahor district of the Republic of Uzbekistan by physical and chemical methods, then the method of synthesis of high-silicon zeolite on the basis of "zol-gel" technology was proposed and the physicochemical, texture and sorption characteristics of the samples were studied.
- 2) The effect of "Zol-gel" synthesis temperature on the size and morphology of crystals was studied, and monodisperse crystals of high-silicon zeolite were synthesized at a temperature of 100–130 °C. It has been proved that an increase in the synthesis temperature of 'Zol-gel' leads to an increase in the average crystal size and crystallinity of the samples.
- 3) Methods for synthesizing high-silicon zeolite crystals in the range of 30 to 5000 nm and morphology of the required size have been developed.
- 4) Based on the values of the adsorption capacity of the samples and the approximation of the phenol molecule to occupy an area of 0.57 nm², the surface occupied by phenol in the single-layer coating of the samples was evaluated.
- 5) The sequence of the potassiumization process is sufficient to activate the nanocrystals, acid treatment of the potassiumized nanocrystals helps to wash away the active centers, and additional acid treatment is required to maximize the activation of the microcrystals.

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