Composite Sorption-Active Materials Based on Zeolite and Ethylene Fluorine Derivatives

Part I. Raw Materials, Production Technology and Research Methodology for Prototypes

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Abstract

Theoretical and experimental studies of promising technologies for the preparation of nanostructured composite adsorbents with a matrix structure based on crystalline NaX zeolite is carried out. The initial reagents (zeolite crystalline NaX by Industry Standard 05766575-2009, fluoroplastic powder-42 of grade "F-42V" by GOST 25428–82, acetone of reagent grade qualification by GOST 2603-79 and methyl ethyl ketone solvent as a rheological additive) and an adsorbent-filler-polymer matrix composition in the synthesis of sheet, granular and block composite adsorbents with a matrix structure have been selected. Methods for studying physical and chemical properties of samples of composite sorption-active materials have been developed. Methods of examining morphological and mechanical properties as well as thermal stability of samples are proposed. Methods of determining the isotherm and kinetics of water vapor sorption by materials' samples under static conditions and methods of measuring the dynamic activity of materials by water vapor and the intensity of air drying by the dew point are developed. The porous structure is studied and the bulk density of samples of composite sorption-active materials is determined.

Keywords

Adsorbent; composite sorption-active materials; physical and chemical properties; dynamic activity; tensile strength; polymer matrix; zeolite; Pressure Swing Adsorption (PSA).

Introduction

One of the promising areas in modern chemical technologies for adsorbents is the development of new composite sorption-active materials and adsorbents based on them with improved physical and chemical properties, and adsorption characteristics for pressure swing adsorption (**PSA**). PSA is widely used for separation and purification of multicomponent gas mixtures and protection of human respiratory organs from chemical and biological negative impacts.

Traditionally, the pressure swing adsorption is used to separate and purify gas mixtures, as a rule, using granular zeolite adsorbents (based on a mixture of zeolite and clay in the form of bulk charge). With prolonged use of adsorbing materials in PSA installations under the influence of thermal and aerodynamic loads and other phenomena, adsorbent materials are destroyed and a fine fraction (dust) is formed. This results in blocking the transport pores of the adsorbent and decreasing the dynamic activity of the adsorbent over the absorbed gas phase component

per unit mass, reducing the kinetic parameters of mass transfer in repeated "adsorption-desorption" cycles and adversely affecting the cleaned gases, pipelines, valves and other units of adsorption plants. The abrasion and dust formation of the adsorbent during its operation reduces the quality of cleaning and separating the multicomponent gas mixture in the PSA technology, which it requires high and stable rate of mass transfer processes of the sorbent over a large number of sorption-desorption cycles (the number of cycles can reach up to 150 per minute) [1–3].

Elimination of the above-mentioned deficiencies is possible when creating composite sorption-active materials (**CSAMs**) with a matrix structure [4–9]. In this case, the matrix should serve as a material binder, which determines its strength and plasticity as a whole under the influence of mechanical, aerodynamic, thermal and other loads [10–14].

The fluoride derivatives of ethylene

$$[-CF2-CF2-]n$$
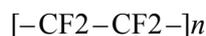
(fluoroplasts) are most suitable for the sorption-active materials, while zeolites of various grades, silica gels, alumina, etc. can be used as fillers (also, there might be combined with additives, activating the material or giving it specific properties) [15]. The third initial component in the synthesis of composite sorption-active materials is the rheological additive necessary to improve the plasticity of the polymer matrix/ adsorbent-filler system in the molding step of the crude product. It follows from [16–18] that beeswax, ethylene-vinyl alcohol, polyolefinglycols, etc. can be chosen as a rheological additive. However, after the molding step, a rather complex technological problem might occur, namely, the removal of a rheological additive from a molded adsorbent material. For these purposes, either thermal treatment, or solvent extraction, or their combination are the most commonly used.

CSAMs can be shaped into virtually any geometric form, which has flexible and elastic properties. Their composition, structure, dispersity, content in the composition fully determine the physical, mechanical and adsorption properties of the adsorbent, its strength characteristics.

It is possible to slow down the process of dust formation significantly when using the adsorbent if organic polymers with a high Young's modulus and capable of levelling the deformation stresses caused by the temperature drop and aerodynamic loads on the adsorbent granules are used as a binder. This will result in high values of mass transfer coefficients of sorption materials because of the developed secondary porous structure of CSAMs [19, 20].

The parameters of the porous structure, the adsorption and strength properties of CSAMs are regulated by applying several groups of factors, such as the nature, dispensability, composition of the initial adsorbent fillers and binder in the moldable mass, the nature of the bond between the adsorbent and binder particles, the molding conditions, the liquid phase removal modes and the subsequent final heat treatment of molded composite adsorbents [21–23]. In addition, it is important for adsorption materials used in human life support systems in extreme situations to have a sufficiently high modulus of elasticity in bending. This will make it possible to create product elements ready for immediate use (cartridges, units, etc., which can be inserted into protective equipment even in the field, which is especially important in emergency situations), while most domestic protective equipment does not have this function due to the use of granular adsorbents.

This research aims to conduct an experimental study of physical and mechanical and adsorption properties of sheet, granular and pre-fabricated CSAMs based on zeolite and ethylene fluorine derivatives for PSA processes of separation and purification of gas mixtures, as well as to develop practical recommendations on the technology of their production in industrial conditions

1. Basic Materials

The basic materials to produce CSAMs included the following reagents: NaX zeolite crystalline by Industry Standard 05766575-2009; powdered fluoroplastic-42 of F-42B grade by GOST 25428–82; acetone of reagent grade qualification by GOST 2603–79 and methyl ethyl ketone solvent [9, 19].

In the development of CSAMs, a solvent selected from the series of ketones – methyl ethyl ketone, dimethyl ketone (acetone) was used as a rheological additive. The use of the latter is preferably because it is good value for money. The use of acetone as a rheological additive provides a number of advantages in the synthesis of CSAMs. It is well known that to achieve high values of the capacitive and kinetic characteristics of the adsorbent materials being developed, the latter must have a developed structure of secondary pores formed, as a rule, during the drying of adsorbing materials following the forming stage [6, 23–25]. In the proposed technology, the solvent acts not only as a plasticizer in the process of forming a crude product, but also as a blowing agent at the stage of its removal, providing the CSAMs with a quite divergent structure of transport pores. In this case, the film of a polymer matrix made of fluoroplastic

on the surface of the adsorbent-filler particles does not have a continuity, which ensures free access of adsorbent molecules to the volume of composite sorption-active materials without creating a large resistance to the mass transfer process [9, 26].

It should be noted that all the above-mentioned initial reagents are products of the main synthesis of the Russian Federation companies. These products are manufactured on a large scale and have low cost, thus eliminating the problem of export dependence of the developed technology on an industrial scale, which is extremely important for providing the Russian Federation's military industrial sector with life support systems for humans against damaging factors of chemical and biological nature.

Type X zeolite in the dehydrated form has the following composition: $Na_2O \cdot Al_2O_3 \cdot SiO_2$, in which the molar ratio $SiO_2 : Al_2O_3$ can vary within 2.2–3.3. Each large cavity has four entrances formed by 12-membered oxygen rings with a diameter of 8–9 Å. The volume of a large adsorption cavity of NaX zeolite is equal to 822 Å3, small cavities have a volume of 150 Å3. The complexes of large and small cavities constitute unit cells, each containing 192 aluminum and silicon ions, as well as 384 oxygen ions; its volume is 7776 $Å^3$. It can hold up to 256 water molecules. Small cavities of type X zeolites are quite accessible for molecules of nitrogen and other gases. Therefore, the limiting adsorption volume of type X crystallite calculated from adsorption measurements is close to the one calculated using geometric dimensions of the unit cells, i.e. for NaX, the limiting adsorption volume is 0.356 cm^3/g [27].

In this work we used crystalline (crystallite) NaX zeolite for the production of zeolite adsorbents [28] manufactured at JSC "Salavatnefteorgsintez".

Fluoroplastic-42 [29, 30] is a copolymer of tetrafluoroethylene $(-CF2-CF2-)n$ with vinylidene fluoride $(CH2 = CF2)$, having high strength and chemical resistance, antifriction properties, resistance to light aging. It dissolves in ketones and esters and is used in the production of fiber from solution, extruded and molded articles, varnish coatings and films resistant to aggressive media at temperatures from minus 60 to plus 120 °C [31].

In this work we used a fluoroplastic powder of grade F-42B. It is not combustible (the oxygen index [32] is 60–70 %), it is insensitive to shock and friction, it does not have harmful effects on the human body during direct contact under operating conditions. When the fluoroplastic-42 is heated to 200 °C or higher, volatile products containing hydrogen fluoride, carbon monoxide, and vinylidene fluoride can be released.

The intensive decomposition of fluoroplastic-42 begins at a temperature above 360 °C. Fluoroplast-42 has a high atmospheric resistance, is resistant to bacteria, at 20–100 °C it is resistant to acids of any concentration, oxidizers (hydrogen peroxide), organic solvents, except for ketones and esters, in which it dissolves. Fluoropast-42 can be subject to any kind of machining.

2. Materials production technology

As is well known from the literature [23, 24, 33], in order to solve a specific practical problem with the use of adsorbing materials, the shape of the absorbent material, determined by the design of the article in which the adsorbent material is used, is of great importance. The use of adsorbing materials in life support systems allows for their operation in the form of various geometric configurations: granules, prefabricated units, sheets, etc. For example, when adsorbing materials are used to dehydrate air in descent space vehicles, sheets with a maximum area are used, when oxygen is concentrated from the surrounding medium for pilots, installations with granular adsorbents are used [9, 34, 35].

2.1. Technology for Production of Granular Materials

In the technology of manufacturing granular composite sorption-active materials, *NaX* crystalline was chosen as the initial adsorbent fillers, fluoroplastic of the "F-42B" brand was used as the polymer matrix, and acetone was used as solvent. The basic process scheme for the production of granulated CSAMs is shown in Fig. 1.

The process was carried out as follows. First, a dry blended composition was prepared by mixing fluoroplastic and powdered NaX crystalline zeolite, having a dispersion of 4–6 μm in a dry form in a closed reactor *3* equipped with a stirrer. The zeolite and fluoroplastic were fed by sector feeders *1* and *2*, respectively. The mass ratio of the adsorbent-filler/polymer was 75–25/90–10, respectively. To the resulting solid mixture from feed tank 4, solvent was added in an amount necessary to completely dissolve the binder and produce a homogeneous suspension. The amount of solvent was chosen on the basis of the proportion of 15–30 ml of acetone per gram of fluoroplastic, which was connected, on the one hand, with the desire to minimize the consumption of solvent per unit of the final product, and on the other hand, with the need to manufacture a slurry with specified density and viscosity. This allowed further injection molding of the adsorbent without significant technological difficulties.
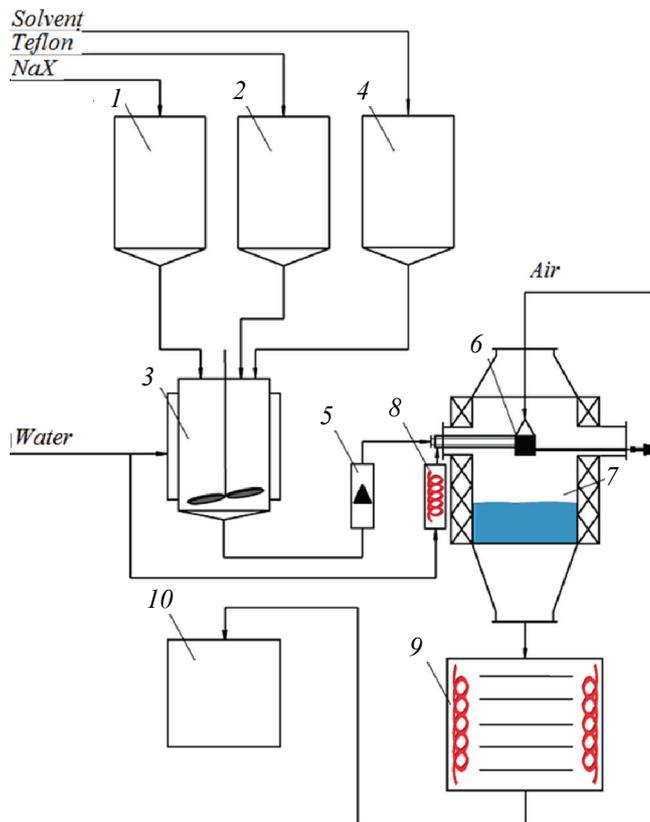
Fig. 1. Process scheme for the production of granulated CSAMs:
1, 2 – sector feeders for feeding zeolite and fluoroplastic,
respectively; *3* – reactor; *4* – feed tank for solvent supply;
5 – peristaltic pump; *6* – atomizer; *7* – water tank;
8 – heater; *9* – vacuum drying oven;
10 – finished product collection container

The resulting slurry of the zeolite powder and binder in the solvent was injected into a container *7* with heated water by means of a pneumatic atomizer *6*. The transport of the slurry from the reactor *3* to the atomizer *6* was carried out using a peristaltic pump *5*. The water temperature was maintained in the range of 50 to 90°C using an adjustable heater *8*. At this stage of the process, the solvent was removed from the initial slurry and the solid adsorbent. The resulting solid granules of the molded adsorbent were separated from the liquid using a screen. The size of the granules of the molded adsorbent was in the range of 1.0 to 4.5 mm, depending on the type of the pneumatic atomizer. The adsorbent granules were then heat treated in a vacuum drying oven *9* at a residual pressure of 1 kPa and a temperature of 140–180 °C for 4 to 5 hours to completely remove the solvent and water from the adsorbent pores. The finished sorbent was collected in the container *10*. It should be noted that the adsorbent granules obtained in such a manner are porous spheres of sufficiently regular geometric shape capable of elastic deformation.

2.2. Technology for production of sheet materials

Crystallite was chosen as initial adsorbent-fillers, fluoroplastic of F-42B grade was used as a polymer matrix, and acetone was used as a solvent. A basic process scheme for manufacturing flexible composite sorption-active materials is shown in Fig. 2.

The technological process was carried out as follows [9]. First, a dry blended composition was prepared, for which the powdered adsorbent filler was mixed with a fluoroplastic in a closed reactor *4* equipped with a stirrer. The mass ratio of adsorbent filler/polymer was 75–90/25–10. The adsorbent and fluoroplastic were fed with sector feeders *3* and *2*, respectively. A solvent in an amount necessary to completely dissolve the polymer matrix and obtain a homogeneous slurry was added to the resulting solid mixture from sector feeder *1*. The amount of solvent was based on the proportion of 20–40 ml of acetone per gram of fluoroplastic, which is associated with the need to obtain a slurry with given density and viscosity values for the further formation of sheet CSAMs.

The resulting slurry of the adsorbent and binder in the solvent from the reactor *4* was pumped through a peristaltic pump *5* and uniformly distributed over the glass substrate *6*. The amount of the slurry distributed over an area of 10 cm^2 of the substrate was in the range of 1.5 to 2.5 ml.
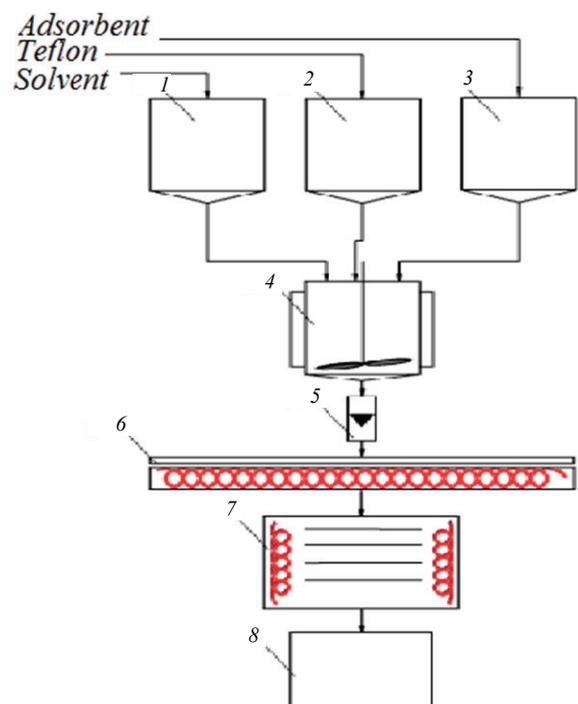

Fig. 2. Process scheme for the production of sheet CSAMs:
1, 2, 3 – sector feeders for feeding solvent, fluoroplastic and
adsorbent fillers, respectively; *4* – closed reactor; *5* – peristaltic
pump; *6* – glass substrate; *7* – vacuum drying oven;
8 – finished product collection container

For a better evaporation of the solvent and separation of the finished sheet materials from the glass substrate *6*, the latter was lubricated with a thin layer of medical vaseline before use [36] and heated to a temperature of 40–50 °C. The finished sheet KSAM was wetted with water, and then it was separated from the surface of the substrate. In order to better evaporate the solvent from the surface of the material and to reduce the drying time, the material was produced in a gas stream. Then, the sheet of CSAM was heat treated in a vacuum drying oven *7* at a residual pressure of 1 kPa and a temperature of 100–110 °C for 2 hours, and then at 140–160 °C for 2–3 hours to completely remove the solvent and water from the pores of the sorbent material. The finished composite sorption active material was collected in a container *8*.

The thickness of the produced sheet composite sorption-active materials was in the range 0.2–0.4 mm, which was determined by the amount of slurry per unit area of the substrate.

The produced sheet CSAMs were thin, porous elastic sheets with a homogeneous structure, from which shapes of various geometric configurations determined by the conditions for further exploitation of the produced materials were cut out.

2.3. Technologies for production of materials of other geometric shapes

It should be noted that the initial system for the formation of composite sorption-active materials, which is a slurry of adsorbent fillers and fluoroplastic in various ketones, allows for the formation of a rather wide range of geometric shapes by using different techniques. To produce composite sorption-active materials in the shape of filaments, the electrostatic spinning technique [37–39], which is widely used in the production of non-woven filter materials (Petryanov's filters) is possible. In this case, the structure of the produced CSAMs resembles beads, where the beads are particles of the adsorbent filler, and the role of the filament with a thickness of 1–3 μm is the polymer matrix.

Among the technological processes for manufacturing composite sorption-active materials in the form of gas permeable units, the most promising method is the duplication of the mesh-cellular structure of a foamed polymeric matrix of various composition and its subsequent removal by thermal destruction [40]. A fixed foamed structure of an openly cellular polyurethane foam and a freely flowing foam of various surface acting agents were used as a structure-forming matrix.

To obtain stable slurries of zeolite crystals, aqueous solutions of high-molecular stabilizing additives were used as the liquid phase: methylcellulose, sodium carboxymethylcellulose, polyvinyl alcohol in an amount of 1 to 10 % by weight. Polyurethane foam was used as a structure-forming base with a cell diameter of 0.5 to 2 mm [41]. The preforms were cut into samples of specified dimensions and impregnated in the prepared slurry of the zeolite and binder. Impregnation was carried out in the bath by the method of immersion and deformation of preforms. After impregnation, the excess liquid phase was removed and its distribution throughout the volume of the preform was leveled by pressing. The degree of spin was adjusted to ensure a given density of the preform. Wet preforms were dried in a stream of heated air at a temperature of 25–40 °C and a flow rate of 0.5–2 m/s. The polyurethane foam was removed from the preforms by heating at a specified rate of 200 to 650 °C for 4.5 hours. To maintain the shape of the preform, to prevent cracking when heated to the final sintering temperature, an intermediate isothermal holding treatment was performed for one hour at which the sample shrinkage occurred. Organic residues were removed by uniform heating of the preform. The temperature of the intermediate isothermal exposure was selected depending on the type of binder used, and for clays, it was 450–550 °C.

In the production of preforms a glue-foam froth foaming agent was used [42] as foaming systems. To increase the frothiness, a foaming activator was added to the foaming agent, and foam stabilizer was used to increase the foam stability. To form a foamed structure in a paste containing zeolite, a gas filler, which was hydrogen peroxide, was added. A suitably prepared composition was subject to swelling and solidification to form a cellular, highly permeable macroporous monolithic structure. In order to accelerate the curing process, a hardener was added before the start of the process. Further, the preform was subject to preliminary drying and high-temperature calcination for the purpose of sintering and removing organic components, and treatment with an alkaline solution to increase its mechanical and adsorption properties [43].

3. Methods for studying the physical and chemical properties of materials

3.1. Morphological and mechanical studies

Morphological and mechanical studies of the obtained CSAMs were carried out in the education and innovation center "Nanotechnologies and Nanomaterials" of the State Educational

Institution of Higher Professional Education "G.R. Derzhavin Tambov State University" [44, 45]. The scanning electron microscope Neon 40 (Carl Zeiss, Germany), coupled with the energy dispersive X-ray spectrometer INCAx-act (Oxford Instruments, UK), were used as tools for morphological studies.

The calibration of scanning electron microscopy elements and systems [20, 45] was carried out immediately before each series of experiments using a test sample (tin on carbon). The energy-dispersive X-ray spectrometer was systematically calibrated against a reference cobalt sample with a passport purity of 99.995 %. For morphological studies, samples of CSAMs with linear dimensions of ~5 mm × 5 mm × 5 mm were used.

Mechanical tests were carried out on a multifunction servo-hydraulic test machine MTS 870 Landmark (MTS Systems, USA), designed to determine the strength and ductility of materials. The experiments were carried out in a constant strain rate regime, which in all compression and tensile tests was 10 μm/s. The force response of the samples resulting from their mechanical deformation was recorded with a force sensor having a relative measurement error of 0.05 %. In compression tests, bulk samples of CSAMs were prepared in the form of a rectangular parallelepiped with an aspect ratio of height to width (length) equal to 3:1. In all tensile tests, the width of the test samples was ~10 mm with a characteristic thickness of ~0.2–0.4 mm.

3.2. Thermogravimetric and differential thermal analysis

Thermogravimetric and differential thermal analysis of the samples was carried out under non-isothermal conditions at the TAG-24 system (Setaram, France). To study the thermal stability of each sample of composite sorption-active materials, five consecutive experiments were carried out using thermal analysis. Samples with a mass of 20 to 80 mg and a dispersion of 4–6 μm (NaX crystallite) were studied. This ensured uniform heating of the sample in the course of the experiment and excluded the occurrence of the effects on thermograms, indicating the transition from the non-equilibrium state to the equilibrium state and capable of influencing the correctness of the results obtained [50]. The studies were carried out in air at atmospheric pressure in the temperature range from 20 to 400 °C. The choice of conditions for carrying out the thermal analysis was due to the fact that thermal regeneration of zeolite-based adsorbents occurs under the given conditions [51, 31, 32, 36–39, 44, 45, 50]. The temperature was recorded with an accuracy of 0.01 °C, changes in the mass of the samples were recorded with an accuracy of 0.01 mg. The samples under study were placed in a corundum crucible, chemically inert with respect to the samples. To measure the temperature, a platinum-rhodium thermocouple was placed in the volume of the sample under study and graduated to the standard reference points. The studies were carried out at a heating rate of 5 to 10 degrees per minute. This heating rate ensured a constant temperature across the entire volume of the samples under study during the experiment. At a sample heating rate of less than 5 deg/min and above 12 deg/min, there was a significant deviation in the curve of the temperature change from the straight line, leading to a significant error in calculating the kinetic parameters of the process under study. This experimental fact is explained by a violation of the temperature equalities between the outer and inner layers of the sample weighed due to the insufficient thermal conductivity of the materials [52–58].

3.3. Determination of adsorption isotherms of water vapor materials

The essence of the method is to determine the weight gain of the active sample after holding in a desiccator with a certain humidity. The desiccator method is based on the assumption that using solutions of certain salts it is possible to maintain a certain humidity in the desiccator. According to the reference data, several desiccators with different humidity were prepared using saturated solutions of zinc chloride, potassium acetate, potassium carbonate, sodium bromide, sodium chloride and copper sulfate.

Samples of composite sorption-active materials of mass ~0.1 g were heat treated in a vacuum drying oven at a residual pressure of 1 kPa and a temperature of 100–110 °C for 2 hours, then at 140–160 °C for 2–3 hours to completely remove the solvent and water from the pores of CSAMs.

The hot adsorbents were then discharged into the weighing cups, capped and cooled to room temperature. The mass of empty cups for weighing with lids was determined beforehand with an accuracy of ±0.0001 g. After cooling, the cups with lids and weighed sorbents were weighed with an accuracy of ±0.0001 g and placed in desiccators with a certain humidity, after removing the lids. The cups were left in the desiccators until a constant weight was established, determined by periodic weighing. The first weighing was performed after 24 hours.

Sorption activity a, mg/g, was calculated by the formula:

$$a = \frac{m_2 - m_1}{m_1 - m} 1000,$$

where m is weight of an empty cup, g; m_1 is weight of a cup with a sample weight, g; m_2 is weight of a cup with a wet sample, g; 1000 is conversion coefficient.

3.4. The study of the kinetics of water vapor sorption by materials under static conditions

The essence of the method is to determine the change in the weight of the sample of CSAM in time due to the sorption of water vapor. The active sample of the absorbent material with a mass of ~0.1 g was placed in desiccators with relative humidity of 10, 20, 44, 60, 75 and 98 % at a temperature of 20–2 °C. Periodically the sample was weighed on analytical scales. The amount of the adsorbed substance (a, mg/g) over a fixed time interval was determined by the formula:

$$a = \frac{m_i - m}{m} 1000,$$

where m_i is the sample weight g over a time interval t_i, min; m is the weight of the original sample, g; 1000 is conversion coefficient.

On the basis of the obtained experimental data, the kinetic curves of water vapor absorption were constructed by samples of composite sorption-active materials under different conditions.

3.5. Determination of the dynamic activity of materials by water vapor and the intensity of air drying by the dew point

The method consists in determining the intensity of air drying by a composite sorption-active material under dynamic conditions and determining the dynamic activity of the produced materials. Fig. 3 shows the diagram of the experimental stand for the investigation of composite sorption-active materials under dynamic conditions for water vapor.

The tests were carried out under the following conditions:

– specific velocity of the steam-air mixture (1,2 0,02) dm³/(min cm²);

– humidity concentration from 5 to 8 mg/dm³;

– temperature of the steam-air mixture (20±5) °C.

When determining the dynamic activity of granular composite sorption-active materials, the samples were placed in a dynamic tube. In determining the dynamic activity of sheet composite sorption-active materials, we proceeded as follows. Before carrying out the tests, a sheet (400±10) mm in length and 280.0 mm in width was cut from BmD-K glass paper. Immediately prior to testing, a sheet of BmD-K glass paper was applied to a whole sheet of CSAM and wound onto a metal or glass rod. The rolled sheet of CSAM with BmD-K glass paper was placed in a dynamic tube 5. To reduce the air rushing through the center, the ball from the BmD-K glass paper was closed with a center on both sides.

Dynamic tube 5 with samples of composite sorption active materials was placed in a vacuum drying oven, and samples were dried at a temperature of (100±10) °C and a residual pressure of 1 kPa for at least 2 hours. Then the temperature was raised to (140±10) °C at a residual pressure of 1 kPa, and samples dried at this temperature for at least 2 hours to remove moisture.

At the end of this time, dynamic tube 5 filled with CSAMs was taken out of the vacuum drying oven, the inlet and outlet apertures were closed with stoppers and allowed to cool to room temperature. Before the test, the plugs from the inlet and outlet of dynamic tube 5 were removed and connected to the stand. Air from air source 1 through Drexel bottle 2 half filled with water entered dynamic tube 5. The air flow was stabilized using rotameter 6: through dynamic tube 5 within (7.0±0.1) dm³/min, through psychrometer 3 (with an internal diameter of 17–18 mm) within (7.5±0.5) dm³/min, through the
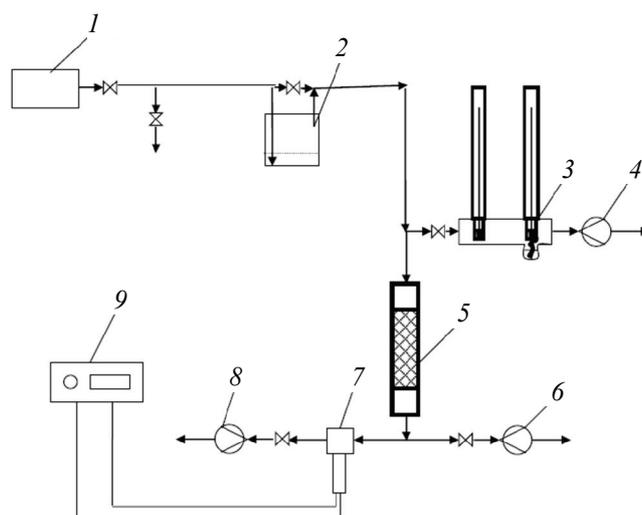

Fig. 3. Stand for determination of the dynamic activity by water vapor of composite sorption-active materials:
1 – air source; *4, 6* and *8* – rotameter; *2* – Drexel bottle; *3* – psychrometer; *5* – dynamic tube; *7* – sensor of the gas humidity meter IVG-MKS; *9* – gas humidity meter IVG-MKS

sensor of the gas humidity meter IVG-1 MK-S 7 within (1.0 ± 0.1) dm^3/min, respectively. Absorption of moisture from the air was monitored by the indications of the gas humidity meter IVG-1 MK-S9 in the mode for measuring the intensity of air drying by the dew point in degrees Celsius.

The moment of air intake from source *1* into dynamic tube *5* was considered the time of the beginning of the tests. Indications were taken after 1 min. When the dew point temperature reached above -75 °C, the tests were stopped. The dynamic tube *5* was detached from the installation and were closed with the plugs.

The dynamic activity (a, %) was determined by the change in the mass of the dynamic tube with CSAM after tests according to the formula:

$$a = \frac{m_2 - m_1}{m_1 - m}100\%,$$

where m is weight of empty dynamic tube, g; m_1 is weight of dynamic tube loaded with CSAM before testing, g; 1 m_2 is weight of dynamic tube loaded with CSAM after testing, g.

3.6. The study of the porous structure of composite sorption-active materials

The porous structure of the obtained composite sorption-active materials was studied in the educational and innovation center "Nanotechnologies and Nanomaterials" of the State Educational Institution of Higher Professional Education "G.R. Derzhavin Tambov State University" [44, 45]. The adsorption-desorption isotherms of nitrogen vapors at the temperature of liquid nitrogen (-195.8 °C) in the range of relative pressures from 0.0025 to 0.995 were carried out on the NOVA-1200e sorption meter (Quantachrome Instruments, USA) [59]. Degassing of the samples was carried out for 2 hours at a temperature of 140 °C under vacuum conditions.

The data was recorded and processed using the NovaWin V.11.0 software supplied with the instrument, using the Dubinin-Radushkevich model equation [24, 60–62] with a similarity coefficient (affinity) of 0.3918 and optionally with an affinity coefficient of 1 (standard steam –nitrogen).

3.7. Determination of the thickness of sheet composite sorption-active materials

The thickness of flexible adsorbent materials was determined using a micrometer with a scale of 0–25 mm [63].

To determine the thickness, a sample of any size was taken and the thickness of the sample was determined using a micrometer. Measurement was carried out at a minimum of three points of the sample. The arithmetic mean of all the tests was taken for the results of the studies.

3.8. Determination of bulk density of composite sorption-active materials

Bulk density was determined according to the procedure in [64]. When determining the bulk density [65–69], compaction of the layer of adsorbent material was carried out manually. To this end, in a preliminarily weighed cylinder with a capacity of 100 cm^3, three to four doses were poured into the adsorbent material, continuously tapping the cylinder with a rubber tip.

The compaction was carried out until the volume of adsorbent material did not cease to change.

Conclusion

The results of the review of theoretical and experimental studies of promising technologies for the preparation of nanostructured composite adsorbents with a matrix structure based on crystalline NaX zeolite made it possible to formulate and solve (Part II: Experimental Studies of Physical and Chemical Properties of CSAMs and Process Modes for Their Preparation) the following problems: 1) experimental determination of the optimal adsorbent filler-matrix compositions in the synthesis of CSAMs based on zeolite and ethylene fluorine derivatives, and the possibility of using these materials for human life support systems; 2) identification of the relationship between the synthesis conditions and the main operational characteristics of CSAMs; 3) complex studies of physical and chemical properties of synthesized CSAMs (resistance to thermal and mechanical effects, adsorption and kinetic characteristics, morphology, etc.); 4) research into properties and modes of the process of manufacturing CSAMs based on zeolite and ethylene fluorine derivatives.

In part 2, we will conduct experimental studies on the methods of obtaining composite sorption active materials based on crystalline NaX zeolite using organic polymers of fluorine derivatives of ethylene as a binder. A scientifically justified choice of the adsorbent-filler-polymer matrix composition in the synthesis of sheet, granular and block composite

sorption active materials will be made and the influence of the types, the ratios of the initial components and the molding conditions on the mechanical and adsorption properties of the composite adsorbent, will be investigated experimentally.

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