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Development of Technology for Obtaining N_4HNO_3 Multilayer Granules with Nanostructured Porous Layers

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The article justifies obtaining of the N_4HNO_3 multilayer granules with nanostructured porous layers in a workspace of a multistage vortex granulator. The selection criteria for an optimal thermodynamic regime, used to obtain nanostructured porous layers at different stages of the granule formation, are specified. The microscopic analysis findings of the granule's nanoporous structure are presented. Each layer of the granule, obtained in the vortex gas flow with different types of the moisturizing solution, was examined. The data obtained are the basis of the engineering calculation methods for the vortex granulators as a part of the unit for production of N_4HNO_3 multilayer granules with nanostructured porous layers.

Key words: 3D Nanostructured porous layer, Vortex granulator, Multilayer granule.

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1. INTRODUCTION

Ammonium nitrate (N_4HNO_3) is a required component of the free flowing industrial explosives, and when blended with diesel fuel distillate (an international term of the explosive is ANFO), it possesses efficient explosive properties [1, 2].

Typical ammonium nitrate, used as an ANFO component, reduces the detonation velocity of this industrial explosive. The reason for such a reduction lies in the peculiar structure of the typical ammonium nitrate granule: a prevailing number of «mechanical» pores – cracks, splits and spalls on the surface; a low number of «modified» pores, which appear when water and ammonia gas vapors release from the granule's core to its surface; pores are located on the surface only, which enables diesel fuel distillate to penetrate the near-surface layer of the granule without retention in it.

To increase the blasting properties of ANFO, the producers of this industrial explosive successfully apply porous ammonium nitrate (PAN) instead of typical [3]. PAN is characterized by the developed network of pores on the surface of the granule, as well as in its near-surface layers. Despite increase in the cost of ANFO, this method is profitable, as far as the PAN production costs are compensated with the reduced amount of ANFO in the blasting charge. Among the existing methods of the PAN production [4], a combined approach (to apply humidification and further thermal processing of the typical ammonium nitrate granule) is one of the most ecologically safe.

Thus, the developed network of pores on the surface enables diesel fuel distillate to penetrate easily the near-surface and deep layers of the granule without retention in it. Reliable retention of diesel fuel distillate is possible in case granules contain the winding nano-sized pores. Such a network can be formed due to different modified additives supplied to ammonium nitrate [5]. This solution increases the production costs and thus compels to search for the less expensive PAN production.

As shown in works [6, 7], there is a necessity to ensure the properties of the PAN granules as follows:

- preservation of the primary crystal structure and phase composition of the granule's core;
- a solid core without mechanical damages;
- a minimum amount of "mechanical" pores in the granule's volume;
- internal layers with some amount of micropores (size less than 2 nm) and mesopores (size between 2 and 50 nm);
- middle layers, containing mostly mesopores and some amount of macropores (size over 50 nm);
- the surface, composed mostly of macropores.

The article aims to justify a possibility to obtain the PAN multilayer granules with nanostructured layers as a component of the ANFO industrial explosive. In this regard, the principal "thermodynamic" implementation of each stage when obtaining the multilayer granules (a change of the thermodynamic operation picture of a vortex granulator in relation to time) is important. The reasonability of obtaining the multilayer structure of granules with different properties is based on a number of case studies, dealing with such granules implemented in the mineral fertilizers production, by Chinese [8], Romanian [9], Thai [10] and Ukrainian [11] researchers.

To improve the quality and to reduce costs of the PAN multilayer granules with nanostructured layers, this work offers the use of multistage vortex granulators [12]. Such a type of the granulator ensures continuous and consecutive application of the moisturizing film and thermal processing of a granule. Granulators with a fluidized bed of granules (vortex in particular) ensure high specific productivity within small dimensions [13-15]. Herewith, the number of thermal processing cycles is not increased, that keeps the granule's core solid. The computer simulation of hydrodynamic and thermodynamic operational conditions of the vortex granulators [16, 17] has justified theoretically an opportunity to control the temperature field in the device.

2. DESCRIPTION OF THE OBJECT AND METHODS OF THE RESEARCH

To elaborate the technology for obtaining the N_4HNO_3 multilayer granules with nanostructured porous layers, a test stand was installed in the laboratory of the Processes and Equipment of Chemical and Petroleum Refinery Department (Fig. 1).

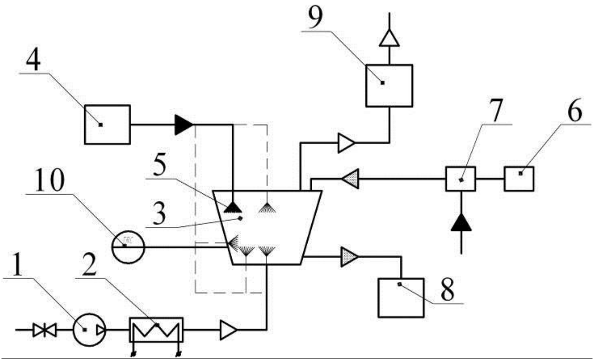


Fig. 1 – Experimental stand of granulator: 1 – gas blower; 2 – heater; 3 – vortex granulator; 4 – container for preparation of humidifier; 5 – nozzle; 6 – container for granules; 7 – pre-wetting capacity (used for humidifying); 8 – container of commodity granules; 9 – waste gas cleaning stage (bubbler); 10 – selfrecorder registration potentiometer; the dotted line shows the nozzle installation variants

Devices and equipment: determine the hydrodynamic characteristics 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 pellets – microscope KONUSPIX-450X KONUS, scanning electron microscope SEM-100U and X-ray spectrometer with an energy dispersion.

3. RESULTS AND DISCUSSIONS

Formation of the nanostructured porous layer with the given properties can be controlled due to the optimal selection of the hydro- and thermodynamic conditions for the application of the moisturizing film and the thermal processing of the granule. The theoretical and experimental research results, obtained by the author, enable optimization projection of the main equipment for the PAN production – a vortex granulator.

A change of the thermodynamic operation picture of the vortex granulator in relation to time may be depicted as follows (Fig. 2):

1) when the nanoporous layer is formed near the core, the main provision for the operation is no overheating of a granule. In this case, the maximum temperature in the workspace of the granulator must be in the "active zone", near the gas distribution unit (Fig. 2a);

2) when the next layers are formed, the maximum temperature front must shift from the «active» zone to the central part of the granulator (Fig. 2 b,c);

3) when the surface nanoporous layer is formed, the maximum temperature front must be localized in the central part of the granulator (Fig. 2 d).

The specific thermodynamic conditions of each layer are determined by the characteristics of the moisturizer.

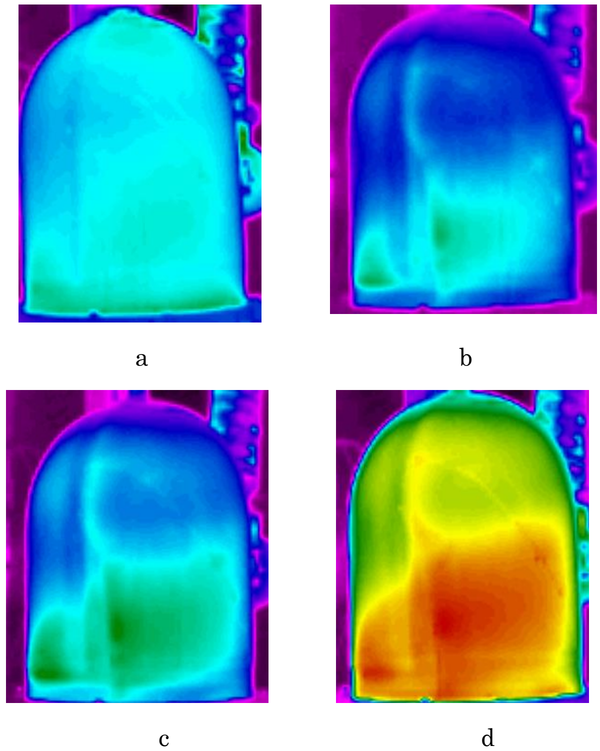
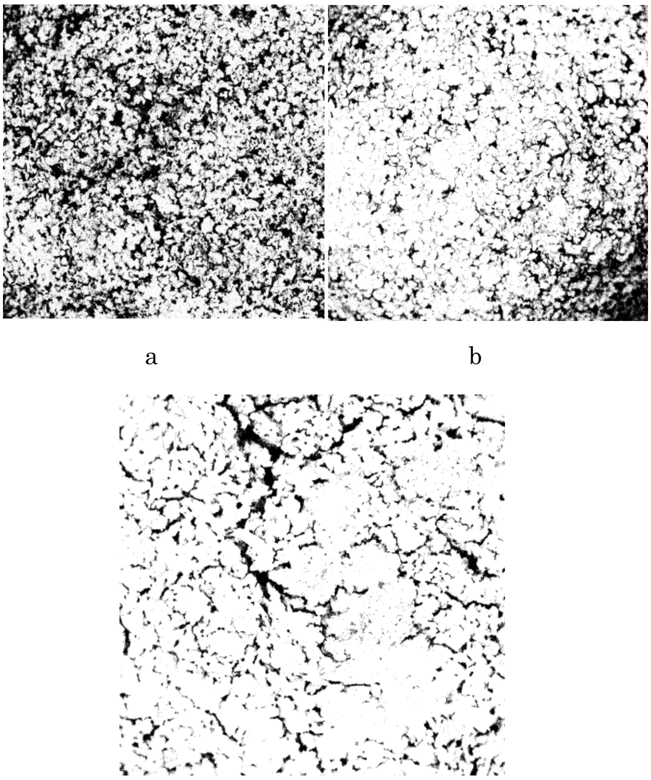


Fig. 2 – Thermodynamic operational conditions of the vortex granulator: a – heating of the "active" zone; b, c – shift of the maximum temperature of the drying agent to the central part of the granulator; d – formation of the maximum temperature zone of the drying agent in the central part of the granulator

Figures 3 and 4 depict the structure of the PAN granule surface at different development stages of the multilayer nanoporous composition. The structure meets the requirements for the system of pores on the surface and inside the granule.

The research results helped to identify the following characteristics of the method, used to obtain the PAN multilayer granules with nanostructured layers:

- an optimal consumption of the heating agent, which meets the range of the operational stability of the vortex granulator from the velocity of the initial vortex motion of granules to the developed vortex motion;
- an optimal temperature and humidity of the heating agent, ensuring heating and dehydration of the PAN granules without considerable overheat;
- the necessary consumption of the moisturizer at each stage;
- an optimal temperature distribution field of the heating agent in the workspace of the vortex granulator;
- minimal residence time of the PAN granules in each



a b c

Fig. 3 – PAN granule surface structure: a – after the first stage of humidification and thermal processing (water as a moisturizer); b – after the second stage of humidification and thermal processing (ammonium nitrate solution as a moisturizer); c – after the third stage of humidification and thermal processing (ammonium nitrate and carbamide solution as a moisturizer)

section of the vortex granulator in dependence with the requirements to the final moisture content and nanoporous structure of each layer.

4. CONCLUSIONS

The application of the proposed method of the PAN production in the vortex gas flow enables us to:

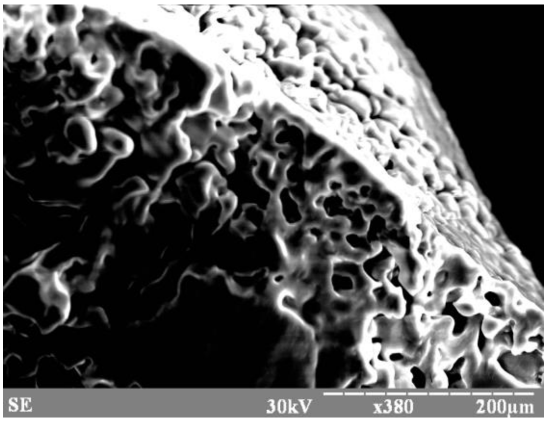


Fig. 4 – PAN multilayer granule structure after the full production cycle completed (granule's section)

- form a developed porous structure on the surface and in the near-surface layers within the core of the fluidized bed at the initial contact stage of the vortex flow and high-temperature heating agent before the granule release to the "glass" of the fluidized bed;
- prevent formation of the non-spherical shaped granules;
- eliminate entirely the influence on the dispersion of the non-uniform supply of the liquid material into the disperser.

The obtained PAN samples were tested to determine the blasting properties of the ANFO industrial explosives. The ANFO detonation velocity was determined using the Dautriche method (comparison of the known detonation velocity of the detonating cord with the unknown detonation velocity of ANFO). The charges of the ANFO industrial explosive, composed of 95% of PAN and 5% of diesel fuel distillate, are reliably detonated by the intermediate charges – the trotyl block, ignited by an electric detonator. The ANFO detonation velocity makes 2.2 – 2.3 km/s.

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Розробка технології отримання багатошарових гранул N_4HNO_3 з наноструктурними пористими шарами

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Стаття присвячена обґрунтуванню можливості отримання багатошарових гранул N_4HNO_3 з наноструктурними пористими шарами в робочому об'ємі багатоступінчастого вихрового гранулятора. Запропоновано критерії підбору оптимального термодинамічного режиму отримання наноструктурних пористих шарів на різних стадіях формування гранули. Представлені результати мікроскопічного аналізу нанопористої структури гранули. Досліджено структуру кожного шару гранули, отриманого в вихровому газовому потоці з різним типом зволожуючого розчину. Отримані дані є основою для створення методики інженерного розрахунку вихрових грануляторів в складі установок отримання багатошарових гранул N_4HNO_3 з наноструктурними пористими шарами

Ключові слова: 3D Наноструктурований пористий шар, Вихровий гранулятор, Багатошарова гранула.

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