

## Evaluation of the Impact Made by the Hydrodynamic Regime of the Granulation Equipment Operation on the Nanoporous Structure of $N_4HNO_3$ Granules

A.E. Artyukhov<sup>1</sup>, J. Krmela<sup>2</sup>, O.M. Gavrylenko<sup>3</sup>

<sup>1</sup> Sumy State University, 2, Rimsky-Korsakov St., 40007 Sumy, Ukraine

<sup>2</sup> Alexander Dubcek University of Trencin, 491/30, I. Krasku St., 02001 Puchov, Slovak Republic

<sup>3</sup> "E.M.A." Ltd, 111, Kharkivska St., 40007 Sumy, Ukraine

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The article deals with the impact of the hydrodynamic feature of the vortex granulator operation and intensity of the heat transfer agent's directional vortex motion on the structure and quality of the porous surface layer and internal nanoporous structure in the ammonium nitrate granules. The analysis results of the porous ammonium nitrate quality indices, obtained in different hydrodynamic regimes, were demonstrated. The structure of a granule, obtained in the vortex gas flow with different twisting intensity, was studied. The received data make the base to create the engineering calculation method of granulators as a part of the unit to obtain 3D nanostructured porous surface layer on the ammonium nitrate granule.

The obtained porous ammonium nitrate (PAN) samples were tested to define the explosive features of the industrial explosive "Ammonium Nitrate/Fuel Oil" (ANFO). The detonation velocity of ANFO was determined by the Dotrish method (it is based on the comparison of the known detonation velocity of the detonating chord with the unknown velocity of ANFO detonation). The industrial explosive (ANFO) charges based on the 95 % of PAN and 5 % of the diesel fuel distillate reliably detonate from the intermediate charge – trotyl block, which is initiated by the electric detonator. The ANFO detonation velocity was 2.2-2.3 km/s. It is possible to regulate the process regarding the nanostructured porous layer formation with the specified properties owing to the selection of the optimal hydrodynamic regime to lay the humidifier's film and granules heat treatment. The results of the theoretical and experimental research carried out by authors enable to perform the optimization projection of the main equipment in the unit to obtain PAN, i.e. the vortex granulator.

**Keywords:** 3D nanostructured porous layer, Vortex granulator, Hydrodynamics, Hydrodynamic modes.

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### 1. INTRODUCTION

Ammonium nitrate ( $N_4HNO_3$ ) is an obligatory component of the free-flowing industrial explosives. In combination with diesel fuel distillate (international terminology of such explosive is ANFO), it provides satisfactory explosive properties [1, 2].

With a purpose to increase the ANFO explosive properties, producers of this industrial explosive successfully implement the porous ammonium nitrate (PAN) instead of the ordinary one, which is also used as a mineral fertilizer [3]. PAN is characterized by the developed network of pores not only on the granule surface but also in its near-surface layers. Although the ANFO cost is increased thanks to PAN use, this step is economically beneficial, because costs for PAN production are compensated by the ANFO volume reduction in filler.

The granulated PAN quality produced for the mining industry is evaluated by the following indicators [4]:

1. Absorptivity – the amount of the diesel fuel distillate (in percent of the total mass of the granule), which is absorbed by PAN during its humidification by the diesel fuel distillate.

2. Retentivity – the amount of the diesel fuel distillate (in percent of the total mass of the granule) which remains in the granule after its transportation and excess drain.

3. The hardness of the PAN granule.

The absorptivity is defined by the total amount of pores (their form and nature are not paid much attention, the distinctive factor is their size), and by the de-

veloped nanoporous structure, which mostly consists of macropores (the size is over 50 nm). The normative indicator of the retentivity is provided by the internal "modified" nanoporous structure. This structure includes twisting mesopores (size is 2-50 nm). The hardness of the granule is defined by the degree of destruction (or integrity) of the core. The low hardness of the granule negatively influences the ecological indicator in production (dust) and leads to the slumping ability of granules. Thus, the developed nanoporous structure has to be provided on the surface of the granule and its near-surface layers.

The aim of the article is to evaluate the impact made by the hydrodynamic operation regime of the vortex granulator on the features of PAN nanoporous structure and to choose the optimal hydrodynamic conditions to obtain the developed nanoporous structure. Therefore, it is important to provide the normative quality indices of PAN on its base. In order to provide stable regimes in the work of the granulated unit, this research proposes a new construction of the vortex granulator with the stabilized zone for fluidizing agent.

### 2. DESCRIPTION OF OBJECT AND METHODS OF RESEARCH

In order to study the influence made by the hydrodynamic regime of the vortex granulator's operation on the nanoporous structure of the granule in the laboratory of the department "Processes and Equipment of Chemical and Petroleum Refinery Department", the experimental stand was created.

The operation principle of the experimental stand is the following. The simple granulated ammonium nitrate is used as the main raw material for the experimental unit. A portion of the ammonium nitrate is poured from the bag into the bunker, and then it is fed into the workspace of the vortex granulator. In order to regulate the flow of the ammonium nitrate into the granulator, the slide shutter is used.

The atmospheric air is used as a solubilizer in the vortex granulator. The atmospheric air is blown by the high-pressure air blower. In order to prevent foreign objects to be sucked into the air pipeline, the air gauze filter is set in front of the air blower. The air is fed by the air blower into the heater, where it is heated to the working temperature and then it is fed into the bottom part of the granulator. The air flow is regulated by a damper. In order to control the temperature of the air, flown into the granulator, the automatic temperature controller is provided. A sensor to control the air temperature is installed in the bottom part of the granulator. The signal from the sensor enters the automatic digital regulator, mounted on the control panel. The temperature controller is equipped with a manual mechanism to set the heated air temperature. The temperature controller is connected to the electromagnetic relay, which closes and opens the contacts regarding the inclusion of the heater's electric heating elements, keeping the fixed air temperature in the granulator's bottom part. An experimental stand is a unit of the periodic action, which provides its operation in four different technological regimes: heating and humidification, drying, cooling and outloading. That is why it is preferable to implement the heating controller with 4 and more positional setter of the working temperature.

Besides, the separately prepared solution is fed into the granulator. The prepared solution is put into the tank; after that, it is sealed and is connected to the pressure inlet of the air compressor. The compressed air displaces the solution from the tank by the flexible hose through a control valve, set on the control panel. The solution is flown to the granulator on the sprayer, which sprays the liquid onto the fluidized bed of the granules. In order to regulate the solution flow into the granulator's workspace, the measuring instrument of the liquid level is provided.

The final product (PAN) is outloaded through the outloading pipe in the bottom part of the granulator. The outloading is carried out by way of turning the outloading pipe to the "open" position. Therefore, it is necessary to set the container for the final product under the outloading pipe.

In order to reduce the dust in the room, where the experimental unit is installed, the air blower, which is similar to the air blower to supply the air, is set on the outlet pipe of the granulator.

Corrugated aluminium or stainless steel hose is used for the air supply and removal to the experimental stand.

The silicone reinforced hose is used to feed the solution from the head tank to the granulator. The compressed air from the compressor to the head tank is fed through the flexible plastic hose.

The vortex granulator is a module construction, which consists of body, made of the stainless steel and

equipped with observation glasses, bottom, intermediate ring, demountable cap, gas distributor, outloading pipe, and sprayer.

There is a stabilization grate between the bottom and the ring of granulator to improve the airflow before feeding to the gas distributor.

The supports of the granulator and bunker are equipped with regulating screws to control the horizontality of the gas distributor in the granulator and stable location of the equipment on the frame. The granulator is constructed in such a way that enables to change the gas distributor construction, which differs from the basic one by configuration and height.

During the installation of the distribution element represented by the downfall perforated grid in the medium part of the ring-type catcher of granules, it is possible to redistribute the heat transfer over the whole cross section in the ring-type catcher of granules, till they reach the vortex gas-distributing unit. In this case, the heat transfer comes to the vortex gas-distributing unit over its whole cross-section, which decreases the probability of the stagnant zones appearance in the bottom of the workspace and greatly intensifies the lateral mixing of granules. The reduction of the stagnant zones and intensification of the lateral mixing of granules cause the levelling of the temperature fields of material in the weighted layer. It enables to carry out the application of the solution or fusion film and granule formation with the same thermodynamic conditions for all granules. On the other hand, the distributive element in the construction of the mentioned device in the form of a downfall perforated grid in the middle of the ring-type catcher of granules enables to simplify the removal of granules due to their linear redistribution in the ring-type catcher of granules.

The dispersing phase is dosed through conveyor with multiengine drive synchronization.

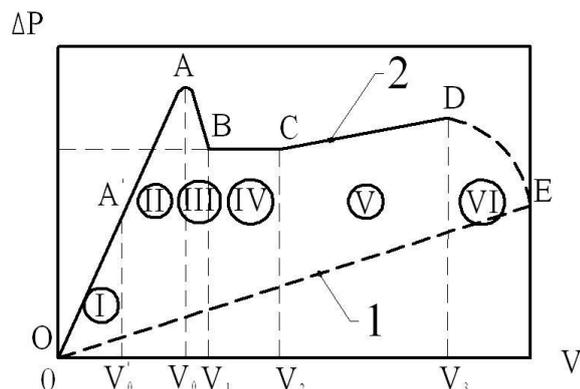
Devices and equipment:

- determination of the hydrodynamic features of the flows motion – TES 1340 Hot-Wire Anemometer;
- temperature measurement in air heater – selfrecording-register potentiometer;
- measurement of granulator workspace temperature – thermal imager Fluke Ti25;
- measurement of moisture granules – Multimeter DT-838;
- measurement of granules' strength – extensometer, device for measuring the strength;
- measurement of retention granules – small-sized centrifuge corner;
- study of the microstructure of granules – microscope KONUSPIX-450X KONUS, scanning electron microscope VEGA3 XM and X-ray spectrometer with an energy dispersion.

The nanoporous structure of the granule, obtained in every regime of the vortex granulator, has its own peculiarities. The dehydration process was carried out in all regimes during 11-12 min, the drying agent temperature was 90-110 °C [5]. The humidification was carried out with the ammonium nitrate solution, drying was carried out from 1 to 0.6 mass percent, the final drying in the gravitational shelf dryer [6, 7] – from 0.6 to 0.2 mass percent.

### 3. RESULTS AND DISCUSSION

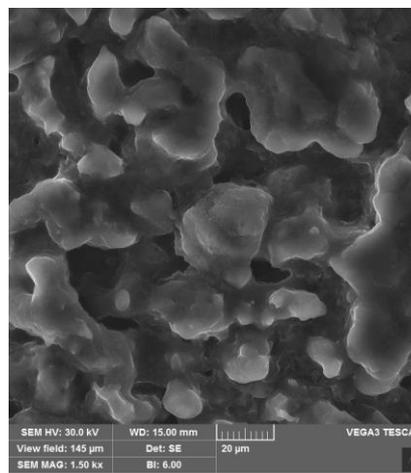
The figures below demonstrate a qualitative change of the porous structure in the PAN granule with an increase of the fluidized bed motion intensity. Demonstration of the main regimes regarding the vortex granulator operation let to evaluate the intensity of the mutual movement of granules in different zones. The intensity can be described by the gas flow velocity, which complies with every hydrodynamic regime. This velocity depends on the diameter (mass) of the granule and the flow restriction degree. According to data [5], Fig. 1 represents the qualitative change of the fluidization agent velocity, which complies with every regime of the vortex granulator's work.



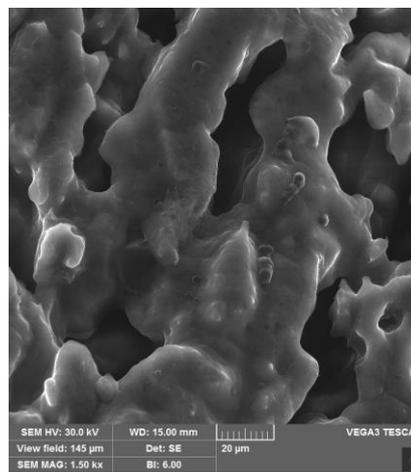
**Fig. 1** – Dependence of differential pressure in the fluidized bed owing to the gas flow velocity: 1 – differential pressure of the gas flow; 2 – differential pressure in the layer; OA' – filtering of the gas flow through the stationary layer; A'A – start of the layer removal; AB – partially fluidized bed; BC – fluidized bed with partial swirling; CD – developed vortex fluidized bed; DE – granules stable ablation regime;  $V_0$  – minimum velocity of the fluidized bed “start-off (transfer to the mobile state),  $V_0$  – minimum velocity of fluidization;  $V_1$  – total velocity of fluidization,  $V_2$  – total velocity of the vortex fluidization;  $V_3$  – velocity of ablation

With the increase of the fluidized bed motion intensity one can observe the following patterns (for example, Fig. 2 and Fig. 3 show the features of fluidized bed motion and nanoporous structure of PAN granules in the working regimes):

- due to the heat-mass transfer processes intensification and dehydration activation with new mesopores formation, their number is growing. This indicator influences the absorptivity of the granule;
- with the increase of the motion intensity, there are curvilinear micropores in the structure of granules. The curvilinear micropores influence the granule's retentivity;
- the porous surface area gradually increases, that is especially seen for three last regimes;
- the depth of the curvilinear micropores “formation” increases. Therefore, such regime is taken with which the granule has an integral core without “modified” pores (“mechanical” pores are possible as a result of the temperature stresses inside the granule and defects of the initial granule);
- due to the motion and smash intensity growth, granules start to lose their hardness (therefore within this work the normative hardness indicator is provided).



**Fig. 2** – Fluidized bed with partial swirling



**Fig. 3** – Developed vortex fluidized bed

Table 1 demonstrates the measuring and calculating results of the quantitative indicators (properties of the granule), which confirm the above qualitative description.

### 4. CONCLUSIONS AND RECOMMENDATIONS

Implementation of the proposed method to obtain PAN in the vortex gas flow enables:

- to create the developed porous structure on its surface and in the near-surface layers within the core of

**Table 1** – Effect of hydrodynamic mode on granule's structure and quality (humidifier – solution of ammonium nitrate, thermodynamic and hydrodynamic characteristics of granulator operation according to [5])

Hydrodynamic mode (number of figure with the configuration of fluidized bed and granule's structure)	Strength, kg/granule	Absorption capacity, %	Holding capacity, %	Relative area of porous surface, %	Relative depth of porous layer, the layer depth mm / radius granule
I – gas flow filtering through stationary layer of granules	0.45	8	5.3	38	≈ 0.1
II – beginning of the granules layer moving	0.45	9	6.5	54	0.1-0.15
III – partially fluidized bed	0.44	9.2	7	60	0.15-0.2
IV – fluidized bed with partial swirling	0.42	9.5	7.8	74	≈ 0.2
V – developed vortex fluidized bed	0.4	11.4	8.7	84	0.2-0.32

the fluidized bed at the initial stage to contact with high-temperature heat transfer agent's vortex flow till the granule reaches the surface of the fluidized bed;  
– to prevent the formation of granules with a form, which differs from the spherical one;  
– to exclude the impact on the dispersing of the fluid material uneven feeding into the disperser.

The obtained PAN samples were tested to define the explosive features of the industrial explosive ANFO. The detonation velocity of ANFO was determined by the Dotrsh method (it is based on the comparison of the known detonation velocity of the detonating chord with the unknown velocity of ANFO detonation). The industrial explosive (ANFO) charges based on the 95 % of PAN and 5 % of the diesel fuel distillate reliably detonate from the intermediate charge – trotyl block, which is initiated by the electric detonator. The ANFO detonation velocity was 2.2-2.3 km/s.

It is possible to regulate the process regarding the nanostructured porous layer formation with the specified properties owing to the selection of the optimal hydrodynamic regime to lay the humidifier's film and granules heat treatment. The results of the theoretical and experimental research carried out by the authors enable to perform the optimization projection of the main equipment in the unit to obtain PAN, i.e. the vortex granulator.

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## Оцінка впливу гідродинамічного режиму роботи грануляційного обладнання на нанопористу структуру гранул $N_4HNO_3$

А.Є. Артюхов<sup>1</sup>, Я. Крмела<sup>2</sup>, О.М. Гавриленко<sup>3</sup>

<sup>1</sup> Сумський державний університет, вул. Римського-Корсакова, 2, 40007 Суми, Україна

<sup>2</sup> Тренчинський університет Александра Дубчека, вул. І. Краску, 491/30, 02001 Пухов, Словаччина

<sup>3</sup> ТОВ «Е.М.А», вул. Харківська, 111, 40007 Суми, Україна

Стаття присвячена вивченню впливу гідродинамічних характеристик роботи вихрового гранулятора і інтенсивності направлено вихрового руху теплоносія на структуру і якість пористого поверхневого шару і внутрішньої нанопористої структури гранул аміачної селітри. Представлені результати аналізу показників якості пористої аміачної селітри (ПАС), яка отримана в різних гідродинамічних режимах. Вивчено структуру гранули, що отримана у вихровому газовому потоці з різною інтенсивністю закрутки. Отримані дані є основою для створення методики інженерного розрахунку вихрових грануляторів у складі установок отримання 3D наноструктурованого пористого поверхневого шару на гранулі аміачної селітри.

Отримані зразки ПАС були випробувані для визначення вибухових характеристик промислової

вибухової речовини "Аміачна Селітра/Дизельне Паливо" (АСДП). Швидкість детонації АСДП визначалася методом Дотріша (заснований на порівнянні відомої швидкості детонації детонуючого шнура з невідомою швидкістю детонації АСДП). Заряди промислової вибухової речовини АСДП на основі 95 % ПАС і 5 % дистилляту дизельного палива надійно детонують від проміжного заряду – тротилової шашки, яка ініціюється електродетонатором. Швидкість детонації АСДП склала 2.2-2.3 км/с. Управління процесом утворення наноструктурованого пористого шару з заданими властивостями стає можливим за рахунок підбору оптимального гідродинамічного режиму нанесення плівки зволожувача і термообробки гранул. Результати теоретичних і експериментальних досліджень, проведених авторами, дозволяють провести оптимізаційне проектування основного обладнання установки отримання ПАС – вихрового гранулятора.

**Ключові слова:** 3D наноструктурований пористий шар, Вихровий гранулятор, Гідродинаміка, Гідродинамічні режими.