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OBTAINING POROUS AMMONIUM NITRATE IN MULTISTAGE AND MULTIFUNCTIONAL VORTEX GRANULATORS

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ОТРИМАННЯ ПОРИСТОЇ АМІАЧНОЇ СЕЛІТРИ В БАГАТОСТУПЕНЕВИХ І БАГАТОФУНКЦІОНАЛЬНИХ ВИХРОВИХ ГРАНУЛЯТОРАХ

Purpose. Substantiation of opportunities to improve the quality of porous ammonium nitrate (as a component of industrial explosives for the mining industry) through the use of multisection and multifunctional granulators with vortex fluidized bed.

Methodology. The data presented in the work were obtained on the basis of series of experimental studies on experimental-industrial samples of vortex granulators with various designs. According to the results of experiments samples of porous ammonium nitrate were obtained and comparative description of the main indicators of quality including surface structure; strength; fractional composition; retention capacity, is presented.

Findings. It is shown that by the application of multistage and multifunctional devices it is possible to obtain a porous ammonium nitrate with developed surface area and sufficient strength. Here, heat transfer agent recycling is also provided in the volume of device as well as using its different thermal capacity at various stages of granules porous structure producing. Evaluation of fractional composition of commodity granules also allows stating the advantages of presented designs of devices.

Originality. For the first time results, confirming the possibility of combining several stages of process of producing porous ammonium nitrate, were obtained. Using scanning electron microscopy, the main features of the porous structure of the granules were shown and recommendations concerning the control of porous surface formation process were given.

Practical value. The presented results of experimental studies are the base for development of multi-stage and multifunctional engineering calculation technique for vortex granulators. Based on these results improved designs of vortex granulators, protected by IP documents were developed. Such designs of vortex granulators can be used directly at the place of mining minerals of ore and non-metallic origin.

Keywords: *porous ammonium nitrate, mining industry, vortex granulator, multifunctional granulator, multistage granulator, quality*

Introduction. Currently specialists are searching for new ways to obtain granules of porous ammonium nitrate (PAN) as a component of industrial explosives. The PAN samples obtained should have high porosity sufficient, strength retention and absorption abilities [1, 2].

Existing methods for obtaining PAN granules are based on the application of pore-forming and modification additives, which increases the cost of finished product [3]. Introduction of such additives may also lead to lower strength characteristics of PAN granules. The use of repeated humidification and subsequent heat treatment of ammonium nitrate granules to artificially increase the pore volume also affects the strength of granules adversely [4].

For blasting, as the experience has shown, ammonium nitrate of any quality (non-porous, with structural defects, substandard (small or destroyed)) can be used.

At the same time, the use of such ammonium nitrate leads to decrease in detonation velocity of industry explosive based on reducing the amount of diesel fuel, which is retained in the nitrate [5].

Researchers are faced with task of finding new methods for obtaining PAN, which should provide higher granules quality scores and reduce the number of PAN production steps.

Analysis of recent publications. "Energy Strategy of Ukraine till 2030" provides more efficient use of energy resources, fuel and energy economy and based on these measures significantly reduces energy production in chemical industry. Such problems are caused by the energy crisis of the late 20^{th} – early 21^{st} centuries and a sharp increase in the cost of materials. Ukrainian chemical industry is the major consumer of energy resources and is characterized by low utilization of energy. Researchers are faced with the problem of development of new arises of energy saving and especially energy efficient technologies associated with the development of large sets of power density [6, 7] and intensive action [8].

In research studies on kinetics [9] and hydrodynamic [10] conditions on PAN obtaining a new method of producing PAN granules, which is based on single humidification of nonporous ammonium nitrate granules and subsequent heat treatment in the whirling fluidized bed, was proposed. In the method there is a task of obtaining granules with porous structure by the humidification of granule with given quantity of moisture before entering the working volume of the fluidized bed and early exposure to high temperature vortex flow of a coolant. This solution provides simultaneous flow pore formation and drying process, reduces the time for granules in fluidized bed to the minimum necessary and increasing the strength of granules without destroying the internal crystalline structure.

Granule humidification with liquid material in the mentioned way allows you to:

- create highly-developed porous structure on its external surface within the fluidized bed core on the initial contact with high-temperature heat transfer gent vortex flow before granules come on the mirror of the fluidized bed;
- prevent the process of formation of the granules with form other than spherical;
- completely eliminate the factor of influence on the uneven dispersion revenues of liquid material into the dispersant.

The uniformity of liquid material drying on the granule surface and homogeneous surface porous structure that is formed during drying in high temperature vortex flow of heat transfer agent is also achieved by reducing the impact of counter axisymmetrical vortex flow of heat transfer agent and uniform distribution of humidified granules in the fluidized bed.

This method provides high indicators of PAN quality, but is characterized by significant energy consumption for heating and discharge of the coolant. In addition, the hardware design of this method provides presence of taken out camera-humidifier. This affects the dimensions of the installation.

Among other disadvantages of existing equipment for obtaining PAN we can note:

- inability to classify granules according to size and uneven application of liquid phase on the granules with surface of different sizes, which leads to deviations granules form from spherical and the degree of polydispersity granules increasing;
- mixing granules of different factions by reducing the angular velocity of heat transfer agent in the height of device and lack of separation devices in the classification and separation area. At the same time the finished product is obtained with the nonhomogeneous granules size, which affects the quality and reduces the efficiency of device;
- absence of clear separation of device working space into separate zones, in which film deposition on the granule, its crystallization, drying formed granules, their cooling and output are conducted. As a consequence, on some granules the process of forming a solid layer after liquid material film deposition is not completed, in some cases, the film is not fully deposited on the granules, some granules do not enter the spray zone and do not obtain the necessary amount of liquid material on their surface, it also includes sticking of some granules and agglomerates formation;
- inability of parallel deposition of films of different materials on the granules surface and therefore obtaining multilayer granules;
- absence of the possibility of simultaneous withdrawal of several monodisperse granules factions;
- significant temperature differentials when humidifying and drying, which can lead to granule core destruction and its crushing;
- impossibility of processing granules with several solutions (for example, anti-caking after formation of granules porous structure surface) to the extent of one device;
- increasing size of analogues by the fact that the preliminary humidification zone is carried off from the unit into the separate block;
- increasing energy costs and necessity to establish equipment for pneumo transportation because after the preliminary humidification unit it is necessary to supply granules into the device.

For partly addressing these disadvantages several successive working vortex granulators can be used. It also becomes possible to obtain multilayer granules. But at the same time the used working area increases significantly as well as the production process scheme becomes more complicated.

Objectives of the article. The question of development of multistage multifunctional devices of combined action, in which simultaneous multiple processes become possible, is very urgent. In such devices efficiency of working volume should be 2–3 times higher than in existing analogues.

To study the possibility of obtaining PAN in the multistage and multifunctional vortex granulators is the main purpose of the article.

Technique and methodology of research. According to the tasks of experimental research of laboratory of Processes and Equipment of Chemical and Petroleum-Refineries Department of Sumy State University an experimental device was designed whose structure is shown in [9].

Physical modeling is based on methods of similarity theory. In studies the installation scale was changed in experimental models of granulators (compared with industrial conditions). Geometric similarity is maintained by equity of constants and invariants of geometric similarity. Similarity of respective particles movement and their trajectories in industrial design and in experimental models is always maintained as well. The reliability of obtained experiment results is caused by the application of time-tested practice methods.

The basic designs of vortex granulators. Within the article three designs of vortex granulators, the features of which are shown below, are presented.

A two-stage granulator (Fig. 1). Availability of partitioned housing ensures compactness of device placement and possibility of parallel flow of two granulating processes which are identical according to mechanism, but different according to a technological mode, which allows obtaining two fractions of granules. The combination of conical and cylindrical parts in each of the sections allows simultaneous multiple processes: separation of granules in different cross sections of conical part of the section as a result of unequal size and, consequently, weight – granules with a larger size under the gravity and velocity of the angular component of the heat transfer agent are moving to the lower section and periphery, and smaller granules, respectively, move to the upper section and center; establishment of steady movement zone in the cylindrical part for selecting granules with the necessary size.

Due to the presence of an additional vortex gas distribution unit additional twisting of gas flow is provided before entering it into the upper section of the housing, which gives greater value to the tangential gas flow velocity component and results in stabilizing the vortex fluidized bed in the upper section of housing.

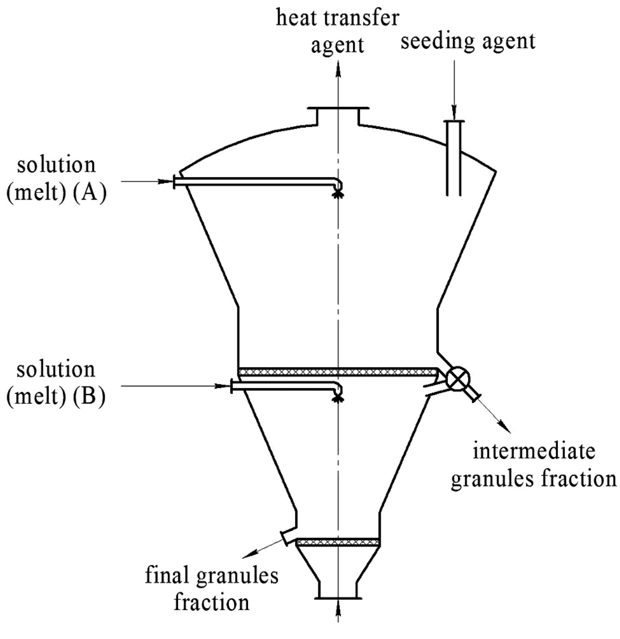

Fig. 1. A two-stage granulator

Availability of a granule overflow unit with all structural elements allows outputting intermediate granules fraction from the upper section of the device housing, if necessary, partially outputting the intermediate granules fraction as a commodity, or providing full flow of intermediate granules fraction from the upper section to the lower section of the device housing. Also we obtain a possibility of device operation without entering a seeding agent into working cavity, i.e. functioning in a closed mode: in the top section together with the application of melt on granules of the seeding agent the formation of crystallization centers from the melt occurs, which further will be a seeding agent, and the second section uses the seeding agent, which is the fraction of intermediate granules from the upper section of the device.

Due to the presence of an additional tube with a sprayer for supplying the liquid fraction, parallel application of liquid film on the granules surface in both cylindrical-conical sections is done. This provides a possibility of obtaining monodispersed granules or obtaining multilayer granules when granules humidification with different fluids in each of the sections of the device.

Application of the proposed vortex granulator will improve the humidification of the liquid material on the surface of granules of fine fraction (seeding agent), that provides a possibility of monodispersed granules or granules of several factions with high monodispersity of each of them or multilayer granules.

Multifunctional spiral granulator (Fig. 2). Performing the top part of the vertical housing in turbinated shape with four turns can divide the inner space of device into separate zones, which will allow separating dif-

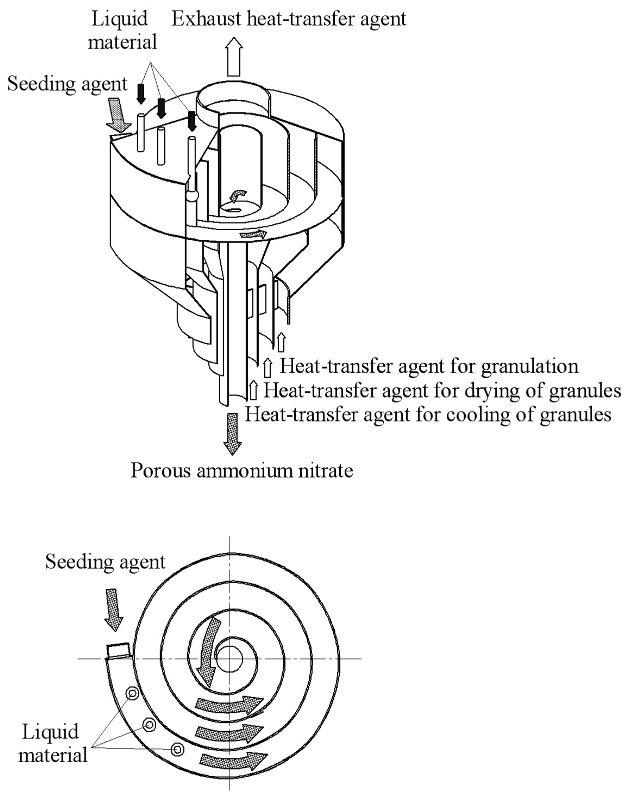

Fig. 2. A multifunctional spiral granulator

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ferent flows and carrying out a phased process of porous structure granules obtaining in volume of a single device. Separation of flows is caused by the fact that at the set time a granule in each zone should receive some thermal capacity necessary for it without incurring thermal stresses. In each following round of turbinated top part of the device a new stage of the porous structure granules obtaining process begins. After the seeding agent humidification (the fourth round of housing with a turbinated shape) it passes consistently through the working volume to the next zones, where it will by dried (the third round of housing with a turbinated shape) in hot heat transfer agent flow, cooled (the second round of housing with a turbinated shape) in the cold air stream and drained from the device (the first round of housing with a turbinated shape).

Performing the bottom part of vertical housing cylinder-conical and composed of external and internal parts allows creating differentiated flow of a heat transfer agent in each zone of the vortex granulator, avoiding the crossing of the heat transfer agent flows and distinguishing clearly the stage of contact of the heat transfer agent with granules in the certain area.

Setting individual nozzles for heat transfer agent supplying to the granulation area (the fourth round of the spiral shape housing), formed granules drying (the third round of the spiral shape housing) and cooling of finished product (the second round of the spiral shape housing) allows controlling the progress due to the fact that each of the first three turns of the spiral shell spiral top of their form created conditions for the formation of granules. This avoids granule overheating phenomena.

Multifunctional sectional granulator (Fig. 3). The innovation is based on the task of device improving by changing the design of the interior space through its division into separate zones. This design solution enables to carry out all stages of granules with porous structure production process to the extent of a single device with high intensity and necessary consistency as well as to improve the quality of the target product.

Operating in the humidification area of the internal additional housing in form of confuser allows for control over moistened granules movement due to the fact that decreasing the free cross section area of the confuser the increasing step of spiral turbinated trajectory of moistened granules is achieved. When installing the liquid material sprayer for granules humidification within the confuser before they reach the drying zone, a developed porous structure on their surface is created, the process of formation of granules with a form other than spherical is prevented, conditions for the uniform application of liquid material on granules surface without the influence of destabilizing factors are created: counter heat transfer agent vortex heat transfer agent flow effect on granule and liquid material film by, a possibility of collision and agglomeration of individual drops, uneven wet material contact time with the drying fluidized agent.

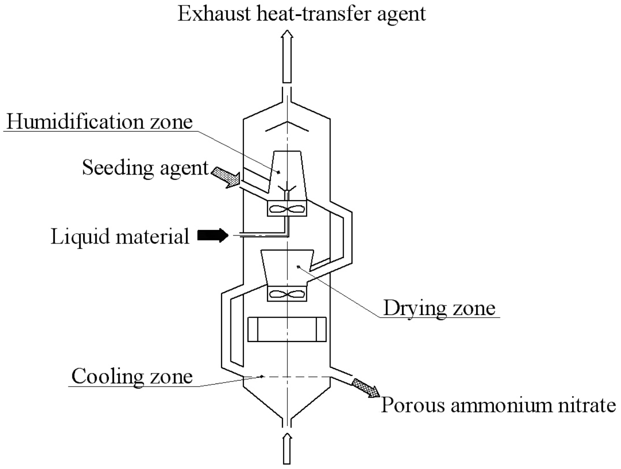

Fig. 3. A multifunctional sectional granulator

Operating in the area of drying internal housing in a diffuser form, promotes uniform liquid material drying on the seeding agent surface, allows obtaining granules with a homogeneous surface porous structure, that is formed during drying when contact with the high temperature vortex flow of the heat transfer agent, creates conditions for even granules drying and porous surface layer creation without the influence of destabilizing factors such as: crossing the trajectories with granules of the created surface layer and moistened granules, which surface layer at creation stage; agglomeration of granules with varying degrees of porous surface layer formation; destruction of the porous surface layer and core granules due to long term contact with vortex flow of coolant at the stage when porous surface layer creation and form granules stabilization are not complete yet. In the diffuser, humidified granules from its bottom are moving to the upper section by spiral trajectory because of reducing weight by removing moisture. Given the variable diffuser cross section, namely its gradual increasing, the velocity rate component of high-temperature heat transfer agent and spiral step turbinated path is decreasing.

Gas distribution grid installation in the bottom part of vertical housing forms a zone of cooling of dried granules, evenly distributes the flow of air through the cross section of the device and supports the layer of granules when device operating mode violation occurs, which prevents materials falling in the under grid space, ensures no dead zones due to mixing material, which is in direct contact with the grid, allows creating highly developed phase contact surface by the developed fluidization regime creation. The surface of granules, which are involved in advanced fluidization mode, is equally accessible for airflow, which significantly contributes to cooling process with high intensity.

The main advantages of the granulators are:

- compactness of the device by simultaneous conducting the main stages of the production of granules with porous structures, such as seeding agent wetting processes, drying of formed granules in one housing;
- ability to manage the time granules spend to the extent of the device;
- classification of granules according to size to the extent of the device, which leads to increasing the degree of commodity faction monodispersity;

- uniformity of the contact high-temperature heat transfer agent with granules in a weighted vortex layer mode;
- absence of granules overheating, and consequently reducing the granules thermal stress and the risk of crack appearing and core destruction through clear separation of heat transfer agent streams input with different enthalpy in separate zones of spiral top part of the device;
- uniform contact of wet granule with the flow of the heat transfer agent in a vortex weighted layer mode, minimally required time to form the porous surface layer on a granule;
- improving quality of finished product and its degree of monodispersity through differentiated granule interaction with the flow of the heat transfer agent at each stage of granulation;
- possibility of obtaining few fractions of granules with high monodispersity of each fraction.

Application of multistage and multifunctional vortex granulators can increase the intensity and uniformity of the porous surface layer on granules formation and create conditions for obtaining a quality product with a high degree of dehydration and developed porous surface without destroying its structure. Using a proposed vortex granulator will increase the efficiency of pore formation on granule surface, uniformity of its growth, and increase the percentage of formation of granules with uniform porous layer of dried liquid material in a given range of size and weight. This will provide increased monodispersity of granulometric structure of material and improve the quality of final product.

Materials of PAN structure and features research. Analysis of ordinary ammonium nitrate surface structure (Fig. 4) at a magnification by 3300 times shows, that the granules surface has only small micropores. The number and size of these micropores are insufficient to ensure the standard ratio of PAN holding capacity.

Fig. 5 shows the microstructure of ammonium nitrate granules surface which was treated with water and then dried in the volume of one-section vortex granulator. As we see from the figure, the pores on granules surface are clearly observed at a magnification by 225 times. Local cracks are also visible, which suggests that during the preparation of PAN significant thermal stresses were present.

Fig. 6 shows the microstructure of ammonium nitrate granules surface, which was treated with water and

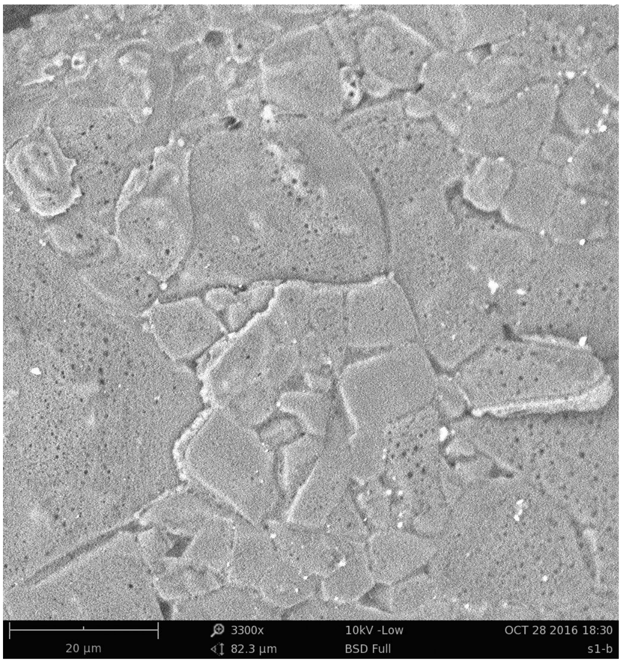

Fig. 4. The surface of ordinary ammonium nitrate prior to treatment

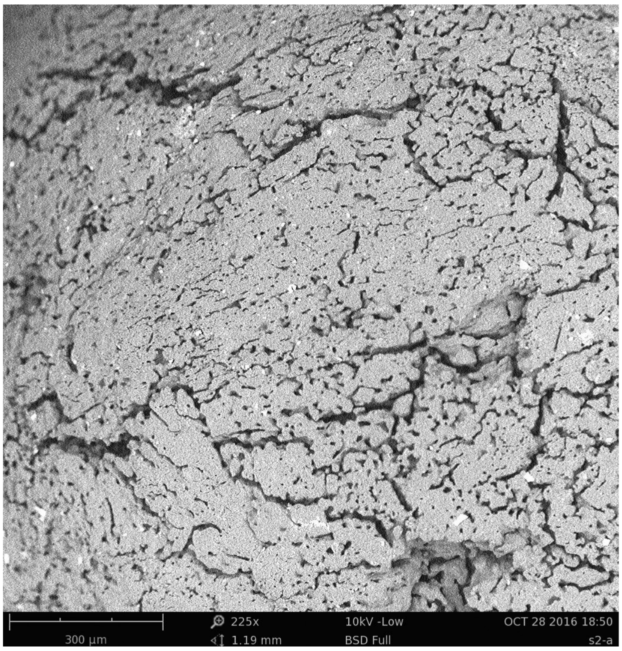

Fig. 5. The surface of PAN, obtained in a single-stage fluidized granulator

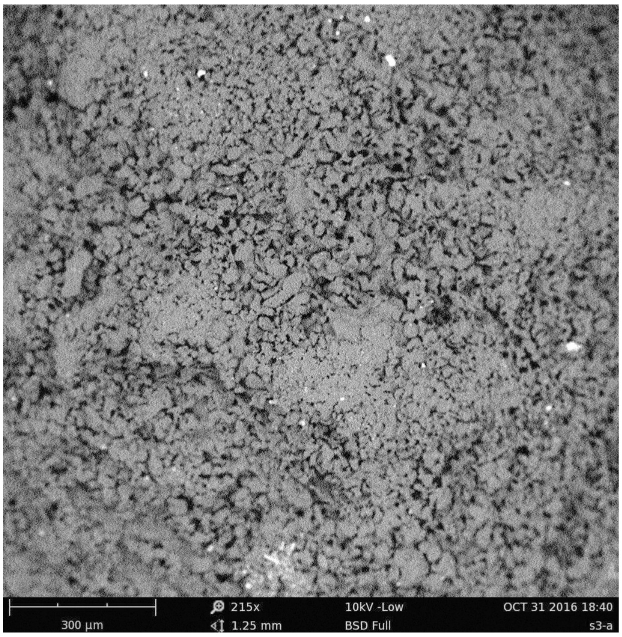

Fig. 6. The surface of PAN, obtained in a multistage vortex granulator with single humidification (granulator-classifier)

pre-dried in the first section of the multisection vortex granulator, and subsequently final drying occurred in the second section. As we can see from the figure, the surface porosity of granules is expressed more clearly than in the granules after processing in the single section vortex granulator. Lack of local cracks indicates softer granules drying mode.

In Fig. 7 microstructure of ammonium nitrate granules surface is presented, which was treated with water and dried in the first section of the multisection vortex granulator and re-processed with water and drying in the second section. As we can see from the figure, the pores on the granules surface became large in diameter and depth (compared to the surface shown in Fig. 6).

In Fig. 8 the microstructure of ammonium nitrate granules surface, which was treated with an aqueous urea solution and dried in the monofunctional vortex granulator, is shown. As we can see from the figure the area covers the ammonium nitrate granule with a non-continuous layer. Some sections of PAN after drying have uniform porous structure.

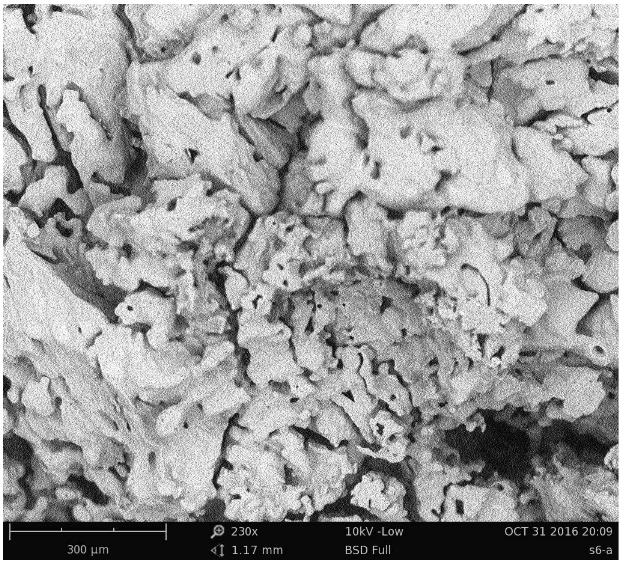

Fig. 7. The surface of PAN, obtained in the multistage vortex granulator with double humidification (granulator-dryer)

In Fig. 9 the microstructure of ammonium nitrate granule surface, which was treated with water, dried and cooled in the multifunctional vortex granulator, is shown. As we can see from the figure, the granule has a developed porous surface. Local cracks in the surface of the granule are absent, which indicates a soft drying mode.

Comparative characteristics of PAN samples, obtained under different conditions (using different types of equipment), are presented in Table.

Conclusions. The results of experimental research studies show that application of multi-stage and multifunctional vortex granulators improves quality indicators of PAN. With virtually unchanged strength (within the standard ratio) the fractional composition and retaining ability of PAN granules is improved. Variation of the hydrodynamic and thermodynamic conditions for PAN obtaining in separate parts of the device (steps or sections) allows to obtain uniform developed porous surface.

As part of this research we have proposed pilot production scheme for PAN obtaining using the multifunctional vortex granulator. Principally, it can be submitted in a form shown in Fig. 10.

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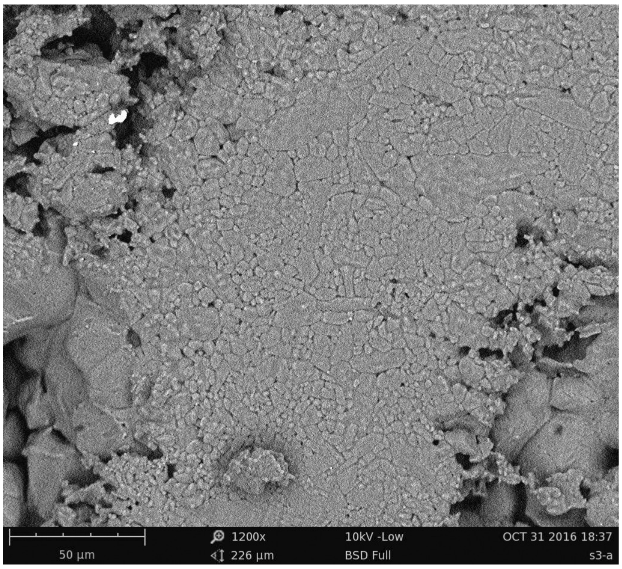

Fig. 8. The surface of PAN, obtained in the monofunctional vortex granulator

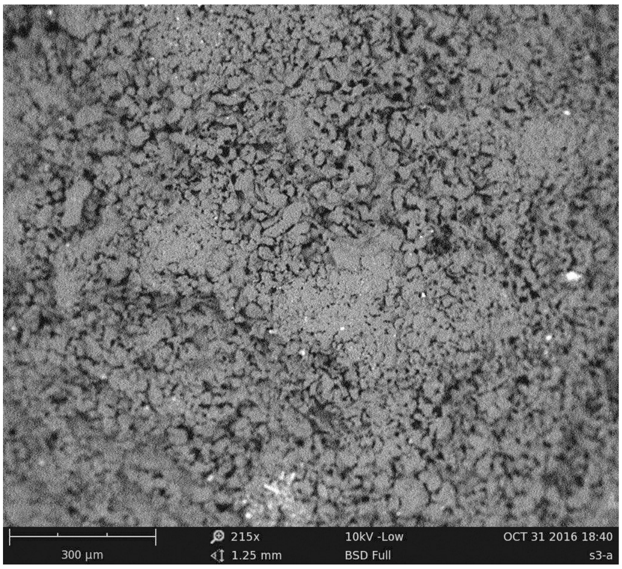

Fig. 9. The surface of PAN, obtained in the multifunctional vortex granulator

Table

Comparative characteristics of PAN samples, obtained in various types of vortex granulators

Equipment for sample obtaining	Characteristics of surface	Strength, kg/granule	Holding capacity, %	Content of commodity fraction 2–3 mm, %
Single-stage granulator	The porous surface with local cracks and single non-porous sections	0.35	8	71
Two-stage granulator (“granulator-classifier” mode)	The porous surface with local cracks	0.38	8.9	81
Two-stage granulator (“granulator-dryer” mode)	Developed porous surface	0.4	9.1	81
Multifunctional spiral granulator	Developed porous surface	0.4	9.5	93
Multifunctional sectional granulator	Developed porous surface	0.4	9.7	94

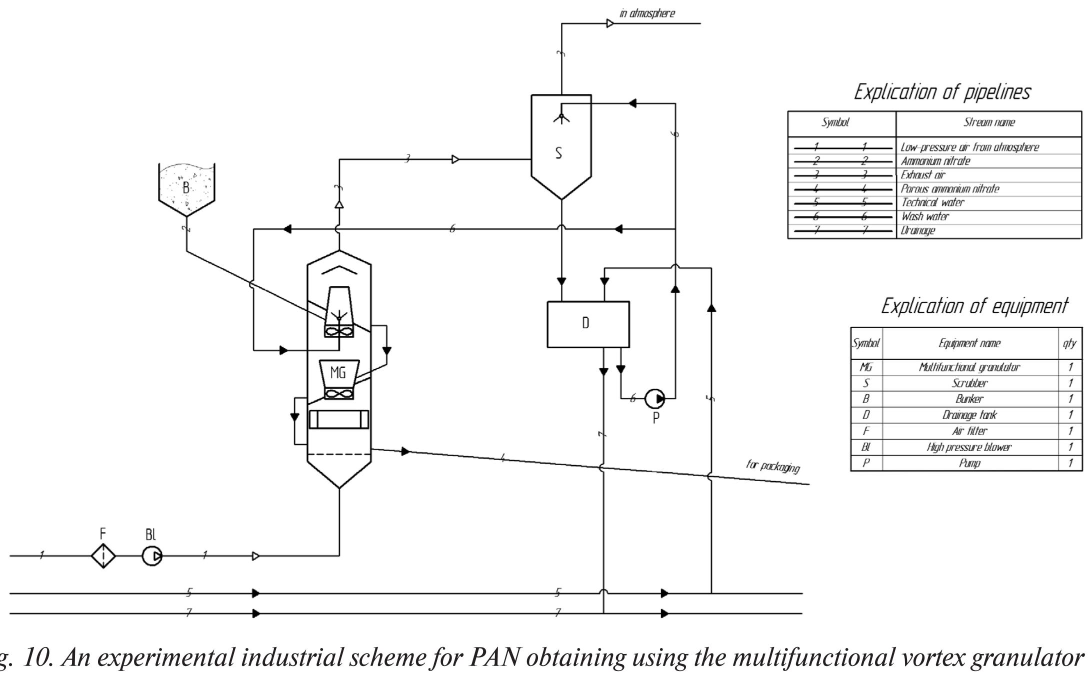

Fig. 10. An experimental industrial scheme for PAN obtaining using the multifunctional vortex granulator

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Мета. Обґрунтування можливості підвищення якості пористої аміачної селітри (як компонента промислових вибухових речовин для гірничодобувної промисловості) за рахунок застосування багатосекційних і багатофункціональних грануляторів із вихровим псевдозрідженим шаром.

Методика. Представлені в роботі дані отримані на підставі серії експериментальних досліджень на дослідно-промислових зразках вихрових грануляторів різної конструкції. За результатами експериментів отримані зразки пористої аміачної селітри та представлена порівняльна характеристика основних показників якості: структура поверхні; міцність; фракційний склад; утримувальна здатність.

Результати. Показано, що при використанні багатоступеневих і багатофункціональних апаратів можливе отримання пористої аміачної селітри з розвиненою поверхнею й достатньою міцністю. При цьому забезпечується вторинне використання теплоносія в об’ємі одного апарата й використання його різного теплового потенціалу на різних стадіях отримання пористої структури гранули. Оцінка фракційного складу товарних гранул дає підстави для твердження про перевагу представлених конструкцій апаратів.

Наукова новизна. Уперше отримані результати, що підтверджують можливість об'єднання декількох стадій процесу отримання пористої аміачної селітри. Із застосуванням растрової електронної мікроскопії показані основні особливості пористої структури гранул і видані рекомендації щодо механізмів управління процесом утворення пористої поверхні.

Практична значимість. Представлені результати експериментальних досліджень покладені в основу розробки методики інженерного розрахунку багатоступеневих і багатофункціональних вихрових грануляторів. На підставі отриманих результатів розроблені вдосконалені конструкції вихрових грануляторів, що захищені охоронними документами. Такі конструкції вихрових грануляторів можуть бути використані безпосередньо в місці видобутку корисних копалин рудного й нерудного походження.

Ключові слова: *пориста аміачна селітра, гірничодобувна промисловість, вихровий гранулятор, багатофункціональний гранулятор, багатоступінчастий гранулятор, якість*

Цель. Обоснование возможности повышения качества пористой аммиачной селитры (как компонента промышленных взрывчатых веществ для горнодобывающей промышленности) за счёт применения многосекционных и многофункциональных грануляторов с вихревым псевдоожиженным слоем.

Методика. Представленные в работе данные получены на основании серии экспериментальных исследований на опытно-промышленных образцах вихревых грануляторов различной конструкции. По результатам экспериментов получены образцы пористой аммиачной селитры и представлена сравнительная характеристика основных показателей качества: структура поверхности; прочность; фракционный состав; удерживающая способность.

Результаты. Показано, что при использовании многоступенчатых и многофункциональных аппаратов возможно получение пористой аммиачной селитры с развитой поверхностью и достаточной прочностью. При этом обеспечивается вторичное использование теплоносителя в объёме одного аппарата и использование его различного теплового потенциала на разных стадиях получения пористой структуры гранулы. Оценка фракционного состава товарных гранул также позволяет утверждать о преимуществе представленных конструкций аппаратов.

Научная новизна. Впервые получены результаты, подтверждающие возможность объединения нескольких стадий процесса получения пористой аммиачной селитры. С применением растровой электронной микроскопии показаны основные особенности пористой структуры гранул и выданы рекомендации относительно механизмов управления процессом образования пористой поверхности.

Практическая значимость. Представленные результаты экспериментальных исследований положены в основу разработки методики инженерного расчёта многоступенчатых и многофункциональных вихревых грануляторов. На основании полученных результатов разработаны усовершенствованные конструкции вихревых грануляторов, защищённые охранными документами. Такие конструкции вихревых грануляторов могут быть использованы непосредственно в месте добычи полезных ископаемых рудного и нерудного происхождения.

Ключевые слова: *пористая аммиачная селитра, горнодобывающая промышленность, вихревой гранулятор, многофункциональный гранулятор, многоступенчатый гранулятор, качество*

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