Investigation of textural, physico-chemical characteristics catalyst obtained method for the synthesis of vinyl acetate

Normurod Fayzullaev[1*], *Baxtiyor* Jabborov[2], *Guldasta* Kummetova[3], *Nargiza* Turdiyeva[4], and *Hamida* Adizova[2]

[1]Samarkand State University, University Boulevard, 15, Samarkand, 140104, Uzbekistan
[2]Bukhara State University, M. Iqbol Street, 11, Bukhara, 705018, Uzbekistan
[3]Gulistan State Pedagogical Institute, Talabalar Street, 49, Gulistan, 120100, Uzbekistan
[4]Asia International University, Street Gijduvan, 74, Bukhara, 200100, Uzbekistan

Abstract. The article investigates the textural and physico-chemical characteristics of a catalyst obtained by the sol-gel method for the synthesis of vinyl acetate. The dispersed properties of a catalyst with high catalytic activity selected for the catalytic vinylation of acetic acid in the presence of acetylene were studied using a scanning electron microscope (JSM-6510 LV). The main adsorption and structural characteristics of the load - specific surface area, pore volume and effective size - were obtained as a result of the work and determination of the isotherm of water vapor adsorption. From the adsorption measurement, the sample was treated in vacuum at 300 ° C for 8-10 hours. Adsorption was measured at room temperature. The adsorption balance was set for 22 hours. The relative error was 3%. The sum of the adsorption volumes of the pores ( ) was determined at P/P0=0.99 for nitrogen with a detection error of 6%. The synthesis products were analyzed by gas-liquid chromatography with a flame ionization detector under the following normal conditions: 15% lestosil in Light chrome -545 with a particle size of a fixed liquid phase of 0.250-0.315 Nm, a glass column measuring 2x0.004 m, column temperature 100 ° C, flow rate of gaseous nitrogen carrier 30 ml/min. Qualitative analysis was carried out by the method of ”witnesses", quantitative -by the method of internal normalization. To carry out the reaction of catalytic vinylation of acetic acid in the presence of acetylene, the total pore volume of the catalyst made on the basis of zeolites with a high silicon content is 0.3-0.41 cm3/g, the specific surface area is 50-170 m2/g.

1 Introduction

Today, special attention is paid in the world to research aimed at studying the activity of catalysts made on the basis of high-silicon zeolites with high catalytic activity, selected for the reaction of catalytic vinylation of acetic acid in the presence of acetylene, obtained by

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* Corresponding author: nfayzullaev1972@gmail.com

various methods [1-4]. Scientific substantiation of the solution of the following tasks for the creation and effective use of catalysts based on high-silicon zeolites with high catalytic activity, selected for the catalytic vinylation of acetic acid in the presence of acetylene to produce vinyl acetate: thermal stability, development of new energy-efficient and resource-saving methods for the synthesis of catalysts based on high-silicon zeolites with high catalytic activity, selected for the catalytic vinylation of acetic acid with high performance and selectivity in the presence of acetylene; determination of the dependence of textural and physico-chemical parameters, as well as structure on its catalytic activity, characterized by a decrease in coking of catalysts made on the basis of high-silicon zeolites selected for the catalytic vinylation of acetic acid in the presence of acetylene; Special attention is paid to the study of the kinetic patterns of the catalytic acetylation reaction using a catalyst based on high-silicon zeolites with high catalytic activity, selected for the catalytic vinylation of acetic acid in the presence of acetylene operating in a cyclic mode, to determine the kinetic model of the reaction and the mechanisms of its course [5-13].

Vinyl acetate (va) is one of the most important monomers (a component for the production of polymers in the organic synthesis industry). Vinyl acetate monomer is a substance with a wide range of applications. Vinyl acetate-based polymers and copolymers have good adhesion, optical, electrical insulation and fiber-forming properties, therefore they are widely used not only in everyday life, but also in industry: in engineering, construction, medicine and other fields [14-19].

In recent years, one of the directions for the development of catalysts for the acetylene acetylation reaction with acetic acid has been the production of catalysts with high catalytic activity and productivity using carbon as a carrier [20]. Carbon materials have several important advantages: a large surface area, the ability to regulate the distribution of pore sizes, and the ability to recover active metals from spent catalysts by burning carbon. An important advantage of carbon as a catalyst carrier is the inertia of the surface, which eliminates the occurrence of undesirable side reactions. Carbon catalysts are resistant to the coke layer. The carbon materials used as carriers vary in nature. These can be activated carbon, technical coal and formulations based on it. The advantages of carbon black formulations such as Sibunite compared with activated carbons are their chemical purity (C content of at least 99.5%), high strength and spherical shape of granules. The creation of materials with hierarchical porosity is currently one of the fastest growing areas of materials science. Due to the improved transport properties of the porous structure, such materials are widely used as sorbent carriers and catalysts. The stencil method of manufacturing hierarchical porous materials, among other approaches to creating hierarchical structures, is characterized by simplicity, versatility compared to its predecessors and the ability to strictly control textural properties, as a result of which the resulting material becomes mechanically durable.

By-products in the synthesis of vinyl acetate from acetylene and acetic acid are: croton aldehyde, ethylidenediacetate, acetone, acetaldehyde, polyvinyl acetate and methane. The selectivity of vinyl acetate to chemically reactive acetylene is about 95-99%, and to acetic acid - about 92-98%.

Today, the issues of improving the catalysts of the process of obtaining vinyl acetate from acetylene in the vapor phase, optimizing the technological scheme and reducing production costs and other new solutions leading to a reduction in the cost of vinyl acetate are relevant.

2 The experimental part

The dispersed properties of a catalyst made on the basis of zeolites with a high silicon content with high catalytic activity, selected for the catalytic vinylation of acetic acid in the presence of acetylene, were studied using a scanning electron microscope (JSM-6510 LV). The catalytic activity of the obtained sample was studied in the vinylation reaction of acetic acid under the action of acetylene.

Adsorption measurements were carried out in order to determine the parameters of the porous structure of the studied samples. The main adsorption and structural characteristics of the load - specific surface area, pore volume and effective size - were obtained as a result of the work and determination of the isotherm of water vapor adsorption.

From the adsorption measurement, the sample was treated in vacuum at 300 ° C for 8-10 hours. Adsorption was measured at room temperature. The adsorption balance was set for 22 hours. The relative error was 3%. The sum of the adsorption volumes of the pores ( ) was determined at P/P0=0.99 for nitrogen with a detection error of 6%.

The mass of the sorbent sorbed in 1 g of sorbent (a catalyst based on high-silicon zeolites with high catalytic activity, selected for the catalytic vinylation of acetic acid in the presence of acetylene) was calculated using the formula:

$$\frac{x}{m} = \frac{\gamma \cdot \Delta n}{m}$$

Δn - spiral elongation, mm;

m - the mass of the sample of the catalyst prepared on the basis of highly siliceous zeolites with high catalytic activity, selected for the catalytic vinylation of acetic acid in the presence of acetylene, g

γ - spiral divisions, g/mm.

The method of Brunauer, Emmett and Wyres and the equation were used to calculate the specific surface area:

$$\frac{\alpha}{\alpha_m} = \frac{C_{BET} \cdot P/P_0}{(1-P/P_0)[1+(C_{BET}-1)P/P_0]}$$

This is: P/P0 is the relative pressure, - the adsorption value; - the capacity of the monolayer; the energy constant of the equation C $_{BET}$

The comparative size of the surface S_{sol} (m^2/g) calculated by the following formula:

$$S_{Сол} = \alpha_{\mathrm{m}} \cdot N_A \cdot \omega$$

α_m - monolayer capacity; N_A – Avogadro number; ω - the area in which one adsorbate molecule is located (settles) in a filled monolayer, for nitrogen $\omega = 0{,}162$ nm^2.

The reaction was carried out by chromatography method. The synthesis of oils using ionization detectors in gas–liquid chromatography was carried out under normal conditions: particles of the liquid phase with particles of 0.250-0.315 Nm in size contained Chromochrome -545 with a 15% carbon dioxide content, with particles of 2x0.004 microns in size.when heated in a column to 100 °C, the greenhouse gas is nitrogen with a boiling point of 30 ml/min. Syphilis used the Gulliver method, a method of internal normalization. A mixture of acetylene and acetic acid with hydrogen acetate and cadmium acetate with acetic acid was selected for the catalytic reaction of vinyl chloride with hydrogen amalgam based on silicon zeolites as oxidation catalysts, shown in Figures 1, 2 and 3.

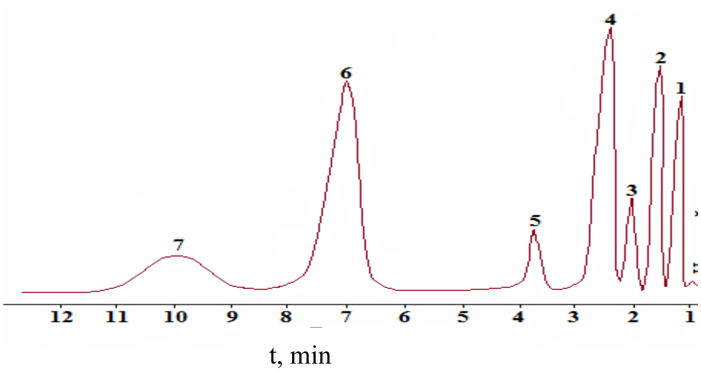


Fig. 1. Chromatogram of vinyl acetate raw materials obtained on the Ruk I acetate catalyst. 1-acetylene; 2-acetaldehyde; 3-acetone; 4-vinyl acetate; 5-crotone aldehyde; 6-acetic acid; 7-ethylidenediacetate.

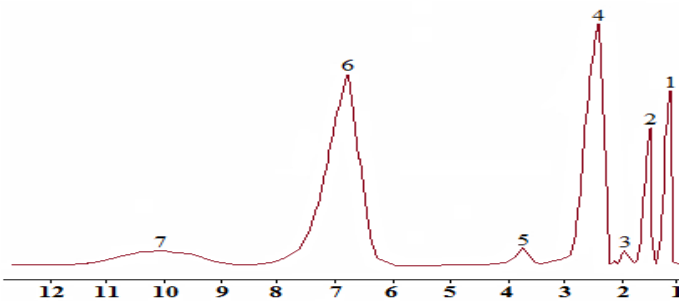


Fig. 2. A catalyst consisting of a mixture of zinc acetate and cadmium acetate was obtained using a chromatogram of vinyl acetate. 1-acetylene; 2-acetaldehyde; 3-acetone; 4-vinyl acetate; 5-crotone aldehyde; 6-acetic acid; 7-ethylidenediacetate.

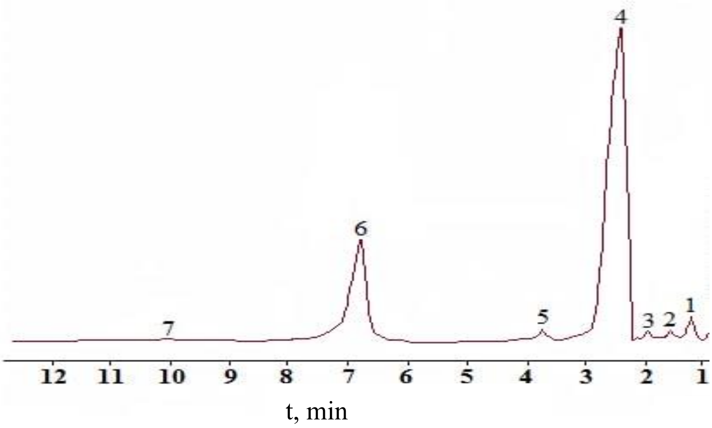


Fig. 3. 1-acetylene; 2-acetaldehyde; 3-acetone; 4-vinyl acetate; 5-crotone aldehyde; 6-acetic acid; 7-ethylidenediacetate.

3 The results of the experiment and their discussion

Acetic acid acetylene participates in the catalytic vinyl reaction of oshirish amalgam as a zeolite-based catalytic solution based on silicon dioxide and is being prepared for catalysis based on silicon dioxide with a total volume of 0.3-0.41 cm3/g, solishtirma sirte 50-170 m2/g.

Acetic acid acetylene with the participation of catalytic vinyl alcohol the oshirish amalgam reaction as a catalytic silicon solution based on EGA zeolite is a ready-made catalyst based on acetylene acetic alcohol containing a substance of the quality of SCC ishlatildi, uning sirte usasa 690-720 m2/g, total quenching volume-0.87-0.92 cm3/g, micro-enrichment volume-0.24-0.25 cm3/g. Granules with a diameter of 200-500 microns. Acetic acid acetylene undergoes a vinyl reaction of 170-2200 ° C at a temperature of 0.54-0.84 l/cm3 (kat) * h, acetic acid weighing 0.3-0.5 g/cm3(kat)*h under elevated conditions.

A catalyst prepared on the basis of highly siliceous zeolites with high catalytic activity, selected for the catalytic vinylation of acetic acid in the presence of acetylene, was prepared under the following conditions: a 5-25% solution of zinc acetate was absorbed by circulating absorption at a temperature of 60 ° C per day. microspherical porous medium of meso-size (SCS). The salt absorption time was changed within 60-90 minutes. Absorption carrier substance (YUKS): the solution was added in a ratio of 1:3-1:8.5. After the end of the absorption process, the catalyst prepared on the basis of high-silica zeolites with high catalytic activity, selected for the reaction of catalytic vinylation of acetic acid in the presence of acetylene, was dried at room temperature for 24 hours, and then in a drying cabinet at a temperature of 100-130 ° C (increase the temperature to 10 ° C every 1 hour). The zinc acetate content was 11-30% in the catalyst prepared on the basis of high-silica zeolites with high catalytic activity, selected for the reaction of catalytic vinylation of acetic acid in the presence of acetylene.

Under the above conditions, the service life of the catalyst prepared on the basis of high-silica zeolites with high catalytic activity, selected for the catalytic vinylation of acetic acid in the presence of acetylene, was 2000 hours.

As a source of zinc $Zn(CH_3COO)_2{\cdot}2H_2O$ A 0.20-0.25 M solution of zinc acetate was used. A 0.20-0.25 m solution of zinc acetate was prepared using ionized water. An aqueous-alcohol mixture was used to obtain an aqueous-organic mesophase. For this, a solution of a water-propanol mixture with a volume ratio of 50/50 was used. A solution of zinc acetate salt was obtained at a temperature of 60-70 °C for 20-30 minutes with constant stirring at a rate of 300-500 ail/min. As a structure-forming component, GMTA and DMOA lar have molar ratios of gmta/Zn and DMOA/Zn equal to 1-3. The ash mass was prepared by stirring at a temperature of 70-80 ° C in a magnetic stirrer for 40-50 minutes.

The resulting chemically active mixture was continuously stirred at a temperature of 96-98 ° C at a rate of 450 ml/min until a gel was formed. The resulting gel was placed in a porcelain dish and subjected to heat treatment for 1 hour.

Table 1. Size, phase composition and synthesis conditions of synthesized ZnO crystals based on X-ray diffraction data.

№	Starting saline	Solt stabilizers	The molar ratio		Crystallite size, ZnO, nm	Phase composition of the product, mass %
			Zn stabilizers	AcAc/Zn		
1	$CH_3COO)_2Zn$	GMTA	1	1	90-110*	ZnO-100
2	$CH_3COO)_2Zn$	GMTA	1	2	90-110*	ZnO-100
3	$CH_3COO)_2Zn$	GMTA	2	1	47	ZnO-100
4	$CH_3COO)_2Zn$	GMTA	2	2	62	ZnO-100
5	$CH_3COO)_2Zn$	GMTA	3	1	31	ZnO-100

6	$CH_3COO)_2Zn$	GMTA	3	2	41	ZnO-100
7	$CH_3COO)_2Zn$	DMOA	1,5	2	36	ZnO-100

ZnO crystals from zinc acetate (Table 1 sample) have a diameter of up to 50 Nm, consist of rods 300-800 Nm long, and the average crystallite size according to X-ray diffraction data is ≥ 36 Nm. Studies obtained by the N2 adsorption-desorption method have shown that ZnO powders heated at 5000c are meso-sized porous with a pore size of 7-9 Nm. The textural properties of mesoporous catalyst samples with high sorption properties were studied by adsorption of benzene vapor using a sensitive quartz spiral structure of McBain-Bakr. The benzene obtained as an adsorbate was first purified under vacuum conditions, frozen until its vapor pressure reached the vapor pressure indicated in the table for pure benzene, and the dissolved gases were not removed. On mesoporous catalysts with high adsorption properties, before measuring the adsorption of benzene vapor, each adsorption was evacuated to a residual pressure in the system of 1.33·10-3 pa and heated at 473 K for 6 hours. Then, the dependences of the amount of adsorbed substance on the pressure of this substance in the gas phase at a constant temperature were obtained (Figure 4).

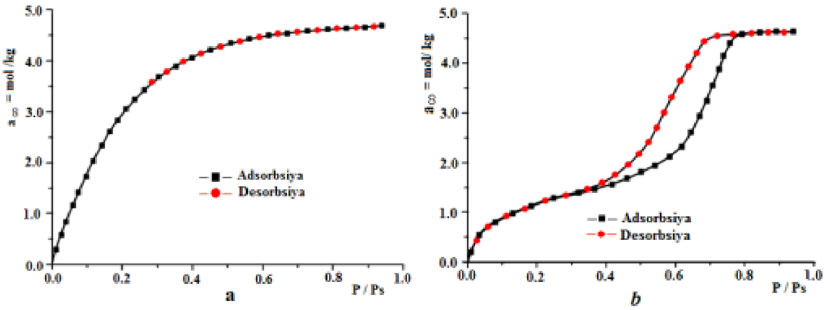
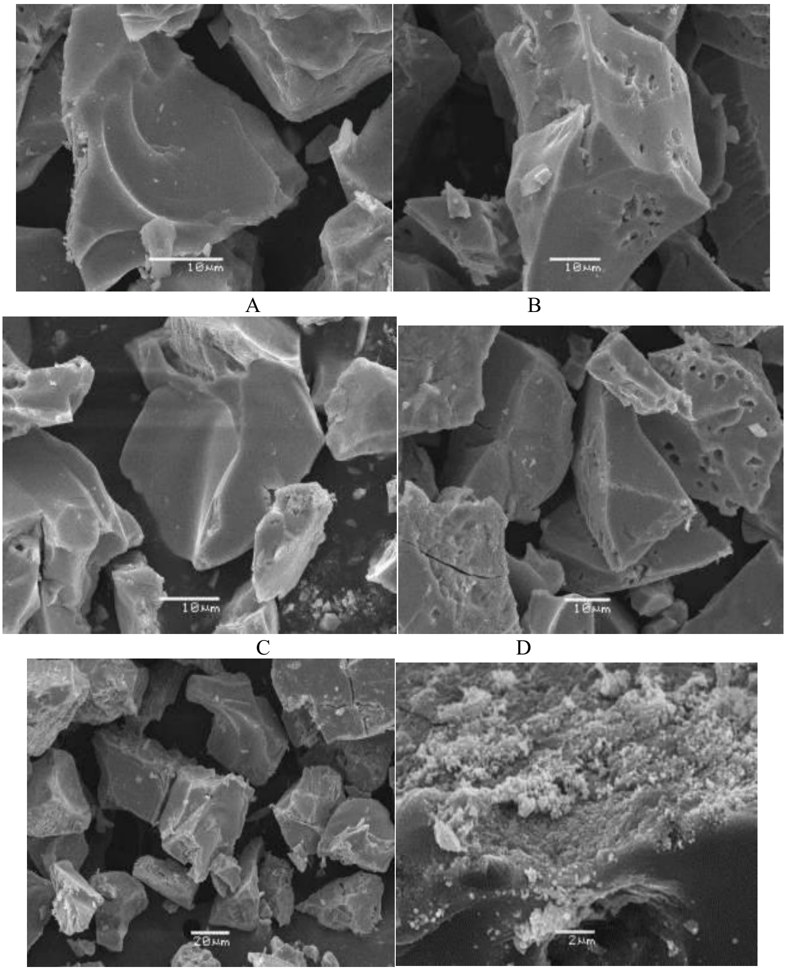


Fig. 4. Dependences of the amount of a substance adsorbed by benzene vapor in mesoporous catalysts with high sorption properties synthesized at 30 ° C (a) and 50 ° C (B) on the pressure of this substance in the gas phase at a constant temperature.

It was found that in a sample of a mesoporous catalyst with high sorption properties obtained at a temperature of 30 ° C, the adsorption of benzene vapor is higher than in a mesoporous catalyst with high sorption properties obtained at a temperature of 50 ° C. In the sample synthesized at a temperature of 30 ° C, it was observed that the amount of adsorbed benzene increases sharply from zero relative pressure to P/po =0.3 and approaches saturation at P/po =0.6, and then remains unchanged (Figure 4-a). The resulting sorption isotherm belongs to type IA according to the UYUPAK classification, and it was estimated that 60-75% of the total pore volume are micropores. However, in a sample of a mesoporous catalyst with high sorption properties synthesized at 50 °C, the adsorption of benzene vapor was observed with a sharp increase in relative pressure to P/po=0.2 and approaching saturation at P/po = 0.8 (Figure 4-b). We can see that adsorbed benzene vapor due to capillary condensation at P/po= 0.4-0.8, the adsorption and desorption lines combine to form a hysteresis ring. It follows from this that a sample of a mesoporous catalyst with high sorption properties synthesized at 50 ° C consists of mesoporous ones, and the dependence of the amount of adsorbed substance on the pressure of this substance in the gas phase at a constant temperature belongs to type H2 IV according to the UUPAC classification.

4 Influence of temperature on morphology and structure of ZnO pores

Similar studies were conducted for ZnO solids obtained by drying hydrosols at room temperature, obtained by drying gel at room temperature, with high porosity (15-50%) and high specific surface area (150-900 m2/g) with pore sizes in the range of 1-10 Nm (Figures 5-7). Figure 5 shows micrographs of ZnO solids obtained by drying the gel at room temperature at various annealing temperatures, with high porosity (15-50%) and high specific surface area (150-900 m2/g) with pore sizes in the range of 1-10 Nm.



A B

C D

E F

Fig. 5. Micrographs of samples of solids with high porosity (150-900 m2/g) and high specific surface area (150-900 m2/g) with pore sizes in the range of 1-10 Nm obtained by drying ZnO gel at room temperature, fired at various temperatures: 250 (a), 350 (b), 450 (c), 600 (D) and 9000C (E, F).

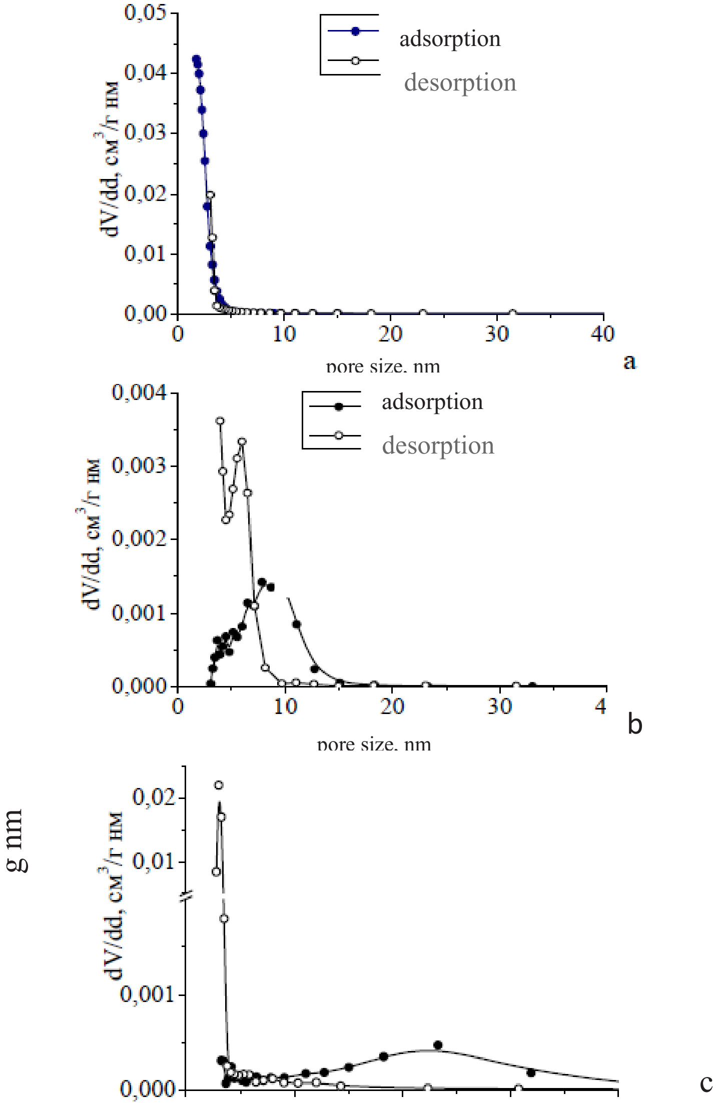


Fig. 6. Gel obtained by drying calcined Mgo at room temperature at various temperatures, differential pore size distribution curves for xerogel samples: 400 (a), 600(B) and 9000c (c).

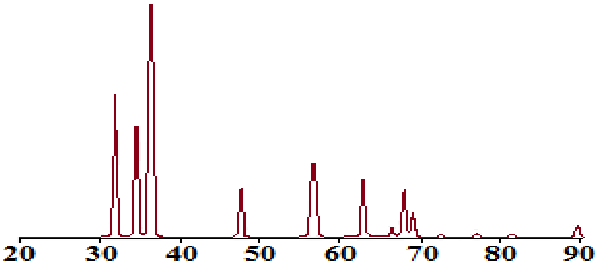


Fig. 7. Diffractogram of ZnO powders heated to 500 °C from zinc acetate.

When heated to 7000 C, the specific surface area decreases from 8.6 to 3.3 m2/g due to the mutual adhesion of particles (sintering). As a result, a new porous structure is formed, and the size of the mesosized pores is 10-50 Nm. Macro-tumors are also formed.

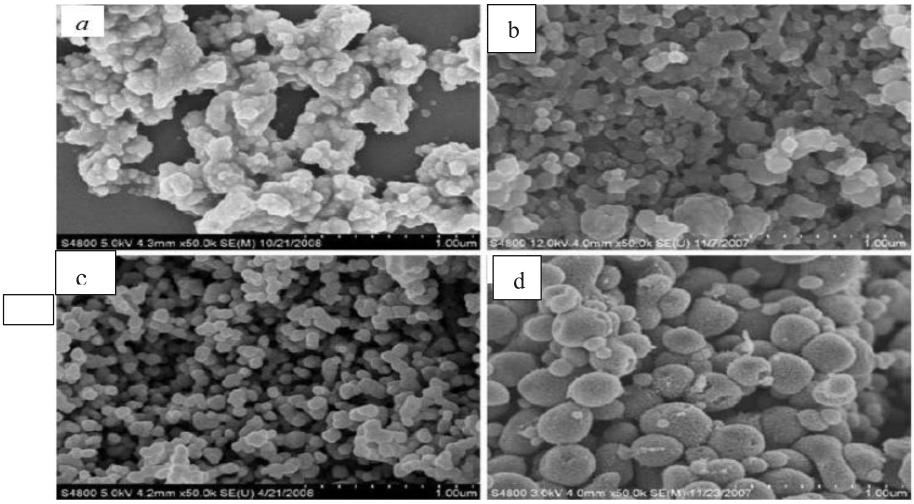


Fig. 8. ZnO micrograph taken with a transmission electron microscope using ethylenediamine (B), hexamethylenediamine (C) and citric acid when the structure-forming agent is not used.

Further analysis of the IR spectrum showed that as the sample heating temperature increases, changes occur in the IR spectra in the region of valence vibrations (2800-3900 cm -1) and in the region of deformation vibrations (4000-900 cm -1) (Figure 8).

In the IR spectra of small-sized ZnO samples heated to 400-750 °c, a maximum is observed, expressed only by in 450 cm-1. This is due to fluctuations in the Zn-O bond in the spectrum (Figure 9).

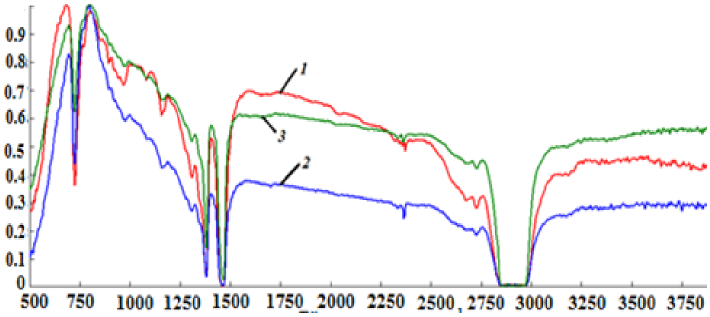
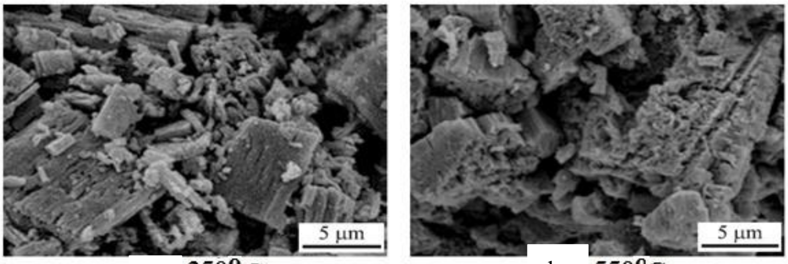


Fig. 9. At different temperatures, the meso-sized structured ZnO of IQ spectrum.

A 0.05 M solution of ZrO(No3)2·8h2o was used as a source of zirconium. The solution was prepared at 70-75°C at 650 rpm with constant stirring for 30 minutes. Monoethanolamine was used as a structure-forming agent. ZOL formation was prepared by mixing 30-35 minutes in a magnesium mixer at 90-95°C. The reactive mixture was then mixed at 90-95°C with a rate of 600 ayl/min to form gel. The resulting gel was put in a porcelain cup and thermally processed to 500°C for 1 hour.



a 350^0C b 550^0C

Fig. 10. Electron micrographs of mesoscale ZnO powders taken at various temperatures.

Figure 10 shows the morphostructure of zinc oxide obtained at 350 °C and 550 °C. A micrograph of the ZnO sample was taken using a JEOL JSM 5510 wave electron microscope. Apparently, synthesized mesoscale crystals are formed from the agglomerates, the average size of which increases with increasing firing temperature.

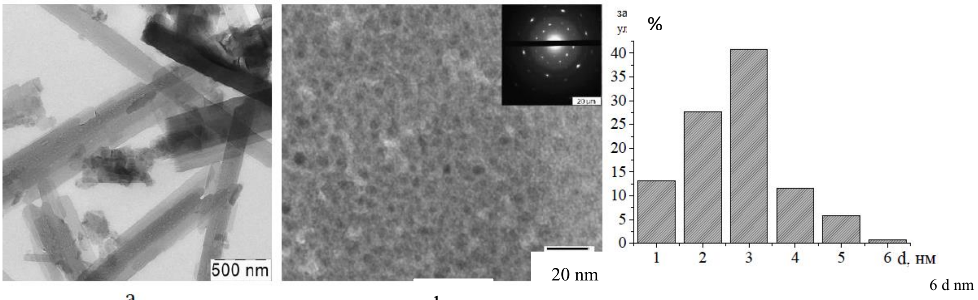
An analysis of the presented electron micrography shows that the firing process leads to a change in the specific surface area of zinc oxide with a mesosized structure, a change in the concentration of defects and, as a result, the width of the band gap of the material.

IR spectroscopy was used to study the processes occurring during the thermal combustion of ZnO. This method makes it possible to verify the qualitative and quantitative composition of mesoscale materials, determine the course of various observed reactions with changes in the concentration of chemical bonds, and determine the average size of mesoscale particles based on experimental data.

Salt of Zn(CH3COO)2×2H2O was used for the synthesis of mesosized zinc oxide. A 25% NH3-H2O solution was used as an oxidizer.

The "sol-gel" synthesis of zinc oxide in mesoscale technology involves the following steps:: 1) synthesis of zinc oxide sol; 2) conversion of Sol into gel; 3) centrifugation and washing of the resulting gel with distilled water; 4) drying and firing of the gel at a certain temperature.

Mesoscale zinc oxide gel samples were examined using beam electron microscopy (Tem). Figure 11 shows the micrograph taken.



a b c

Fig. 11. Histogram of the size distribution of meso-sized particles on the surface of zinc oxide plates and radiation of meso-sized particles with different magnification electron microscopy micrography.

Micrography analysis The structure of the zinc oxide gel consists of a sum of GI plates of different lengths (from 0.4 to 20 microns, width from 100 to 250 Nm, thickness from 5 to 15 nm).

The effect of the annealing temperature of small-sized zinc oxide samples on their spatial composition and structure, the synthesized samples were dried to 125 °C and fired from 175 °C to 750 °C for verification. All samples were then X-ray analyzed. The results of the X-ray phase analysis are shown in Figure 12 below.

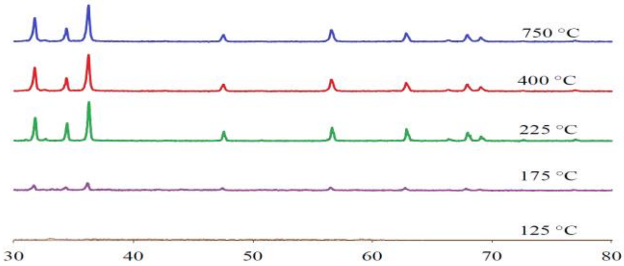


Fig. 12. Results of X-ray phase analysis of samples burned at 125°C - 750°C.

As can be seen from the diffraction pattern in the image above, ZnO samples fired at various temperatures have peaks characteristic of structural zinc oxide of the wurtzite type. In addition, the intensity of the peaks increases with increasing annealing temperature, which is explained by particle aggregation and disruption of the gel structure during rounding (sintering).

The porous structure of the solid support is of great importance for catalysis, since in most cases it determines the functional properties of the material. A well-developed pore system and specific surface area make it possible to achieve uniform distribution and dispersion of surfactants, its resistance to aggregation, as well as to ensure the supply of oxidizing reagents to the active component and the release of reaction products. Using the method of low-temperature nitrogen adsorption, it was shown that the specific value of the solid carrier of the block does not exceed 13.5 m2/g. The porous structure of the solid carrier is characterized by the presence of pores from 3 nm to 100 Nm, the volume of which is 0.065 cm3/g. According to mercury pyrometry data, in addition to mesoscale pores, macropores up to 200 microns in size are present in the holder, with a total pore volume of 0.22 cm^3/g.

Most of this volume is accounted for by pores up to 500 Nm in size (86%), of which pores in the range of 3-100 Nm occupy a volume of 0.07 cm3/g, which is consistent with the data of the low-temperature nitrogen adsorption method, which measures pores in the specified range**.**

5 Conclusion

Despite the small specific surface value, the porous structure of the solid carrier is considered to be sufficiently developed, and meso–sized pores, as well as small-sized macropores, are formed, while meso-sized pores ensure a uniform distribution of active components over the surface of the solid carrier, and macropores have high catalytic activity, selected for the catalytic vinylation reaction of acetic acid in in the presence of acetylene. It provides transport of oxidizing reagents to the active centers of a catalyst made on the basis of zeolites with a high silicon content.

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