UDC 66.021.1:532.5,661.15,661.15 66.021.1:532.5
No of state registration 0120U100476
Inv. No

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REPORT
ON RESEARCH WORK
Technological bases of multistage convective drying in small-sized devices with utilization and heat recovery units
CREATION OF THEORETICAL BASES OF DRYING IN MULTISTAGE SHELF DEVICES WITH VERTICAL SECTIONING OF WORKING SPACE
(intermediate)

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2020

The manuscript was complete on November 18, 2020.

The results of this work were considered by the Scientific Council, Minutes from 2020.11.25 No. 4

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ABSTRACT

Report on research work: 42 p., 17 figures, 3 tables, 3 references.

DIRECTIONAL MOTION OF THE FLUIDIZED BED, DRYING, MULTISTAGE DRYER, PERFORATET SHELF, HYDRODYNAMICS, KINETICS

The following new *scientific results* were obtained during the research:

1. The model of calculation of hydrodynamic indicators of movement of streams in the multistage shelf dryer is created.

2. A model for calculating the kinetics of changes in temperature and humidity parameters of the dispersed phase and the drying agent at the stage of the dryer is created.

3. Theoretical data and data of experimental researches concerning the description of processes of heat and mass transfer during drying are received.

4. The main hydrodynamic modes of operation of shelf dryers are determined.

5. Data on the mechanisms of motion control of the dispersed phase in the dryer in different hydrodynamic modes are obtained.

The practical significance of the obtained results. Based on the received data, the technique of engineering calculation of shelf dryers with vertical sectioning of working space, and also a technique of an estimation of energy efficiency of convective dryers is created. An improved design of a multistage shelf dryer is proposed, which allows controlling the residence time of the dispersed phase in the volume of the apparatus, ensures uniform contact of the drying agent and dispersed material in the suspended layer mode, avoids unwanted changes in the properties of dispersed material (overheating, abrasion, cracking reduces the probability of removal of the commodity fraction of the dispersed material from the boundaries of the inclined perforated contact shelf. Experimental samples of porous ammonium nitrate were obtained, which passed the final drying stage in a gravity shelf dryer and are characterized by a developed porous surface. The obtained scientific results are prepared for implementation in the

implementation of economic agreements. A project proposal for participation in the competition of joint Ukrainian-Czech research projects for implementation in 2021 - 2022. Together with representatives of Alexander Dubcek University of Trencin (Slovakia), as well as the University of Pardubice and J.E. Purkyne University in Usti nad Labem (Czech Republic) based on the obtained theoretical and experimental data is the development of a software package for calculating the technological parameters of the gravity shelf dryer and its design dimensions.

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INTRODUCTION

Report section "Introduction" is prepared in according to data [1,2].

Among various dispersed flows, fluidized bed plays the most significant role in modern technology. The fluidization technique has become widely used due to high intensity of processes.

Disadvantages of the fluidized bed device can include: different residence time of a particle in the device, a need for thorough cleaning of the exhaust air and material entrainment (in particular it is applicable to small particles of the system). In addition, a significant drawback is the return of fine particles back in the fluidized bed zone. The analysis of various fluidized bed granulation equipment in chemical (Caiyuan et al., 2004), food (Patel et al., 2011) and pharmaceuticals (Agrawal and Naveen, 2011) industries showed the urgent need to organize the mutual flow motion, which will enhance the quality of the final product.

It is rational to use the same device for multiple processes in low-tonnage and multi-assortment production in order to reduce the range of the equipment. Implementation of the new forms to organize mutual flows motion (while keeping the same principles of the fluidized phases contact), which would intensify processing of the dispersed materials without considerable increase in the energy costs, is a promising direction for the development of heat and mass exchange processes in the heterogeneous systems.

Among the major techniques of controlling the polydisperse particles residence time in the device, one should notice the following:

1. To develop a direct movement of particles using accelerating elements (gas distributors of vortex type).

2. To design the device with a variable cross-sectional area applied to the granulation, cooling and dedusting process.

3. The promising direction to reduce financial and energy costs on the heat and mass transfer processes in the fluidized bed is the application of sectioning (vertical

and horizontal) to create different conditions for the particle heightwise (lengthwise) motion in the device.

There are various possibilities to apply suspended layer (fluidized bed, weighted layer). There are many successful cases regarding the introduction of this method in industrial practice, while in others it is at the stage of laboratory research. The processes taking place in heterogeneous systems using the suspended layer method have a great industrial application. It can be used to stove sulphide, arsenic and antimony ores to facilitate the extraction of gold or silver, for pyrite and pyrrhotine roasting to obtain SO2 in the production of sulfuric acid.

The suspended layer is in great demand in metallurgy for stoving of copper, cobalt and zinc sulfide ores to obtain valuable metals.

Fluidized bed devices for drying solid materials (coal, cement, limestone, etc.) are known in the whole world. Economic considerations make the use of these devices particularly interesting when large-tonnage materials are to be processed. A suspended layer dryer can also be used as a classifier since the drying and classification processes take places simultaneously in the device.

The world practice of recent years in the small power engineering has shown that the fluidized bed technology is effective for the utilization of different biomass types, industrial waste and effluents.

The development and implementation of fluidized bed devices are held back in many industries by the lack of reliable methods for calculating them. One of the main reasons is the lack of knowledge of complex and varied processes in the fluidized bed.

When constructing a mathematical model, one should take into account that there is a huge amount of particles in the operating device. These particles are of different sizes and they move at a different rate in all directions.

Mathematical modeling lets to establish and to optimize mode and technological parameters of the investigated process, as well as to minimize the use of additional automation tools.

The flows motion hydrodynamics modeling is carried out by the finite-volume method (a numerical method of the differential equations system integration in the differential derivatives). An important benefit of the finite-volume method is that the conservation law of integral values (of the flow rate, of motion quantity) is implemented on every chamber of the computational grid, but not only within it, due to the strong thickening of the computational grid. A closed area of the fluid or gas flow is selected in the calculation. The macroscopic values fields (in this paper – velocity), describing the environment in time and satisfying the definite laws, formed mathematically, are searched for it. The conservation laws in Eulerian variables are mostly used. This model is the most common and the most complicated among multi-phase flow models. The substance of every phase is a solid phase, and the substance motion of every phase is modeled with Navier-Stokes (Reynolds) equations system, equation of continuity and energy. According to this model, the motion equations for every phase, are solved together.

Modern manufacturing requires development of the new energy- and resource-saving technologies for the disperse materials drying in order to obtain high quality products, to increase the specific production capacity, to reduce the size of equipment, and to intensify the process. Therefore, the scientists should deal with the topical task, that is the improvement of the drying methods and the design of efficient dryers.

Various energy-consuming methods are applied for the disperse material drying.

Even though energy-consumption in convection drying is not the lowest, the convection dryers are often used for the disperse materials due to numerous advantages. An active hydrodynamic regime used in such dryers contributes to the intensification of the process without reducing its cost efficiency, and it has the following advantages:

- the hydrodynamic sustainability of the process;
- an increase in the relative velocity of the interacting phases motion;
- a developed surface of the contacting phases interaction;

- the approximation of a hydrodynamic model of flows in the apparatus to an ideal displacement model;

- the reduced energy-consumption of the process and a lower specific amount of metal in apparatuses.

The choice of such a dryer is justified by its productive capacity, energy and construction costs, work safety, the reliability, the possibility to control technological parameters, the maintainability and the availability of the appropriate transporting equipment.

Convection drying requires transporting of the large volumes of the drying agent. It implies big financial and ecological problems – significant losses of heat energy with the waste drying agent, particularly during the drying of materials with a high moisture content. As a result, these losses make 70 % out of total losses during the drying. It involves a need for utilization and re-use of heat of the waste drying agent.

The drum dryers and apparatuses with a boiling layer are of large sizes and highly energy-consuming. The pneumatic tube dryers do not ensure the necessary contact time of the damp material with the drying agent, and they are characterized by a high altitude . A perspective direction in the improvement of the drying equipment is a design of the combined apparatuses with an active hydrodynamic regime, which ensures the intensified heat exchange and minimal energy expenditures for the fluidized bed. An effective method of the process intensification is the organization of local, counterflow and/or combined regimes of the interaction of the drying agent flow with the product, and a selection of an optimal method of the drying agent inlet to the workspace of the apparatus and its re-use.

The literature analysis of the author presents the following measures on how to reduce the energy costs for the disperse materials drying:

- to re-use heat of the waste drying agent;

- to improve the construction of the dryers;

- to improve the dryer layout and the efficiency of their use (for the multistage dryers – the drying efficiency on each stage);

- an introduction of the improved technology of a recycle dryer;

- a differential thermal regime;

- to reverse and to recirculate the drying agent, including the waste one.

There has been little research reported on the patterns for the dehydration of the materials upon their multistage contact with the drying agent, which changes its properties. The intensity of dehydration differs on each stage of the shelf dryer, and the drying kinetics on each stage must be additionally examined.

Due to repeated contacts of the disperse material with the drying agent in the gravitational shelf dryers, a reduction in energy consumption per unit of the extracted moisture is achieved. It is of special interest to study the moisture removal efficiency on each stage of the dryer under different organizations of the drying agent motion.

1 MODELING OF THE AERODISPERSE SYSTEMS HYDRODYNAMICS IN APPARATUSES WITH DIRECTIONAL MOTION OF THE FLUIDIZED BED

This section is prepared in according to data [1].

In order to carry out modeling, it is convenient to solve the above equations by the numerical methods (DEM - Discrete Element Method), identifying a discrete set of values in the proper place (mesh, node of grid), space (during the stationary regimes of the flows motion) instead of the continuous solution. In order to achieve the maximum accuracy of solutions, that method to demonstrate discrete values is chosen, which due to the discretization complies with algebraic equations analogues. As a result, the mathematical problem to solve the differential or integral equations system can be reduced to the algebraic equations system.

There is also a method to describe the axisymmetric motion of the swirling gas flow, based on a system of Navier-Stokes equations (1) and the continuity equation (2) (Artyukhov and Sklabinskyi, 2015):

$$\frac{\partial}{\partial t}\left(\rho_g V_i\right)+\frac{\partial}{\partial q_j}\left(\rho_g V_i V_j\right)=-\frac{\partial p}{\partial q_i}+\frac{\partial}{\partial q_j}\left[\varepsilon\left(\frac{\partial V_i}{\partial q_j}+\frac{\partial V_j}{\partial q_i}\right)\right]+f_i, \tag{1}$$

$$\frac{\partial \rho_g}{\partial t}+\frac{\partial}{\partial q_j}\left(\rho V_j\right)=0, \tag{2}$$

where ε is a coefficient of turbulent viscosity; t – time; V_i – instantaneous component of the velocity on the given axis; ρ_g – gas density; p – pressure; f_i – component, characterizing the action of the bulk forces; q_j – axes of coordinates.

Analysis of the previous works in the two-phase flows modeling sector, which consists of gas as a disperse phase and disperse particles, showed that one of the most perspective methods to calculate the motion of the particles is a trajectory method.

Authors concluded that in case of the compressed motion modeling of particles with large diameter (0.5-5 mm), the Lagrangian model regarding the force analysis of the particle motion by the differential equations of motion forms the base for it. The trajectory method with highly accurate results can be used only if there is any software which enables to export theoretical model of the single-particle motion and to consider the flow constraint degree.

The motion of particles in the workspace was simulated on the basis of the differential equations system of the particle motion:

$$\left.\begin{aligned} m\frac{d^2q_1}{dt^2} &= -g + \psi\cdot\frac{\pi\cdot\mu_g\cdot d}{8\cdot m}\left(V_1 - W_1\right), \\ m\frac{d^2q_2}{dt^2} &= \frac{W_3^2}{r} + \psi\cdot\frac{\pi\cdot\mu_g\cdot d}{8\cdot m}\left(V_2 - W_2\right), \\ m\frac{d^2q_3}{dt^2} &= -\frac{W_2W_3}{r} + \psi\cdot\frac{\pi\cdot\mu_g\cdot d}{8\cdot m}\left(V_3 - W_3\right) \end{aligned}\right\}, \tag{3}$$

where m – particle's mass; r – a current radius of the workspace in a unit; w_1, w_2, w_3 – radial, circumferential and flow rate components of the particle's velocity correspondingly; g – gravitational acceleration; ψ – a linear coefficient of particle resistance to the gas flow; μ_g – gas flow viscosity; d – a particle's diameter.

Criteria to choose the two-phase system flow model are the following.

1. Ratio β of the disperse phase substance mass (d) to the mass of the carrier phase substance (c):

$$\beta = \gamma\frac{F_d}{F_c}, \tag{4}$$

where F_d and F_c –volume fractions, γ – the ratio of the disperse phase density to the carrier phase density, $\gamma = \rho_d / \rho_c$.

If β has high values, the dispersed particles have a great impact on the carrier phase flow, so it is necessary to use only the multi-phase Eulerian model for the proper flow modeling.

2. Stokes number St:

$$St = \frac{t_d}{t_c}, \tag{5}$$

where t_d – time, which describes the particles motion, $t_d = (\rho_d\ d_d^2) / (18\ \mu_c)$, d_d – diameter of the particle, μ_c – viscosity of the carrier phase substance, $t_c = L_c / U_c$ – time, which describes the carrier phase,
L_c – characteristic length, U_c – characteristic velocity.

The authors suggest taking into account the constrained mode of motion when we calculate the particle residence time in the workspace of the device

$$\tau_{cm} = \tau f_{e\tau}(\psi), \tag{6}$$

where τ_{cm} - velocity of the constrained motion of particles in the disperse phase; τ - a velocity of a single particle motion; $f_{e\tau}(\psi)$ - an empirical influence function of the constraint conditions on the residence time of a particle in the workspace.
The function $f_{e\tau}(\psi)$ is:

$$f_{e\tau}(\psi) = (1-\psi)^{-m}, \tag{7}$$

where m – an empirical measure of the stage (a constraint coefficient for the time calculations).

Ansys CFX and Ansys Fluent (https://www.ansys.com) software is based on a finite-volume method of solving hydrodynamic equations and using a rectangular adaptive mesh with local grinding. In order to approximate curvilinear geometry with higher accuracy, the technology of geometry grid density is used. This technology enables to import geometry from CAD systems and to share information with finite element analysis systems. This technology has solved the problem of automatic mesh generation – in order to generate a mesh, it is enough to set only a few parameters, after which the mesh is automatically generated for a computational area that has the geometry of any complexity degree.

The calculation starts with some initial approximation set as the initial data. At each global iteration, the velocity and pressure fields, obtained after the time specified as the integration step, are calculated. The stationary solution of the problem is achieved after a sufficiently large number of global iterations corresponding to a long period of time.

When performing the calculation, the program automatically breaks the calculation area into separate subareas, subdomains, the size of which is proportional to the velocity of the appropriate processor. The calculation algorithm works in such a way that each processor calculates the flow only in its own subdomain, and after each global iteration, the resulting solution is “adhesion”. The parallelization algorithms used in modern software products let to achieve acceleration with an efficiency close to 100%. In other words, the calculation time is inversely proportional to the total computing power

As a rule, the adhesion condition is set as boundary conditions on all solid walls (the velocity is zero), the distribution of all velocity components in the inlet section, and the first derivatives (in the direction of flow) of the velocity components in the outlet section are equal to zero. In practice, if the flow rate at the inlet is approximately uniform in the intersection, the user sets only the average velocity (or flow rate) at the inlet, and sets nothing at the output - it is assumed that the user chose the output section far enough from the areas of intensive restructuring of the flow.

In the upper section of the device the inclined shelves (angle of lean 30°) with ratio l/B (where l is the distance from the end of the shelf to the wall of the device; B – width of the device) in the amount of l/B=1 (free channel) to l/B=0.3 were installed. The results of the previous investigations, show that the efficiency to extract the small fraction (with the size less than 1 mm) into the ablation has the maximum value with the ratio l/B=0.5.

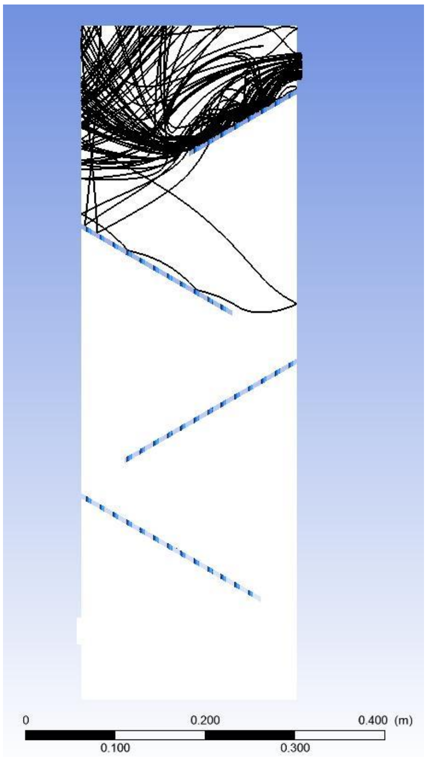
Thus, at the first stage of theoretical studies, it was necessary to find the optimal value of the free area in the shelf for the ablation efficiency of the fraction less than 1mm.

The results of the computer modeling show that the efficiency to extract the small fraction into the ablation is achieved with the reduction of the perforation degree of the shelf. While installing the solid shelf in the separation zone of the device, the whole gas flow guaranteed passes through the outloading space. It defines the extraction of the small fraction of the material precisely in this zone. 5 % of perforation in the shelf causes the redistribution of the gas flow, directing its share through the hole in the shelf. Therefore, the gas flow intensively is interconnected with the material and not only in the outloading space zone but also on the shelf surface. It causes the increase of the fraction ablation efficiency, which is less than 1 mm.

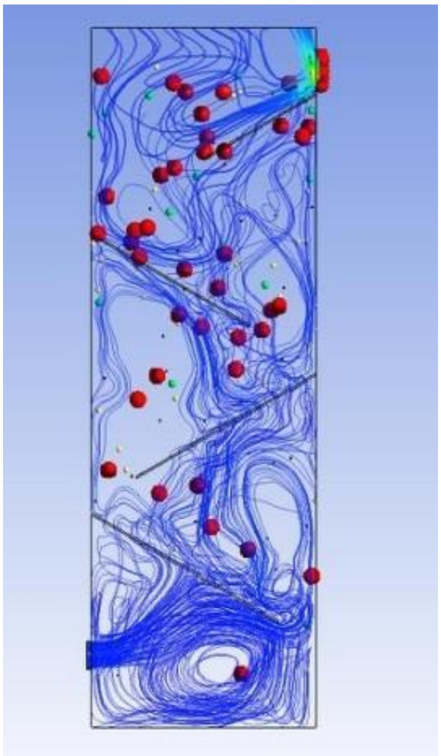
The further increase of the free area of the inclined contact shelf is followed by the reduction of the small fraction extraction efficiency into the ablation. It is explained by the increased effect of the gas flow redistribution. The gas flow velocity in the outloading space decreases so much that the small fraction extraction process of the material in this zone is not very intensive. The total area of holes does not provide sufficient phase contact.

There are three perforated shelves in the drying (granulation) zone of the device. Moreover, while changing the distance between the end of the shelf and the wall of the device, it is possible to create different velocities of the gas flow

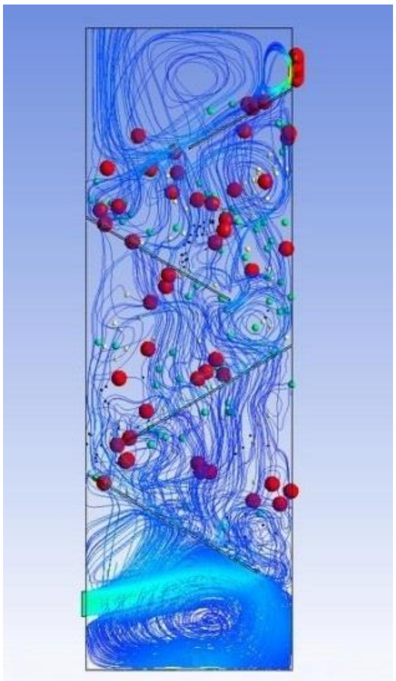
Regime I. Transition regime (figs 1a,b). The upward gas flow force in this regime causes the gradual change of its motion trajectory from the translational to the pulse-forward ones in direction of the outloading gap. Therefore the dispersed material starts to move into the weighted state, and the inertial force is compensated by the upward flow force of the drying agent.



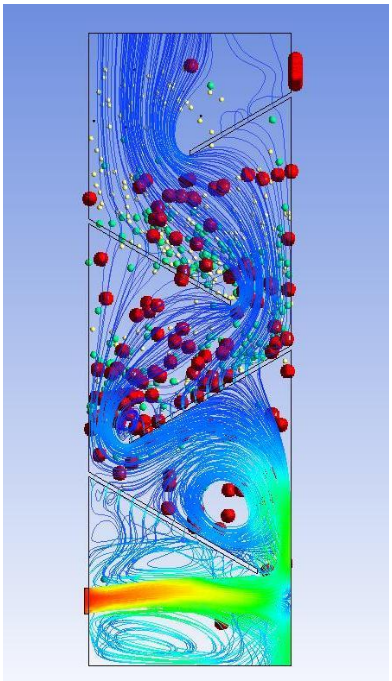
a



b



C



d

Fig. 1. Typical operation regimes of the shelf multistage device: a – separation of the small fraction in the shelf dryer; b – transition operation regime of the dryer; c – weighted layer regime of the dispersed material motion; d – developed weighted layer regime of the dispersed material motion

Regime II. The weighted layer regime (fig. 1c). The effect, made by the upward flow of the heat transfer agent in such regime leads to the creation of the stable weighted layer thanks to the compensation of the inertial force and the rolling-down force on the sloping surface. The gas flow velocity reaches the first critical value, and then it increases and is within the range of the working velocity.Regime III. The regime of the developed weighted layer (fig. 1d). This regime is described by a dominant effect of the upward gas flow force on the dispersed material and by the increase of its displacement vertical component. Therefore, at first, the share of the dispersed material is taken out from the surface of the shelf and then it moves to the outloading gap.The above results for the modeling of the operating regimes in devices let to select the following optimal parameters to implement the target process:

- configuration of the workspace in the device and the way to create the directed motion of the dispersed phase flow;
- duration of the dispersed phase in the workspace of the device;
- technological features of the related processes (separation, classification, heat exchange)
- properties of the raw material and commodity product – temperature and humidity features, fractional composition, hardness index, special properties (porosity, selectivity etc.).
- energy indicators of the process implementation.

In general, the algorithm to calculate the device with the directed fluidized bed can be represented by means of blocks (fig. 2), which are logically interconnected between each other. The peculiarity of the algorithm includes its universality and ability of “flexible” implementation of separate blocks.

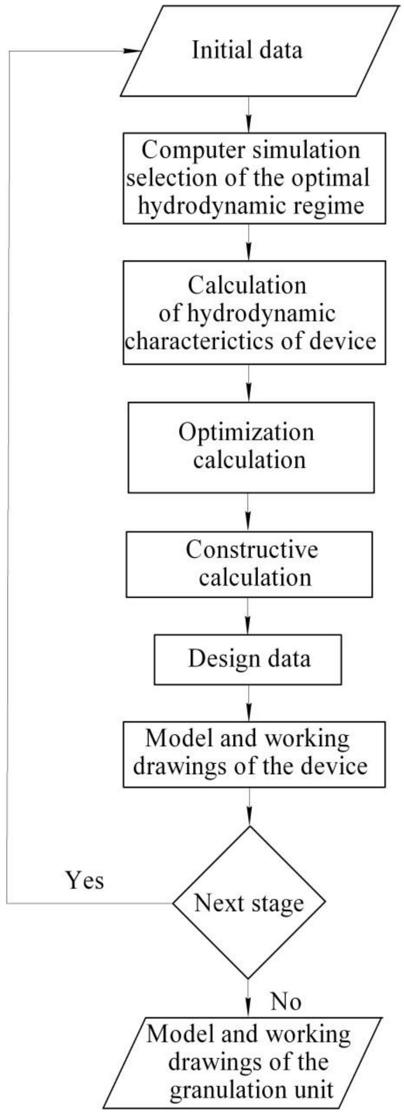


Fig. 2 Block-scheme of the granulation unit calculation

The modeling and calculation results with the use of authors' software, description of which is represented in the work, are exported to the cluster Granulation Unit© in the form of separate structural blocks (fig. 3).

Fig. 3. Cluster Granulation Unit©

Fig. 4 demonstrates the technological scheme of the porous ammonium nitrate production. In this scheme the devices, operating regime modeling of which is shown above, are used as the main technological equipment.

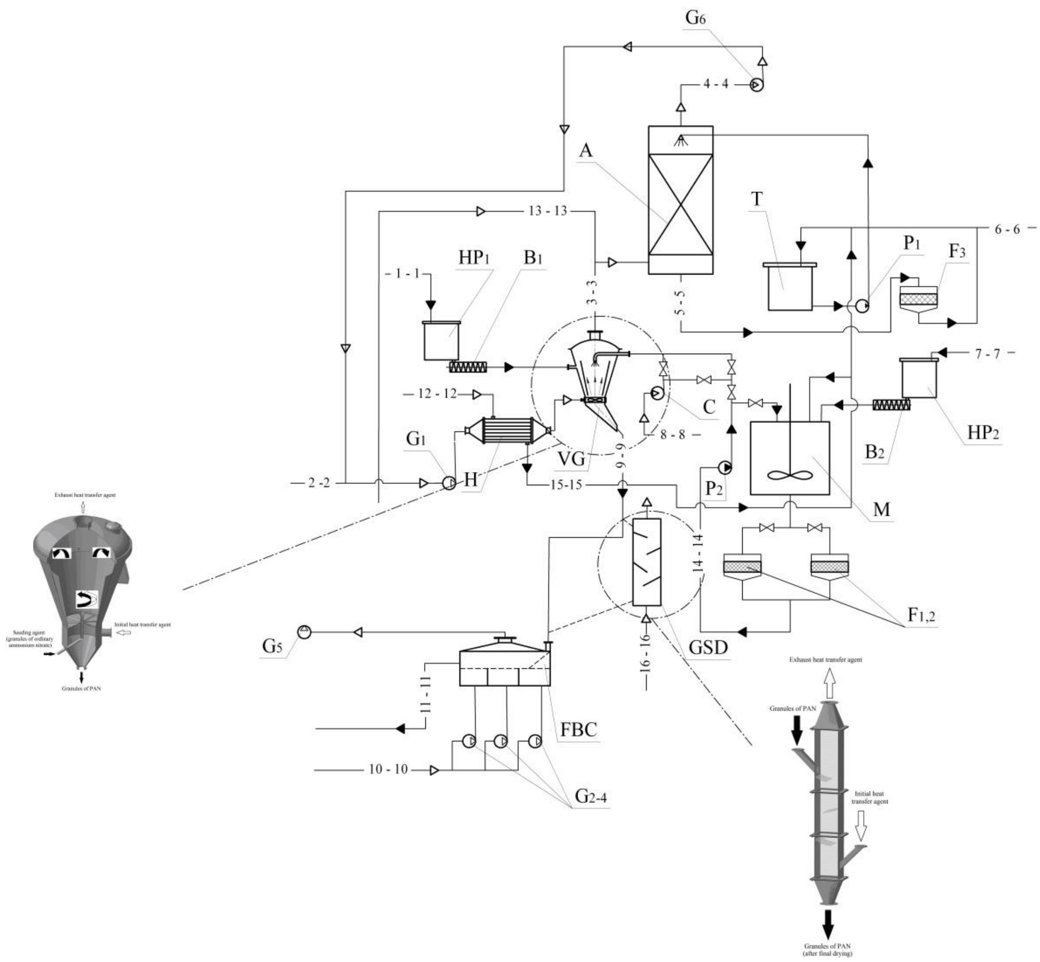


Fig. 5. A unit for the production of PAN granules by using a vortex granulator (multistage shelf granulator) and a gravitational shelf dryer

Elements of the unit:

VG – a vortex granulator; H – a heater; GSD – a gravitational shelf dryer; FBC – a fluidized bed cooler; A – an absorber; F – a filter; M – a mixer; B – a batcher; HP – a hopper; G – a gas blower; P – a pump; T – a tank; C – a compressor.

The main flows:

1-1 – a seeding agent; 2-2 – manufacturing air; 3-3 – polluted air; 4-4 – purified air; 5-5 – polluted water; 6-6 – water; 7-7 – substandard granules; 8-8 – air for the spraying of the liquid materials (solution, melt); 9-9 – the product; 10-10 – air for cooling of granules; 11-11 – granules for packaging; 12-12 – steam; 13-13 – dusty gas; 14-14 – liquid materials (solution, melt); 15-15 – water condensate; 16-16 – a drying agent.

The operation principle of the gravitational shelf dryer is the following.

The drying agent, which at first is evenly distributed over the intersection of the body and then rises upwards, is brought to the lower section of the device. Therefore, it passes some (calculated, in this case – three) perforated inclined inserts (shelves), then it is output from the upper section of the device. Together with the motion of the drying agent, the dispersed material, which is necessary to dry, is fed to the upper shelf of the body. During the counteraction with the drying agent flow, the dispersed material is heated and the small fraction is removed with a purpose to balance the weighted layer porosity on the upper inclined contact shelf. The length of the inclined contact shelf provides the residence time of the dispersed material, which promotes the complete heating of the material, separation of the small dispersed particles and removal of the unbound humidity from the surface layer of the dispersed material during the constant drying velocity. The dispersed material is gradually moving along the upper shelf, is output through its free end and comes to the middle shelf, during drying by the drying agent flow. As the dispersed material moves through the middle shelf, unbound humidity is further removed from the surface layer of the dispersed material during the constant drying velocity as a result of the counteraction with the drying agent flow. The dispersed material is gradually output through the free end of the shelf and reaches the lower shelf. As the dispersed material moved through the lower shelf, the bound humidity is removed from the depth of the material during the drying velocity decrease. The dispersed material is output from the device after the drying process.

This device may be used to dry granular materials, for granulation, capsulation of the mineral fertilizers granules. In order to obtain the final product with a low level of the small dispersed (fine) particles, the process should be carried out with the initial separation, which is implemented on the upper shelf of the device. There are hydrodynamic conditions under which the small fraction of particles (with the size less than 1 mm) is removed from the basic material.

2 INVESTIGATION OF THE CONVECTION DRYING PROCESS IN A MULTISTAGE APPARATUS WITH A DIFFERENTIAL THERMAL REGIME

This section is prepared in according to data [2].

Consider the drying efficiency on the i-stage of the dryer as a ratio of moisture differences of the disperse material before drying to after drying $x_{i-1}-x_i$ and the maximum possible (theoretic) moisture differences of the flows on the stage $x_{i-1}-b_i$,

$$E_i = \frac{\Delta x_m}{\Delta x_{\max}} = \frac{x_{i-1}-x_i}{x_{i-1}-b_i}, \tag{81}$$

where x – moisture of the disperse material; b – moisture of the drying agent

To determine the maximum efficiency of the process on each stage of the gravitation shelf dryer, the test installation ensured the conditions under which the drying was performed until no change in the moisture content of the disperse material has occurred.

To determine the maximum value of Δx_m the shelf in the test installation was fixed horizontally without the uploading gap, and the intake of the disperse material charge was complemented by the determination of its moisture. The determination of the maximum value of Δx_m was completed when the value of Δx_m did not change between the two measurements of the moisture content.

The calculation results of the maximum difference in the moisture contents of the material, corresponding to the maximum efficiency on each stage, are presented in Tab. 1.

Tab. 1 The maximum difference in the moisture contents of the material (polypropylene), corresponding to the maximum efficiency on the stage

A stage number (according to the path of the material)	Maximum difference in the moisture contents Δx , mass fraction
1	0.083
2	0.076
3	0.069

While studying the drying kinetics in the gravitational shelf dryer by computer modeling, we investigated the counterflow motions of the drying agent and the disperse material with the recirculation of the drying agent.

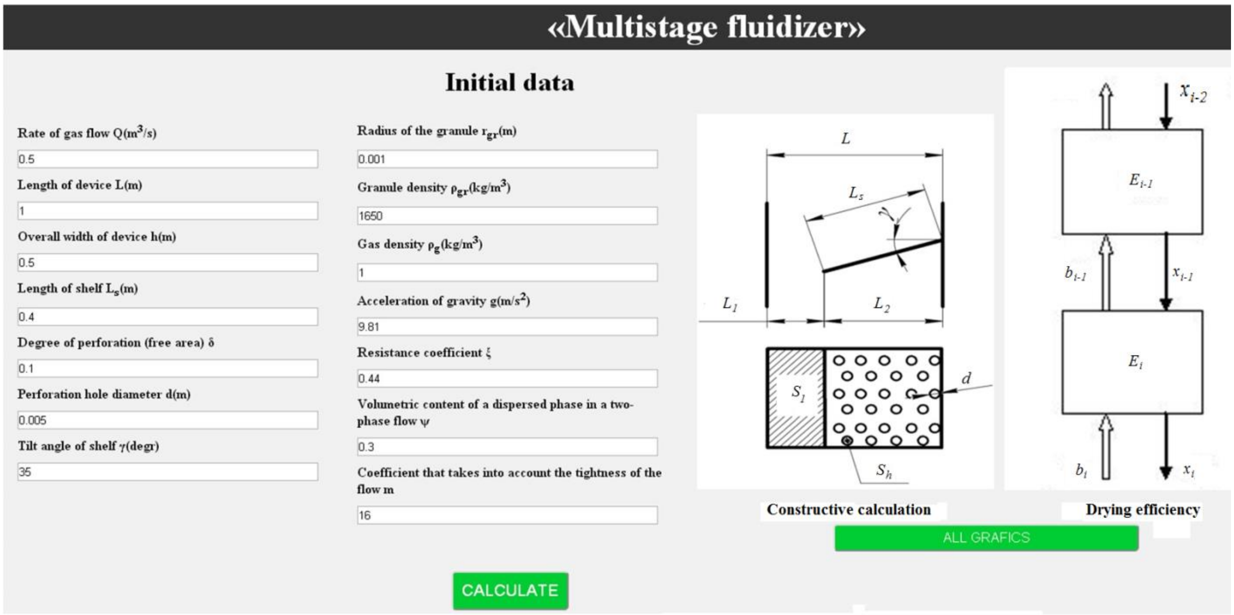


Fig. 6. The main page of the Multistage fluidizer® software

The program Multistage fluidizer® used Hyper markup language HTML, cascading style sheet (CSS) and programing language JavaScript (including the library JQuery). HTML is presented as a tagging of web based app, CSS pages formatting. JavaScript is used to calculate and to transfer data, to create animation and data validation effect. In the validation block of JavaScript data accuracy is checked. In the block input info basic data fields indices are accepted and they are written to the object of input information.

Index.html (fig. 6) is the main page of web based app. It is responsible for reflection of the main menu, for main calculation of gas flow and for jumping the other pages, where main dependences between key features to calculate gas flow and resistance time of the material on the shelf are calculated and dependences diagrams are formed.

The organization of the drying agent motion may have a considerable influence on the quality indicators of the dried material and the properties of the drying agent. That has evolved several studies, the results of which are presented in figs. 7 – 11. Their analysis allows us to select the method of the organization of the drying agent motion, which consumes the least energy, and ensures the necessary complete removal of moisture from the disperse material.

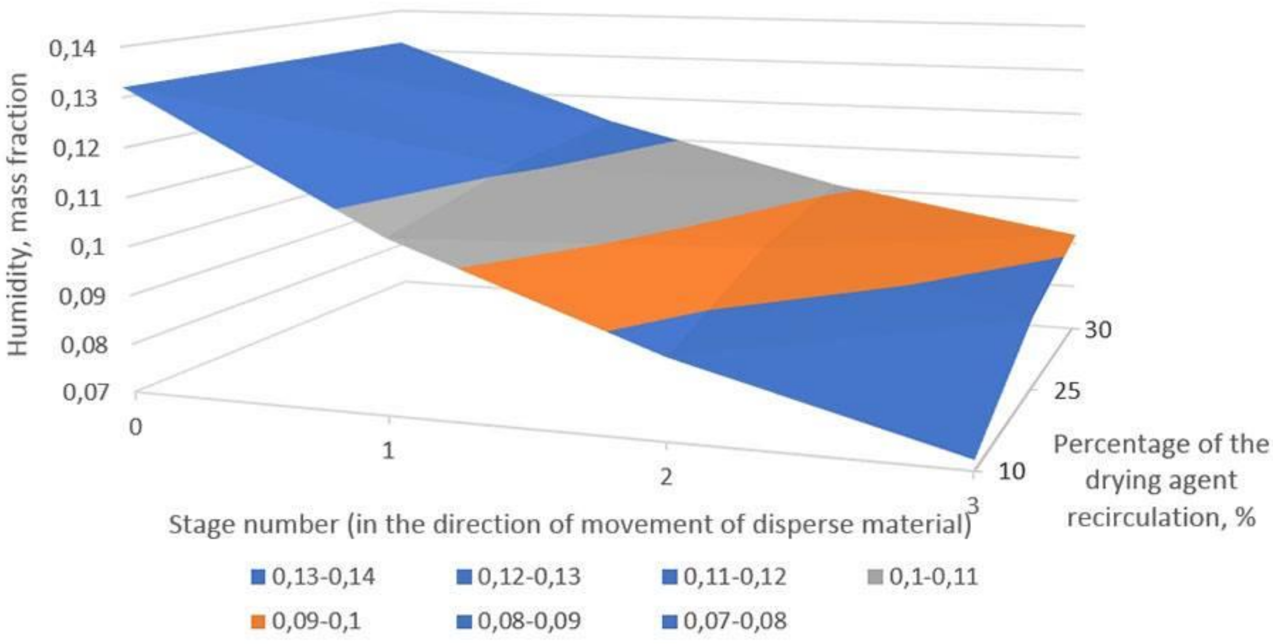


Fig. 7. An influence of the drying agent recirculation method on the change of the moisture content in the disperse material

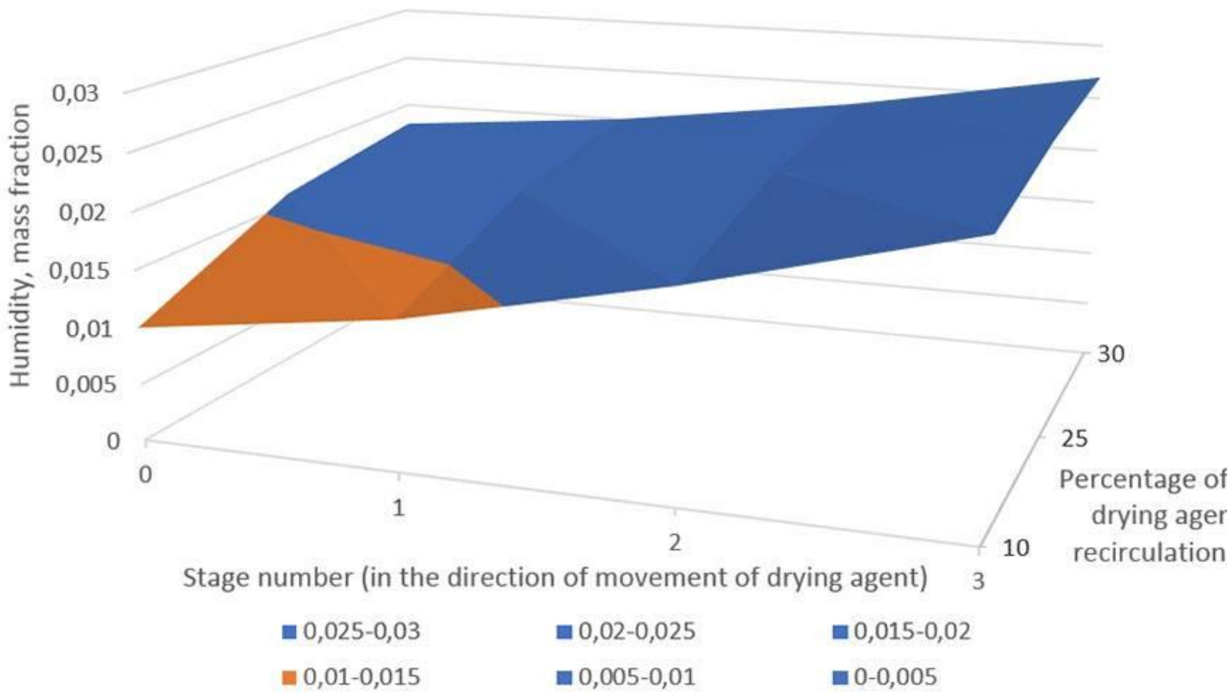


Fig. 8. An influence of the drying agent recirculation method on the change of the moisture content in the drying agent

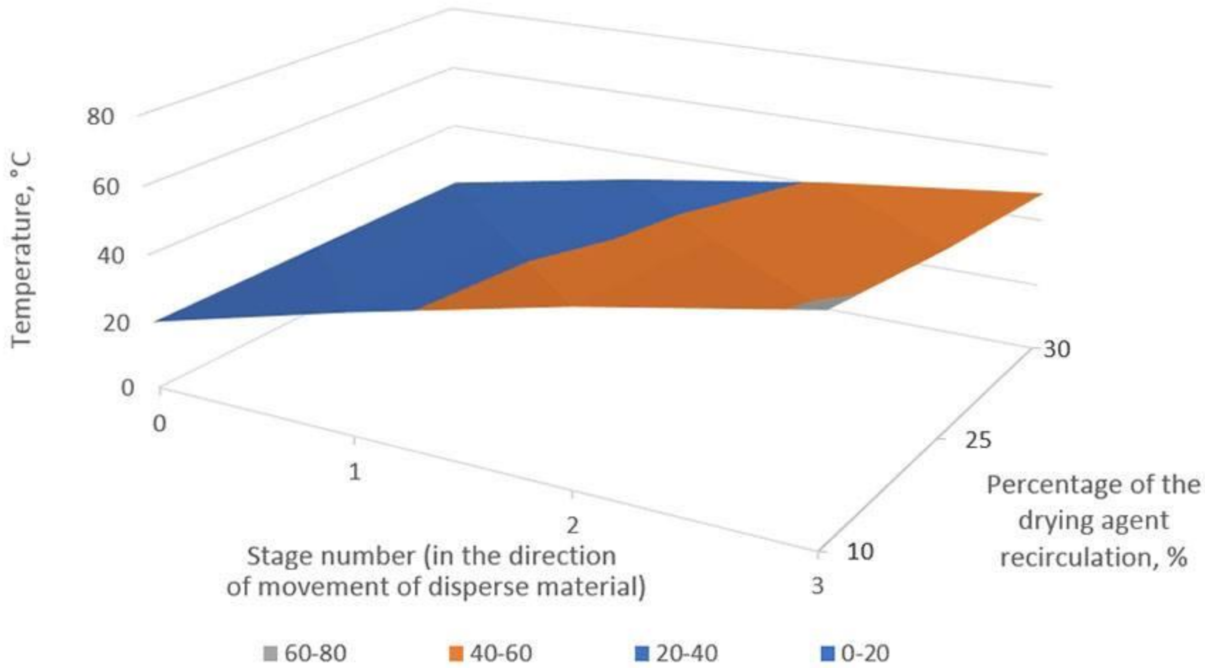


Fig. 9. An influence of the drying agent recirculation method on the temperature change of the disperse material

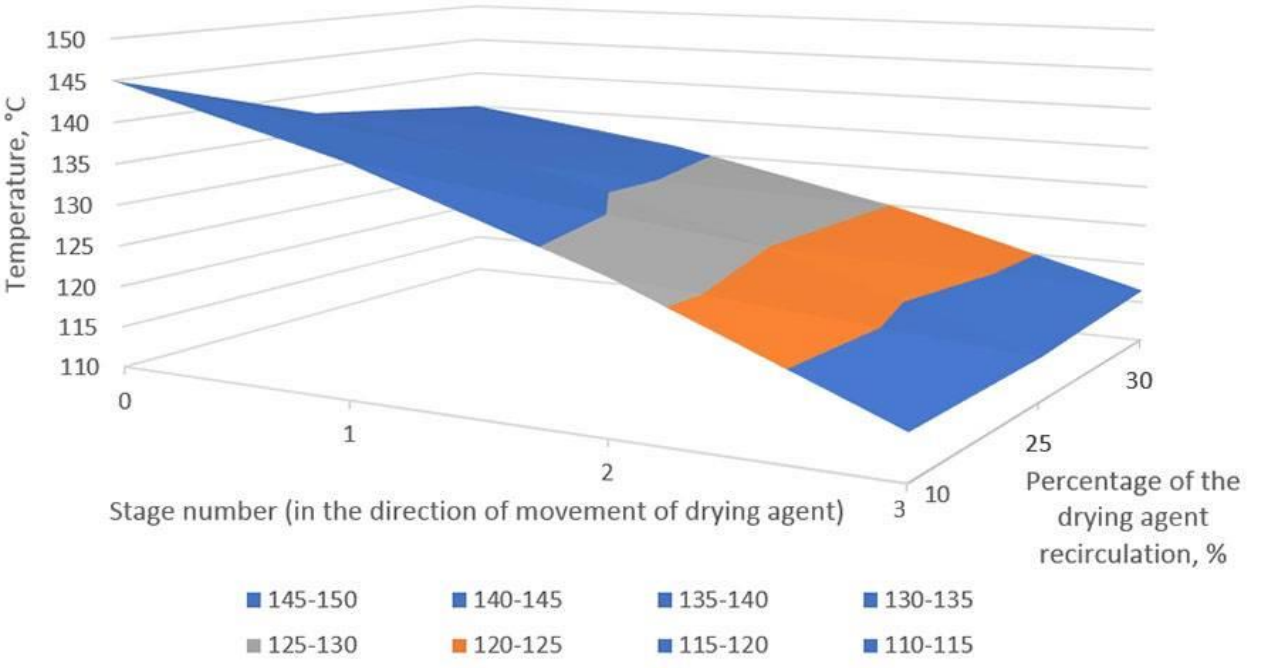


Fig. 10. An influence of the drying agent recirculation method on the temperature change of the drying agent

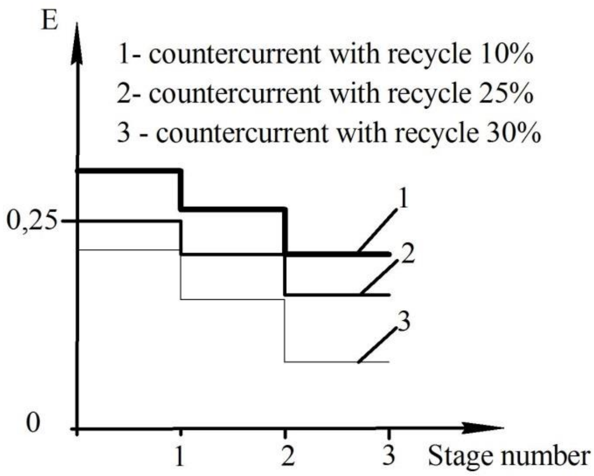


Fig. 11. An influence of the drying agent recirculation method on the drying efficiency (stage number - in the direction of movement of disperse material

3 MORPHOLOGICAL FEATURES OF THE NANOPOROUS STRUCTURE IN THE AMMONIUM NITRATE GRANULES AT THE FINAL DRYING STAGE IN MULTISTAGE DEVICES

This section is prepared in according to data [3].

The most cost-effective way is to use ammonium nitrate granules as a component of industrial explosives. An ordinary ammonium nitrate (mineral fertilizer) and ammonium nitrate with increased porosity (porous ammonium nitrate - PAN)] can be used to prepare a mixture of ammonium nitrate with diesel distillate (ANFO).

The ordinary ammonium nitrate can be prepared to be used as a component of an industrial explosive using the following operations:

- the crushing of granules;
- removal of the shell (powder from various inert substances), which reduces the caking of the granules.

All these operations are aimed at increasing the volume of gaps and cracks after mechanical destruction of the original granules. In this case, despite the high absorptivity, the retentivity does not fit the minimum standard indicator. Therefore, ANFO based on the ordinary ammonium nitrate, should be prepared directly at the blasting places and stored for a short time (due to the intensive draining of diesel distillate).

It is more successfully to use PAN in the technology for obtaining ANFO. Granules with a developed porous structure can reliably hold diesel distillate during transportation to the place of blasting and storing the industrial explosives.

Researchers face the task to investigate new methods for producing PAN with a developed nanoporous structure, which will provide the necessary standard indicators of granules for their successful use as ANFO components.

Table 2. PAN production methods

No	A method to obtain granules	The structure of granules
1	Prilling of “dry” melt and cooling of granules in a monotonic mode	Spherical bodies with a smooth surface and a shrink shell in a dense crystalline array
2	Prilling of “dry” melt, foaming of droplets with poroform and cooling in a monotonic mode	Spherical bodies with a smooth surface and closed gas inclusions in a dense crystalline array
3	Prilling of “wet” melt and cooling with drying in a staged mode	Rounded bodies with a rough surface and cavity in a loose crystalline array
4	The formation of granules by spraying melt on the seeding agent (granulation)	Rounded bodies with a rough surface and homogeneous granular structure in the array
5	The formation of granules with additives of iron salts, followed by surface-active reagents in a drum	Rounded bodies with such formations as flakes on the surface and a granular structure of the body
6	Heat treatment - “porization” of finished granules according to paragraphs 1,2,5 with phase transitions	It fits the original ammonium nitrate, but is saturated with cracks on the surface and inside the granule
7	Humidification followed by heat treatment (drying) with phase transitions	The core of the granule fits the initial ammonium nitrate, the surface and nearsurface layers of the granule form a combination of nanopores of various sizes, depths and configurations

Porous ammonium nitrate can be obtained directly during the formation of granules (product manufacturing) or by physical and thermal effects on the already formed ammonium nitrate granules to change its structure and create the so-called "mechanical" pores. The first group of methods requires various additives to intensify the pore formation process. The necessity to introduce these additives is the reason to increase the cost of the finished product. The second group of methods lets to obtain a developed porous structure without the pore-forming additives. A brief description of the PAN obtaining methods within the presented classification is shown in Table 1.

Method 6 from table 2 was used as a basic variant. However, the research part shows that as a result of heat treatment, only "mechanical" pores are formed in the micrometer range that do not reliably retain diesel distillate during transportation and long-term storage. Therefore, the authors propose to produce PAN industrial explosives directly at the blasting place, which is not always advisable.

An improved scheme for producing PAN (method 7 in table 2) enables to obtain pores in the nanometer range, which positively affects the retentivity of the granules. However, this method provides destruction of the ordinary ammonium nitrate granules due to the high value of the final humidity.

Table 2. Modification of the ammonium nitrate

Modification	Temperature variation range for various modifications, ° C	Type of symmetry	Change in volume,%
I	169,6-125,2	Cubic	-2,13
II	125,2-84,2	Tetragonal	-1,33
III	84,2-32,3	Rhombic monoclinic	+0.8
IV	32,3-(-17)	Rhombic bipyramidal	-3,3
V	(-17)-(-50)	Tetragonal	+1,65

PAN granules with excess final humidity are characterized by the following properties:

- insufficiently developed nanoporous structure;
- many "mechanical" pores that have a straight configuration;
- weak core of the granule (as a result of a significant number of "mechanical" pores);
- many rectilinear macropores in the nanoporous structure of the granule.

It is explained by the high intensity of moisture removal when the drying velocity increases (heating of the material and the first drying period in accordance with accepted terminology).

Here one should consider the regarding the ammonium nitrate modifications (table 3). Solid ammonium nitrate in the temperature range from -17 to 169.6 ° C has five crystalline modifications, each of which has a strictly defined temperature range.

In terms of the production and storage conditions, the polymorphic transformations of ammonium nitrate can adversely affect its physical and mechanical properties. The transition from one modification to another (polymorphic transition) is accompanied by a change in the crystal structure and volume of the crystal grid. The transition processes of one modification of ammonium nitrate to another are reversible. Transitions from one modification to another are reversible and proceed with breakage of the structure, which negatively affects the strength of the granule with an increase in the transitions' number. The preservation of the initial flowability of ammonium nitrate granules under long-term storage conditions significantly depends on the frequency of III↔IV conversion, proceeding at 32.3 ° C with a decrease in the specific volume by 3.7%. The strongest granules will be produced if the modification transformations of ammonium nitrate proceed with minimal volume and structural changes, i.e. without significant deformation of its crystals. This condition will be observed when the sequentially flowing chain of transformations II → III → IV is replaced with the transformation II → IV. The transformation II → IV is stabilized with a decrease in the residual moisture content in PAN.

The balanced temperatures of the modification transformations of ammonium nitrate can be changed by increasing the amount of moisture in the granule, which will affect the polymorphic transitions kinetics. According to [9], so far as the water content increases from 0.5 to 3.4%, the transition temperature IV → III decreases from 48 to 37 ° C, with an increase in water content from 0.03 to 0.5%, the transition temperature I → III decreases from 125 to 120 ° C. This fact justifies the need to carry out not only the heat treatment process of the granules, but also its preliminary humidification. However, moisture does not change the nature of the modification transitions.

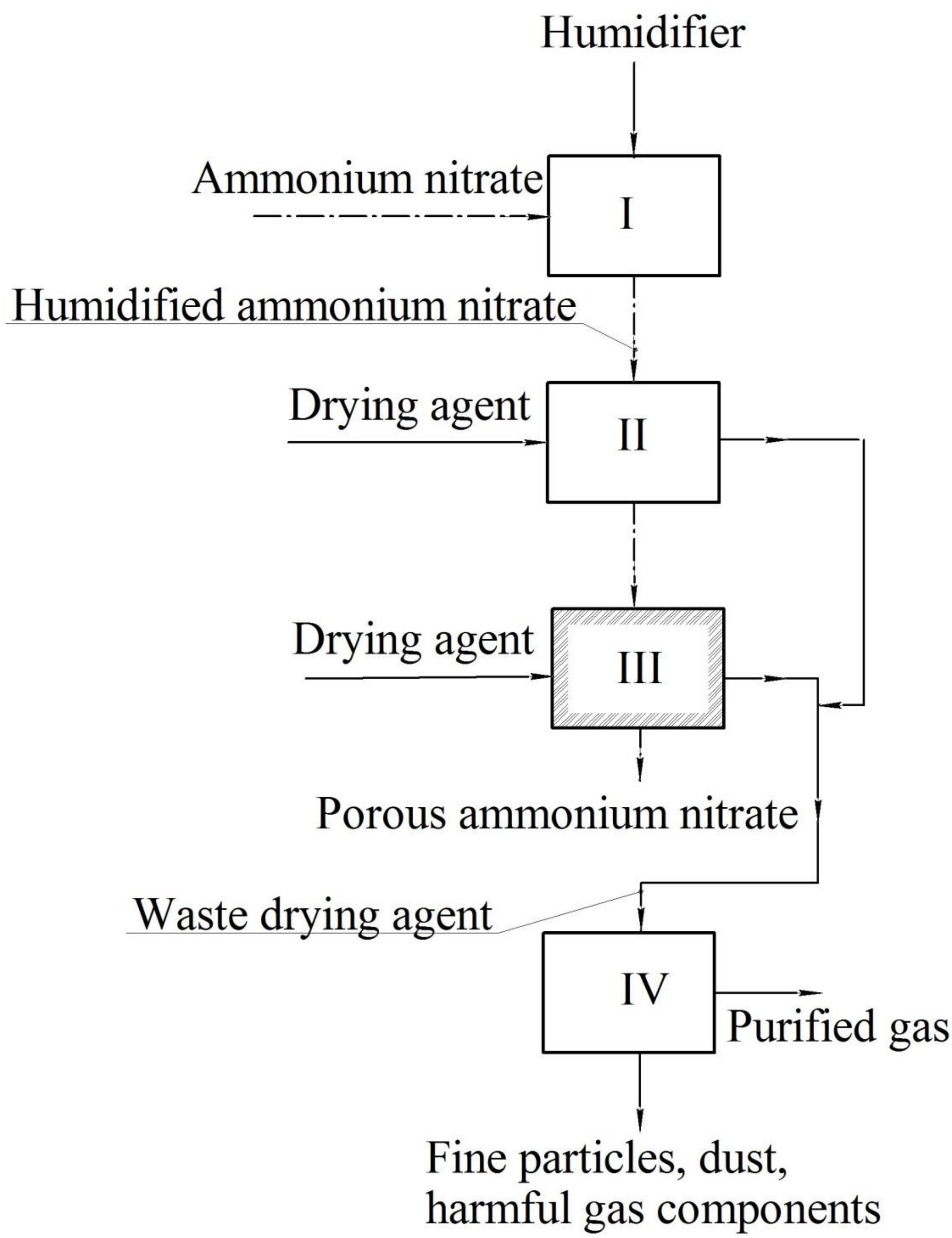


Fig. 12**.** Scheme of PAN production: 1 – humidification of ordinary ammonium nitrate granules; 2 - granulation-drying; 3 - final drying; 4 - purification of exhaust gases.

The final value of the absorptivity can be increased due to more intensive drying under falling velocity. It is possible to carry out this process with prolonged drying in a drying agent's vortex stream only with a decrease in the strength index of the granules.

One should also note that ammonium nitrate is highly hygroscopic, that is one of the reasons for its caking. In the open air, it quickly becomes wet, then spreads out and loses its crystalline form. Therefore, the final drying stage, in addition to the possibility to obtain a developed nanoporous structure, can increase the storage period of PAN granules keeping their normative indicators.

An additional drying stage under the falling velocity mode in the active (but less turbulized) hydrodynamic mode will let us to achieve the necessary changes in the nanoporous structure of the granules.

Thus, the finally improved scheme for PAN production with the final drying stage is in fig. 12.

The experimental unit provides three stages of final drying.

The morphology of the NH_4NO_3 granules surface was studied by scanning the electron microscopy using the device FEI Nova NanoSEM 650.

Other devices and equipment:

- thermocouple TC10-C; recording potentiometer KSP-3 – to measure the temperature in the heater;
- thermal imager Fluke Ti25, pyrometer Victor 305B – to measure the temperature in the granulator’s workspace;
- multimeter DT-838 – to measure humidity of granules and air.

FTIR spectra were obtained by ATR technique using Nicolet iS50 FT-IR spectrometer (fy Thermo Scientific) in range from 4000 to 400 cm^{-1} with resolution of 4 cm^{-1} (no. of scans: 32) (sensitivity for finding of peaks was 95 %).

Four samples are prepared for research:

- the original granules of ordinary ammonium nitrate;
- granules of ammonium nitrate after heat treatment;
- granules of ammonium nitrate after the "humidification-heat treatment" stages in a vortex granulator;
- granules of ammonium nitrate after the “humidification -heat treatment” stages in a vortex granulator and the “final drying” stage in a multistage shelf device.

A thin film of an electrically conductive substance (carbon) was sprayed onto the surface of the samples to carry out the research.

The initial granule structure of ordinary ammonium nitrate is shown in fig.13.

A complete surface without a developed network of pores is peculiar for the sample. The surface structure is characterized by "mechanical" pores in the micrometer range. These pores arose directly upon production of the granules due to temperature stresses and mechanical effect on the packed granules during their transportation from the production place. Such granules can be used as an ANFO component without prior preparation. The absorptivity of the sample is quite high due to the large "mechanical" pores. The retentivity of the sample does not meet the standard indicator. The reason is that the diesel distillate easily leaves the granule (it flows from faults on its surface influenced by the gravity).

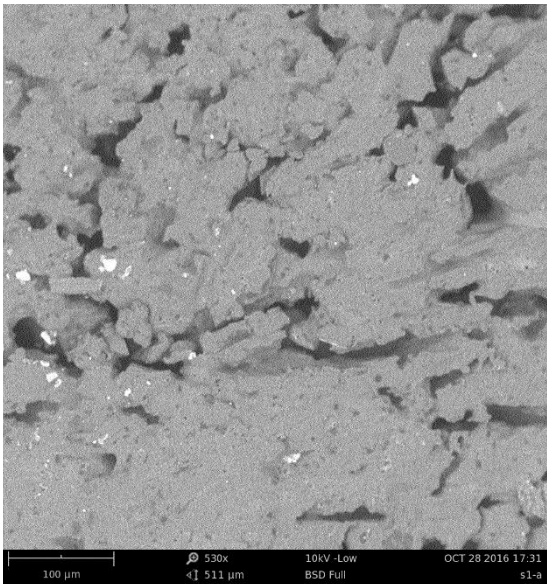


Fig. 13. The initial granule structure of ordinary ammonium nitrate

A sample of ordinary ammonium nitrate granules after heat treatment (“porization”) is shown in fig. 14.

The formation of new "mechanical" pores in the micrometer range, appeared due to temperature stresses in the granule after an additional heat treatment cycle, is peculiar for the sample. Such granules have a high absorptivity. As for the retentivity in relation to the diesel distillate, it is sufficient to obtain an effective ANFO without its subsequent long-term storage and transportation.

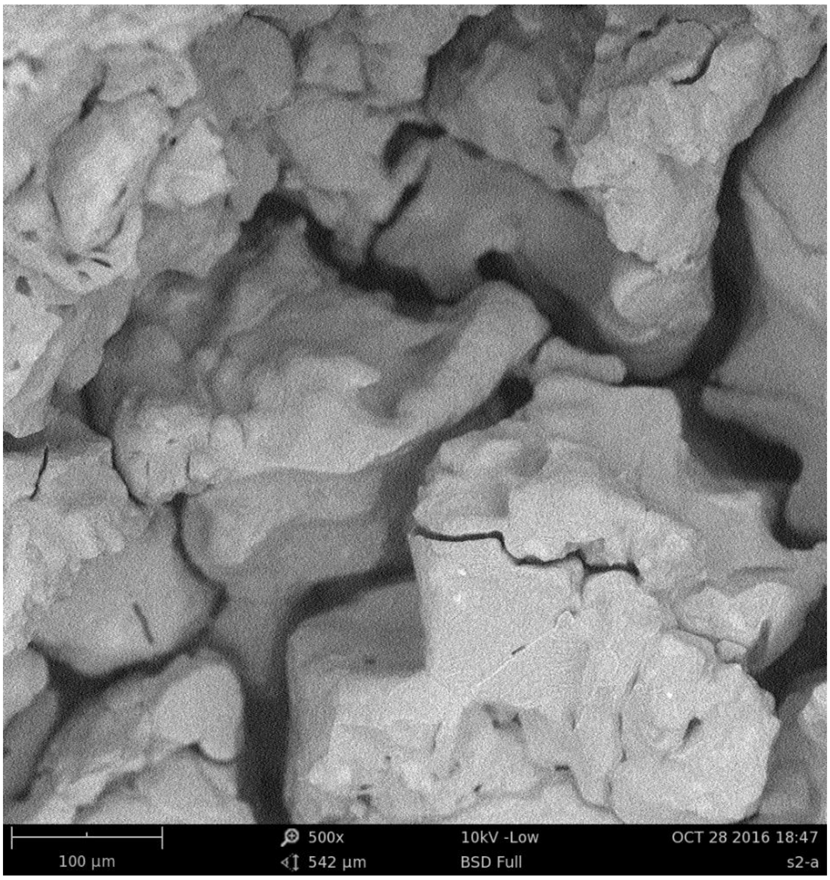


Fig. 14. The structure of the ammonium nitrate granules after heat treatment ("porization")

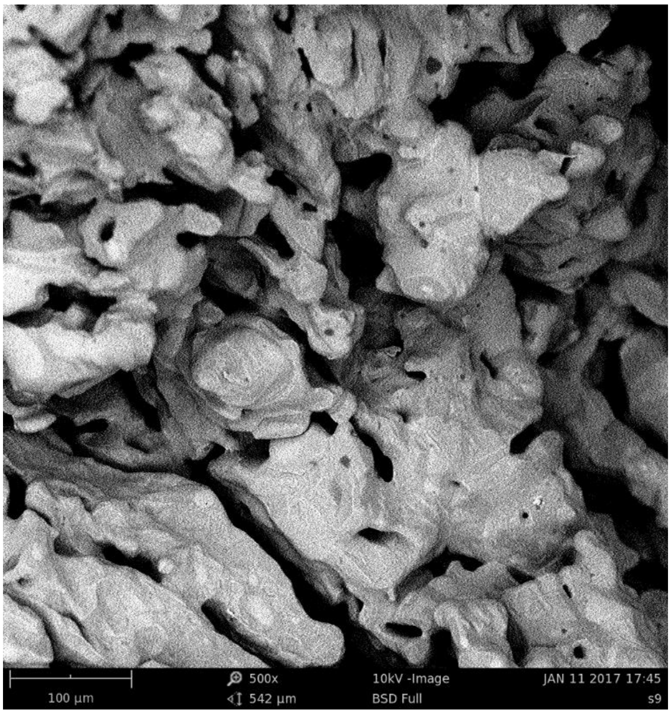


Fig. 15. PAN granule sample, obtained by humidification in a vortex granulator with subsequent heat treatment in the workspace of the same device

The PAN granule sample, obtained by the humidification method in a vortex granulator with subsequent heat treatment in the workspace of the same device, is shown in fig. 15. In addition to “mechanical” pores (their number is not greatly increased in comparison with the initial ordinary ammonium nitrate sample), “modification” pores of nanometer size are also present in the surface structure. The presence of these pores increases the retentivity of the PAN granules and provides the storage and transportation of ANFO to the blasting place. However, the presence of moisture in the granule leads to an undesirable change in the polymorphic transformations chain. A sequentially flowing chain of transformations II → III → IV replaces the transformation II → IV (the advantages of such a chain is described above). The granule becomes more porous and can be destroyed even due to a small external physical impact.

A PAN sample after the final drying stage in a multistage shelf device is shown in fig. 16.

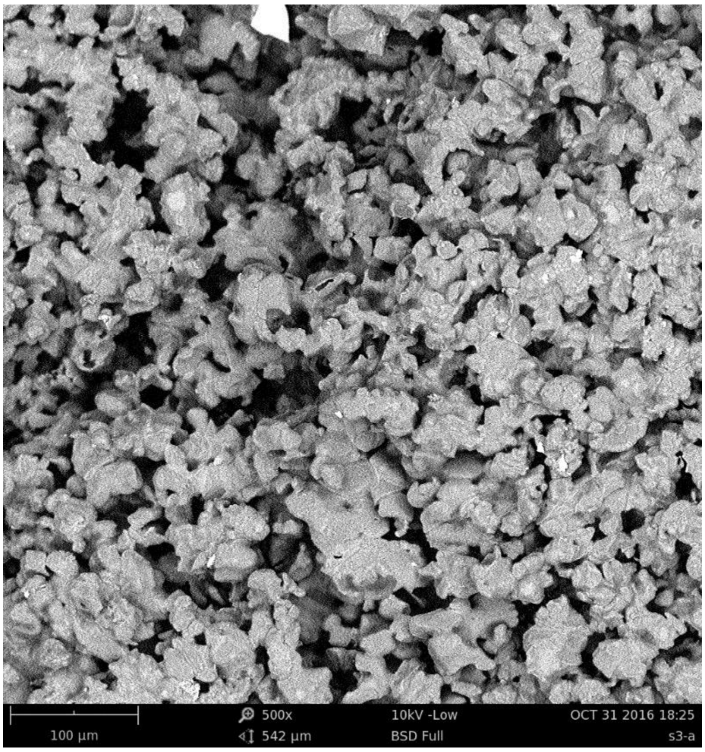


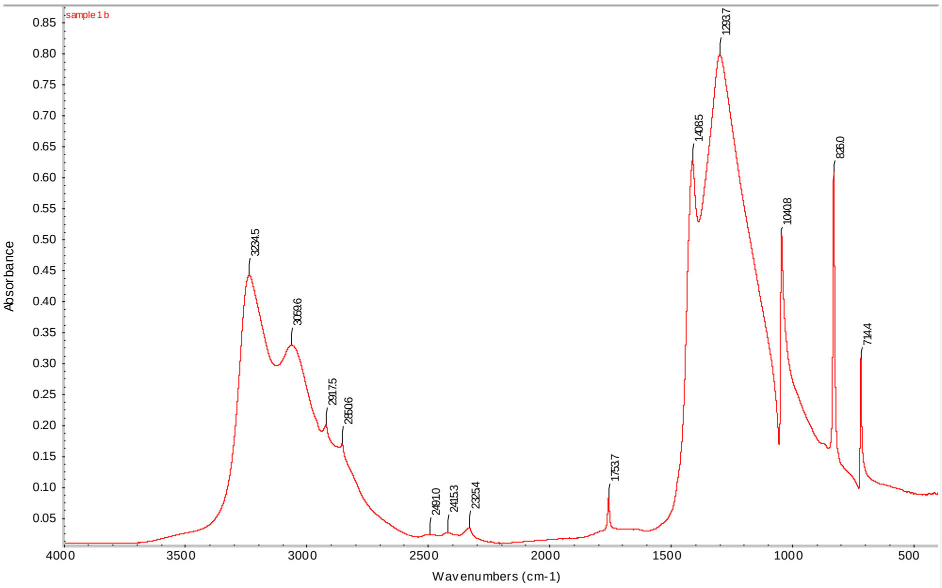
Fig. 16. PAN granules after the “humidification-heat treatment" stages in a vortex granulator and the "final drying" stage in a multistage shelf device.

The sample is characterized by an extensive network of deep curved (tortuous) mesopores of a “modification" nature and their uniform distribution over the entire surface of the granule. In this case, each pore is physically separated from the other and there are practically no united pores in the surface structure. Such a PAN sample (among other inherent advantages in the previous sample) can be successfully stored together with a diesel distillate in the pores due to a slight increase of the granules retentivity.

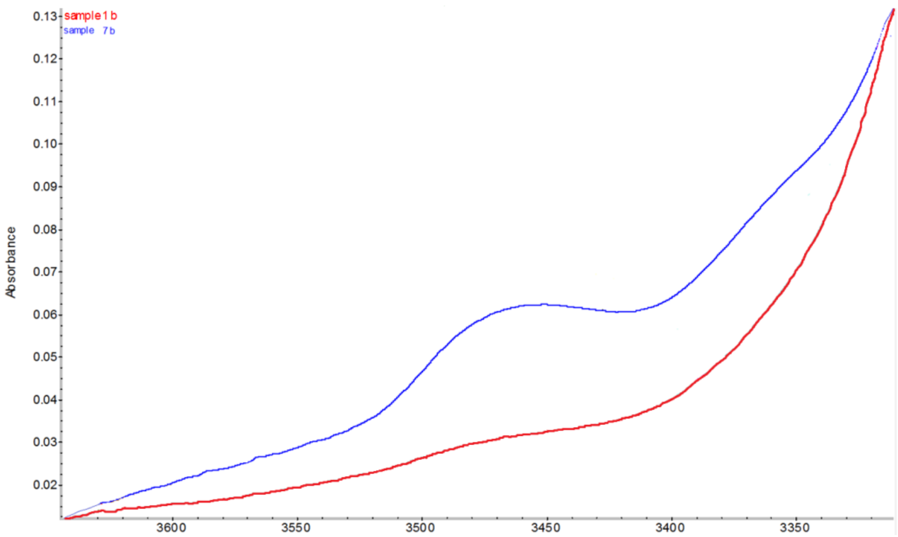
Sectioning of the dryer’s workspace allows to carry out the final drying stage in the "soft" mode. At the same time, at each stage of the dryer’s stage there are conditions, which ensure the minimum temperature stresses in the granule.

One should note that as time goes by, granules influenced by the diesel distillate, begin to loosen and soften. Therefore, it is not recommended to store PAN granules together with diesel distillate for a long time.

During the PAN obtaining process by the method of physical and thermal effects on the ordinary ammonium nitrate granule, it is important to ensure the constancy of the sample phase composition.



a



b

Fig. 17. FTIR spectra of PAN granule after the “humidification-heat treatment” stages in a vortex granulator and “final drying” stage in a multistage shelf device (a), an example of the comparative description of the FTIR spectra part of the initial granule (sample 1b) and PAN granules after the “humidification - heat treatment” stages in a vortex granulator and the “final drying” stage in a multistage shelf device (sample 7b,

area between wavenumbers 3390 and 3540 cm^{-1}) (b)

The results of studies of FTIR spectra regarding the PAN granules for the last sample show that new phases are not formed during the implementation of the described method (fig 17 a). The presence of some splashes of intensity (as, for example, shown in fig 17 b) relates to the presence of moisture in the ammonium nitrate sample and to an increase in the heat treatment time due to the use of a multistage dryer.

CONCLUSIONS

The analysis of the computer simulation results proves that the amount of the drying agent, which is repeatedly used as a recirculating flow, has a considerable influence on the moisture of the dried disperse material. Herewith, the quality of the final product is also predetermined by the initial characteristics of the drying agent (temperature and moisture).

The selection of an optimal technological mode of the dryer with the inner space sectioning is a multifactor task, the solution of which considers the peculiarities of the technological process, a constructive design and energy expenditures on heating and transferring the drying agent in the porous medium.

Based on the analysis of the ways to obtain the granulated product in units with implementation of the directed fluidized bed of the dispersed particles, it was defined that today there are no reliable mechanisms which control the residence time of the dispersed particle in the workspace of the device for such methods. It made impossible to obtain the granulated product with given qualitative indices with minimum energy costs for this process.

Cluster Granulation Unit© enables to carry out the optimization calculation of the main technological equipment in the granulation unit at the stage of its design without the expensive multifactorial experiment. The software peculiarities also let to carry out the equipment changeover if the raw material features and the productivity of the unit are changed.

The results of the optimization calculation with the software products and cluster implementation let to provide:

- a minimum “hydrodynamic” residence time of the particle in the device, which will not exceed “thermodynamic” time (minimum time of particles dehydration to the normative index is determined by the dehydration kinetics laws, thermodynamic indices of the dehydration process);

- implementation of different temperature and humidity potential of the heat transfer agent in some sections in the devices;

- variety of the device construction;
- possibility to use the recirculation of the heat transfer agent.

The original methods of the equipment engineering calculation with the directed fluidized bed as a part to obtain the granulated products are found.

The main recommendations to improve the technological equipment construction of the granulation unit have been implemented in the new methods of granulation, drying and classification in the directed fluidized bed and in devices for their fulfilment, protected by the patents.

Studies regarding the nanoporous structure morphology of PAN granules demonstrate that the final drying stage can improve product quality indicators. Although the PAN production technology using the final drying stage increases the cost of production (compared with the basic scheme for PAS production in a vortex granulator), the cost for storing granules can be reduced. Besides, due to the compactness of the multi-stage dryer, it seems possible to create mobile eco-friendly installations to obtain PAN [11].

Samples of PAN granules after final drying are characterized by a developed network of nanopores with various configurations. Nanopores do not only absorb diesel distillate but also successfully retain it in the granules. Thanks to the improved technology, the granules undergo a polymorphic transformation, which does not affect the granule's strength. Moreover, the granule's low moisture content allows it to be stored for a long time without loss of strength and specific (absorptivity and retentivity) properties. Compared with the “porization” process, the proposed technology has several advantages, including:

- minimal destruction of the granules;
- minimal formation of “mechanical” micropores with an emphasis on the “modification” nanopores formation;
- the shape of the nanopores allows increasing the retentivity of the granules.

The objective of further research is to study the dependence of the final drying time effect on nanopores' structure and the quality indicators of PAN granules.

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