Multistage Finish Drying of the N₄HNO₃ Porous Granules as a Factor for Nanoporous Structure Quality Improvement

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The article deals with a study of ammonium nitrate granules structure with nanostructural porous layers, which are obtained after humidification and multistage finish drying. The necessity for an additional stage of granules dehydration in the technology to obtain porous ammonium nitrate is justified. Design of the multistage finish drying phase is represented in the study. The comparative analysis of the granule nanoporous structure after every finish drying stage in thermodynamic and hydrodynamic conditions is shown. The received data is a base to form a technique for an engineering evaluation of the multistage dryers with vertical sectioning of the workspace as a part of installations to obtain 3 D nanostructural layers on the surface of ammonium nitrate granule and inside it.

Key words: 3D nanostructural porous layer, Multistage shelf dryer, Crystal structure.

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1. INTRODUCTION

Ammonium nitrate granules are necessary components of the industrial explosives in a mixture with diesel fuel distillate (ANFO) [1, 2]. Given the relative cheapness, such an industrial explosive type lets to carry out wide range of explosive works in the mining industry [3]. It should be mentioned that the most popular way to create developed porous structure of the ammonium nitrate granules is to add poreforming and modification additives to the fusion with its further crystallization [4]. Such method is attended with an environmental degradation in the production zone and in the region on the whole [5].

Having analyzed a number of works [6, 7], it is evident that an environmentally friendly process to obtain nanoporous structure in the ammonium nitrate granules with high effectiveness can be fulfilled in the vortex granulators with variable cross-section height area. Therefore, the obtained porous ammonium nitrate (PAN) satisfies quality parameters, including the absorptivity towards the diesel fuel distillate.

However, final absorptivity index may be increased thanks to more intensive drying in the period of the reducing velocity (the second drying stage in accordance with regular terminology, see Fig. 1). It is possible to carry out this process with long-term drying of the dryer agent in the vortex flow only by the reducing of the granule strength. Much time of processing is required for the full finish drying, and the granule can be cracked, given the active flows turbulence. Retentivity is also reduced given the cracking and forming of "mechanical" pores (faults and cracks).

PAN granules with an excessive final humidity are characterized by the following peculiarities:

- underdeveloped nanoporous structure;

- a great number of "mechanical" pores, which have the inline configuration;

- weak core of the granule (as a result of many "mechanical" pores in the granule);



Fig. 1 – Periods of PAN granule drying: I – warming-up of the granule; II – period of the drying constant velocity; III – period of the drying reducing velocity

- preferential presence of the straightline macropores in the nanoporous structure of the granule that is explained by high intensity of dehydration during the drying growing velocity (warming-up of the material and the fist drying period in accordance with regular terminology).

An additional stage of the finish drying in terms of the reducing velocity in the active (but less turbulizing) hydrodynamic regime will let to achieve the following changes in the granules nanoporous structure (in comparison with an undried sample):

 expansion in the number of the curved configuration mesopores;

- increase of some curved macropores in the total number of nanopores;

- increase of the surface nanopores depth.

These changes allow to improve retentivity parameter of the granule and time of diesel fuel distillate firm retention in the granule.

This research proposes to introduce an extra stage of

the multistage finish drying in the differentiated regime to the existed technological scheme of PAN obtaining process (parameters of the dryer agent and dryable granules are changed at every stage of finish drying) (Fig. 2). As main technological equipment at this stage, one suggests to use gravitational shelf dryers, which proved themselves in the technology of porous concentrates drying in the mining industry [8].



Fig. 2 – Scheme of PAN obtaining process: 1 - humidification of ammonium nitrate granule; 2 - granulation-drying; 3 - finish drying; 4 - cleaning of waste gas

The aim of the article is to evaluate an impact of PAN granule finish drying regime on its nanoporous structure.

2. DESCRIPTION OF OBJECT AND METHODS OF RESEARCH

The scheme of test stand to investigate the finish drying process of granules in gravitational shelf dryers is demonstrated in Fig. 3. The test stand provides three stages of the finish drying.

Morphology of the NH_4NO_3 granules surface was investigated by method of scanning electronic microscopy by using device FEI Nova NanoSEM 650.

Other devices and equipment include:

- temperature in the calorifier is measured by TC10-C thermocouple; selfrecording potentiometer KCII-3;

- temperature in the workspace of granulator is measured by thermal imager Fluke Ti25, pyrometer Victor 305B;

- humidity of granules and air is measured by the multimeter DT-838.

Dryer agent, which is firstly regular spaced along body section and then rises up, is brought to the bottom part of the dryer. Therefore it gets through a definite number (in our case – three) of perforated inclined shelves, and then is taken from upper part of the device. At the same time as dryer agent is moving, PAN is brought to the body on the upper shelf, which has to be dried. On the upper inclined contact shelf during the contra-flow contacting with dryer agent flow



Fig. 3 – Schematic diagram of the experimental setup for the study of shelf devices: 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; 6 – fine particles

the unbound humidity is extracted from the surface layer of the disperse material in the period of drying constant velocity. PAN moves by the dryer agent flow to the upper shelf during drying, comes through its free end and appears in the middle shelf. In the process of disperse material moving along the middle shelf, the bound humidity is being intensively extracted during the reducing velocity of drying. During the gradual moving of the disperse material is removed through free end of the shelf and appears in the bottom shelf. As far as the disperse material is moving in the bottom shelf, the bound humidity extraction from the material depth is finished.

Unlike the vortex granulator, the finish drying of PAN granules is carried out in softer hydrodynamic regime. Therefore, the dehydration intensity is not reduced during the reducing velocity of drying in comparison with the vortex granulator. The finish drying process is conducted at temperature of 90 to 110 °C; minimum temperature of drying is provided at the first (upper) stage, maximum – on the last (bottom) stage. Drying time is 8-15 minutes depending on hydrodynamic regime of dryer work and humidity conditions of PAN granule.

3. RESULTS AND DISCUSSIONS

The structure of PAN granule, which is obtained in the optimal regime of the granulator work (equality of "thermodynamic" and "hydrodynamic" residence time of the granule in the device), is demonstrated in Fig. 4. MULTISTAGE FINISH DRYING OF THE N4HNO3 POROUS GRANULES...

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Fig. 4 – Structure of PAN granule, which is obtained in the optimal regime of the granulator work

The structure has some "mechanic" pores in form of cracks. In general, these cracks do not have an essential impact on the PAN quality. The granule strength exceeds the standard parameter, however, in order to provide necessary number of mesopores, it is necessary to classify granules by sizes and mass.

The Fig. 5 presents structure of PAN granule, which is obtained after drying in the vortex granulator with drying overtime. In the granule structure one can clearly see "mechanic" macropores, which mainly consist of inline cracks (Fig. 5a). There are also separate cracks of granule (Fig. 5b). This structure lets only to absorb diesel fuel distillate without its effective retention. Such obtaining way is possible only when it is necessary to provide free "entrance" of diesel fuel distillate into the inner layers of the PAN granule.





Fig. 5 – Structure of the PAN granule, which is obtained after drying in the vortex granulator with drying overtime: a - cracks; b - partial destruction of the granule

The Fig. 6 also shows the structure of PAN granule, which is obtained after drying in the vortex granulator. In this case drying time is defined by the parameter of the granule core retentivity. Drying is finished when core retentivity becomes lower, than a standard ones.

Great reduction of nanopores in any variety of sizes is observed on figures thanks to the humidified granule together with cooperation in particles group under the developed turbulent conditions (Fig. 6a). Besides, one can observe dissolution of the granule part with significant reduction of pores in the granule (Fig. 6b).

Fig. 7 demonstrates the structure of PAN granule after finish drying stage in the gravitational shelf dryer. One can see the branchy net of curving mesopores, which will absorb diesel fuel distillate. The size of these





Fig. 6 – Structure of PAN granule with an excessive humidity: a – abrading of the granule; b – dissolution of the granule part



Fig. 7 – Structure of the PAN granule after the finish drying stage in the gravitational shelf dryer

pores is adequate for diesel fuel distillate to come inside the granule. The form of pores prevents from great outflow of the diesel fuel distillate during ANFO transporting and storing processes.

The quantitative representation of pores in the PAN granule for an optimal hydro- and thermodynamic regime of gravitational shelf dryer work, is represented in Fig. 8.

4. CONCLUSIONS AND RECOMMENDATIONS

The results of investigations regarding nanoporous structure of the PAN granules after



Fig. 8 – Number and sizes of pores per unit of PAN granule surface area (granule, humidified with water): a – surface; b – subsurface layers

multistage finish drying have the following regularities:

 a branchy net of deep curving modified mesopores is created;

- pores are evenly distributed on the whole surface of the granule;

smaller pores are not practically united into assemblies;

- number of "mechanic" pores and destructions of the granule is being reduced.

Thus, it is better to carry out the process of nanopores creation in the vortex granulator within warming-up, the first and partially second periods of drying, if it is possible to extract standard parameter of humidity. Therefore, technological calculation of the granules classification process has to be clear. In other cases it is recommended to implement finish drying in another device.

The received results may be put to the technique of the drying devices engineering calculation with differential dehydration regime in different hydro- and thermodynamic regimes.

The task of further research is:

- to select an optimal thermodynamic regime to implement the finish drying process at every stage of the gravitational shelf dryer;

- to determine the optimal number of stages in the gravitational shelf dryer depending on humidifier type.

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Багатоступеневе досушування гранул пористої N₄HNO₃ як фактор підвищення якості нанопористої структури

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Стаття присвячена вивченню структури гранул аміачної селітри з наноструктурними пористими шарами, які отримані в результаті зволоження і багатоступінчастої досушки. Обґрунтовано необхідність додаткової стадії зневоднення гранул в технології отримання пористої аміачної селітри. Представлено конструктивне оформлення стадії багатоступінчастої досушки. Наведено порівняльну характеристику нанопористої структури гранули після кожної стадії досушування в різних термодинамічних і гідродинамічних умовах. Отримані дані є основою для створення методики інженерного розрахунку багатоступеневих сушарок з вертикальним секціонуванням робочого простору в складі установок отримання 3 D наноструктурних шарів на поверхні і всередині гранули аміачної селітри.

Ключові слова: 3D Наноструктурований пористий шар, Багатоступінчаста Полична Сушарка, Кристалічна структура.

Многоступенчатая досушка гранул пористой N₄HNO₃ как фактор повышения качества нанопористой структуры

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Статья посвящена изучению структуры гранул аммиачной селитры с наноструктурными пористыми слоями, которые получены в результате увлажнения и многоступенчатой досушки. Обоснована необходимость дополнительной стадии обезвоживания гранул в технологии получения пористой аммиачной селитры. Представлено конструктивное оформление стадии многоступенчатой досушки. Приведена сравнительная характеристика нанопористой структуры гранулы после каждой стадии досушки в различных термодинамических и гидродинамических условиях. Полученные данные являются основой для создания методики инженерного расчёта многоступенчатых сушилок с вертикальным секционированием рабочего пространства в составе установок получения 3 D наноструктурных слоёв на поверхности и внутри гранулы аммиачной селитры.

Ключевые слова: 3D Наноструктурированный Пористый слой, Многоступенчатая Полочная Сушилка, Кристаллическая структура.

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