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Nanoporous Structure of the Ammonium Nitrate Granules at the Final Drying: the Effect of the Dryer Operation Mode

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The article deals with the study of the hydrodynamic operation modes effect, made by the gravitational shelf dryer for final drying, on the structure and quality of the porous surface layer and inner nanoporous structure of the ammonium nitrate granule. Therefore, the main requirement of granules is to keep the hardness of the granule and to form a minimum quantity of "mechanical" pores. The results of the quantitative and qualitative analysis regarding the indicators of the porous ammonium nitrate (PAN), which was produced in different hydrodynamic operation modes of the gravitational shelf dryer, were demonstrated. The additional final drying in the falling velocity mode in the active (but less turbulized) hydrodynamic mode enables to achieve the following changes in the nanoporous structure of granules (in comparison with under-dried sample): an increase in the number of micropores and mesopores with the curvilinear configuration; an increase in the curvilinear macropores in the total number of nanopores; an increase in the surface nanopores deepness. It should be noted that the final drying of PAN does not cause a significant increase of the retentivity index. The second task of the final drying is to remove the bound moisture and to reduce the humidity of the granule. As a result, the caking of granules is reduced (ammonium nitrate is very hygroscopic) and the retentivity index is kept for a long period. Introduction of this stage will increase the general energy capacity of the production; however, implementation of the multistage shelf dryers enables to reduce the specific costs for removal of the moisture. The received data are the base to create the technological calculation technique of the final drying stage to obtain the 3D nanostructured porous surface layer on the ammonium nitrate granule.

Keywords: 3D nanostructured porous layer, Gravitational shelf dryer, Final drying, Hydrodynamics, Hydrodynamic modes.

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1. INTRODUCTION

Production of the porous ammonium nitrate (PAN) is one of the main stages to produce the industrial explosives based on the ammonium nitrate and diesel fuel (oil) distillate — ANFO [1]. The nanoporous structure of the granule is formed in order to provide the normative index regarding the absorptivity and retentivity of the granule in relation to the diesel fuel distillate.

Therefore, the absorptivity mainly depends on the pores or mechanical damages (cracks, caverns). High absorptivity can be provided in the granule, which is damaged owing to the mechanical and thermal effects. The retentivity of the granule depends on the configuration of the pores and their size (particularly, proportionality with the diesel fuel molecule), and on the hardness of granules. Granules with non-standard (lower) hardness tend towards destruction during transportation, and as a result towards the sharp decrease of the curvilinear nanopore number.

It should be mentioned that the granule can be also destructed at the stage of the nanoporous structure formation during the humidification and heat treatment, e.g. in vortex granulators [2]. Despite the satisfactory results of the vortex granulator introduction into the PAN production technology, the object of the research includes mechanisms to control the time of the granule staying in the workspace of this device. In order to exclude the possibility of the granule overheating and its collision with other granules and the wall of the device in the intensive flow of the twisted heat transfer agent, this work continues to study the additional drying stage. The necessity to introduce the final drying with the use of

the multistage shelf devices [3] is proved in the work [4] in more detail.

The additional final drying in the falling velocity mode [5] in the active (but less turbulized) hydrodynamic mode [6] enables to achieve the following changes in the nanoporous structure of granules (in comparison with under-dried sample):

- an increase in the number of micropores and mesopores with the curvilinear configuration;
- an increase in the curvilinear macropores in the total number of nanopores;
 - an increase in the surface nanopore depth.

These changes let to an increase in the retentivity index of the granule and time of the safe retentivity of the diesel fuel distillate.

The aim of this research is to study the effect, made by the hydrodynamic mode of the gravitational shelf dryer on the nanoporous structure of the PAN granule. Therefore, the main requirement of granules is to keep the hardness of the granule and to form a minimum quantity of "mechanical" pores.

2. DESCRIPTION OF OBJECT AND METHODS OF RESEARCH

In order to study the influence made by the hydrodynamic regime of the gravitational shelf dryer's operation on the nanoporous structure of the granule after final drying in the laboratory of the department "Processes and Equipment of Chemical and Petroleum Refinery Department", the experimental stand was created (Fig. 1). The operation principle of the experimental stand was described in [4].

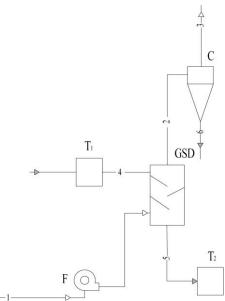


Fig. 1 – Schematic diagram of the experimental setup for the shelf dryer: F – fan; GSD – gravitational shelf unit; C – cyclone; T1, T2 – containers (tanks); 1 – drying agent; 2 – waste drying agent; 3 – purified gas; 4 – PAN; 5 – PAN after final drying and 6 – fine particles

The work [3] defined the following main modes of the gravitational shelf dryer operation, which would be modelled in the final drying of the ammonium nitrate with nanoporous structure:

- 1. The gravitational falling layer mode. This mode was studied with a flow rate of drying agent of 0.41 m³/min and dispersed material of 12 kg/h. The dispersed material moves through the surface of the shelf thanks to the inertial force, caused by the transfer of impulse when it is loaded from the pipe or transferred from the previous shelf, and rolling force on a sloping surface. At the same time, the upward flow force of the drying agent does not have a great impact on the dispersed material motion mode.
- 2. Transition mode. If the flow rate of the drying agent increases to $0.5~\rm m^3/min$, the force of its upward agent, while keeping the flow rate of the dispersed material, will lead to the gradual change of its motion trajectory from translational to translational and pulsating in the direction of the outloading gap. At the same time, the dispersed material begins to move into the weighted state, the inertial force is compensated by the upward flow force of the drying agent; the forward direction of the motion is caused only by the rolling force on the sloping surface.
- 3. Moving fluidized bed. If the flow rate of the drying agent increases to 0.55-0.6 m³/min, the force of its upward flow with a constant flow rate of the dispersed material will lead to the creation of the stable weighted layer owing to compensation of the inertial force and rolling force on the sloping surface.

The study of the nanoporous structure in the granule was carried out with the use of the author's program Converter Image[©] (Fig. 2).

Technological parameters of the final drying:

- the drying temperature is 90-96 °C.
- a number of shelves in the gravitational shelf dryer is equal to 3.

- time of the PAN granules staying on one shelf, s - gravitational falling layer - 10-20, transition mode - 20-40, moving fluidized bed - 40-100.

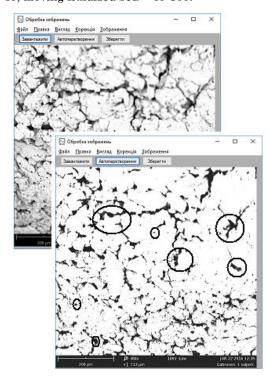


Fig. 2 – Interface of the program Converter Image^{\mathbb{C}} to study the nanoporous structure

3. RESULTS AND DISCUSSION

Fig. 3, Fig. 4 demonstrate the results to define the quantitative composition of nanopores with different sizes on the surface of the granule and in its inner structure.

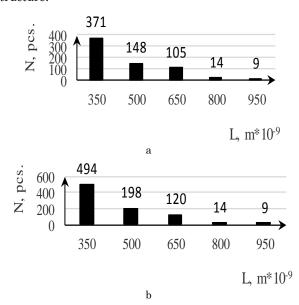
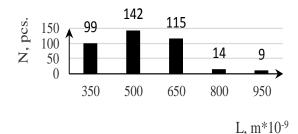


Fig. 3 – Quantitative composition of pores (per $1000~\mu m^2)$ in the inner structure of the granule: a – the granule after the vortex granulator; b – the granule after final drying in the gravitational shelf dryer in the moving fluidized bed mode



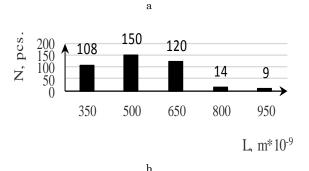


Fig. 4 – Quantitative composition of pores (per $1000~\mu m^2)$ on the surface of the granule: a – the granule after the vortex granulator; b – the granule after final drying in the gravitational shelf dryer in the moving fluidized bed mode

Results of the studies regarding the PAN granule nanoporous structure after the multistage final drying showed the following regularities:

- new pores are not practically formed on the surface of the granule, hydrodynamic mode of the dryer operation influences only the formation of the inner pores;
- a number of macropores on the surface and inside the granule does not grow;
- the hardness of the granule is not changed;
- absorptivity of the granule is not changed, the retentivity of the granule grows.

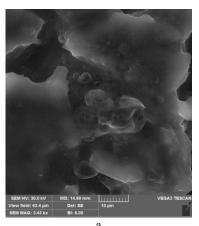
Taking into account the quantitative study results, only the inner nanoporous structure will be demonstrated in the article (Fig. 5, Fig. 6).

The results of the microscopy data analysis showed that:

- thanks to the bound moisture removal from the inner layers of the granule in the falling drying velocity mode (with further output through the macropores formed on the surface in the vortex granulator), an extensive network of deep curvilinear (wavy) mesopores of the "modified" nature is created;
- pores are evenly distributed inside the whole granule;
- a number of the "mechanical" pores does not grow.
- smaller pores are not practically merged together into ensembles;

The generalized results of investigations are demonstrated in Table 1.

It should be noted that the final drying of PAN does not cause a significant increase of the retentivity index. The second task of the final drying is to remove the bound moisture and to reduce the humidity of the granule. As a result, the caking of granules is reduced (ammonium nitrate is very hygroscopic) and the retentivity index is kept for a long period.



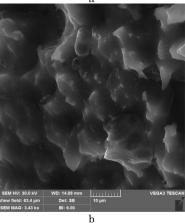
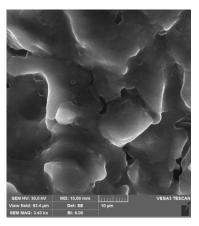


Fig. 5 – Results of the granule inner structure microscopy after final drying in the gravitational shelf dryer: a – the granule after drying in the gravitational falling layer mode; b – the granule after drying in the transition mode



 ${f Fig.~6}$ – Results of the granule inner structure microscopy after final drying in the gravitational shelf dryer: the granule after drying in the moving fluidized bed mode

4. CONCLUSIONS AND RECOMMENDATIONS

The obtained results prove that the final drying stage of the PAN granules provides the increase of their quality indices and storage time without the retentivity loss in relation to the diesel fuel distillate.

Introduction of this stage will increase the general energy capacity of the production; however, implementation of the multistage shelf dryers enables to reduce the specific costs for removal of the moisture thanks to:

 $\textbf{Table 1} - \textbf{Effect of hydrodynamic mode of final drying in gravitational shelf dryer on granule's structure and quality (humidifier on the stage of treatment in vortex granulator - solution of ammonium nitrate)$

Hydrodynamic mode	Absorptivity,	Retentivity,	The ratio of the number of the modified pores to the total number of pores ("modified"+ "mechanical")
The initial granules (after vortex granulator) before final drying	11.4	8.7	0.79
Gravitational falling layer	11.4	8.8	0.8
Transition mode	11.5	9	0.83
Moving fluidized bed	12	9.6	0.87

- sectioning of the inner space of the device with the formation of some stages in the heat and mass transfer process;
- differentiated distribution of the interacting flows between stages;
- gradual regulation of the heat and mass transfer driving force;
- optimization of the interacting flows costs ratio;
- creation of the active hydrodynamic mode regarding the flows interconnection.

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Нанопориста структура гранул аміачної селітри на стадії фінального сушіння: вплив режиму роботи сушарки

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Стаття присвячена вивченню впливу гідродинамічних режимів характеристик роботи гравітаційної поличної сушарки для фінального сушіння на структуру і якість пористого поверхневого шару і внутрішньої нанопористої структури гранул аміачної селітри. При цьому основною вимогою до гранул є збереження міцності ядра і мінімальне утворення «механічних» пор. Представлені результати кількісного та якісного аналізу показників якості пористої аміачної селітри, яка отримана в різних гідродинамічних режимах роботи гравітаційної поличної сушарки. Додаткова стадія досушки в режимі падаючої швидкості в активному (але менш турбулізованому) гідродинамічному режимі дозволить досягти таких змін в нанопористій структурі гранул (у порівнянні з недосушеним зразком): збільшення кількості мікропор і мезопор криволінійної конфігурації; збільшення частки криволінійних макропор в загальній кількості нанопор; збільшення глибини поверхневих нанопор. Слід зазначити, що фінальна сушка пористої аміачної селітри не призводить до значного збільшення показника утримуючої здатності. Другим завданням фінальної сушки є видалення зв'язаної вологи і зменшення вологості гранули. Як наслідок, зменшується злежуваність гранул (аміачна селітра дуже гігроскопічна) і на більш тривалий термін зберігається показник утримуючої здатності. Впровадження цієї стадії підвищить загальну енергетичну ємність виробництва; однак, використання багатоступінчастих поличних сушарок дозволяє знизити питомі витрати на видалення вологи. Отримані дані є основою для створення методики технологічного розрахунку стадії фінальної сушки установок отримання 3D наноструктурного пористого поверхневого шару на гранулі аміачної селітри.

Ключові слова: 3D наноструктурований пористий шар, Гравітаційна полична сушарка, Фінальне сушіння, Гідродинаміка, Гідродинамічні режими.