

## Thermodynamic Conditions for Obtaining 3D Nanostructured Porous Surface Layer on the Granules of Ammonium Nitrate

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The paper deals with the study of the influence of thermodynamic conditions on the structure of a nanostructured porous surface layer on ammonium nitrate granules. Calculation of the heating of the granule and moisture removal from it for different thermodynamic factors of the coolant was conducted based on the theoretical model of drying and dehydration kinetics. The surface structure of granules obtained under different thermodynamic characteristics of the coolant was investigated. The obtained data is the base for selection of optimal technological parameters of vortex granulators being a part of devices for producing 3D nanostructured porous surface layer on the ammonium nitrate granule.

Keywords: 3D Nanostructured Porous layer, Granulator, Thermodynamics.

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### 1. INTRODUCTION

The quality evaluation of porous ammonium nitrate is determined by two factors: the retention and absorption capacities with respect to diesel fuel [1]. These parameters along with the granule hardness ensure the effectiveness of industrial explosives [2].

Production of a nanostructured porous surface layer on ammonium nitrate granules can be realized by several methods [3]. Among them, the method of moistening with subsequent heat treatment in a vortex fluidized bed has favorable advantages, such as

- improvement of the environmental performance of production;
- no loss of granule strength;
- simplified production scheme.

Despite the apparent simplicity, this method is very sensitive to the hydrodynamic and thermodynamic conditions of its realization. It is possible to operate the granule production process using vortex-type devices [1-5], which have proven themselves in many heat and mass exchange processes [6, 7]. The optimal hydrodynamic conditions of the vortex granulator operation, at which it is feasible to obtain a product with sufficient strength and minimum moisture content, were selected in [8, 9]. At the same time, the thermodynamic parameters of the vortex granulator working space should be chosen in such a way as to ensure uniform heating of the granule and moisture removal from it [10]. Underheating of the granule can lead to degradation of the porous surface layer structure, and overheating – to rapid evaporation of moisture from the granule, intense emission of oxygen and ammonia resulting in the formation of cracks and the granule core destruction.

The search for optimal thermodynamic conditions for obtaining a nanostructured porous surface layer determines the relevance and the main objective of the work.

### 2. DESCRIPTION OF THE OBJECT AND METHODS OF STUDY

The main tasks of the first part of the present work are the following:

- the investigation of heating kinetics of the granule and moisture removal from it;

– the study of the structure of a porous surface layer obtained under different thermodynamic conditions.

Based on the assigned tasks of experimental studies in the scientific research laboratory of the department “Processes and equipment of chemical and oil refinery industries” of Sumy State University, the experimental setup, the scheme of which is illustrated in Fig. 1, was designed.

The mathematical model [11] is the basis for calculating the heating kinetics of the granule and moisture removal from it. This model allows to obtain data on the granule heating along the radius and the relative granule mass (which is determined by the amount of the moisture removed from it) in a given time period.

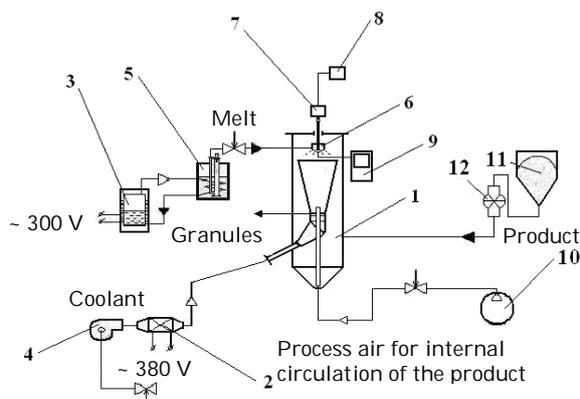


Fig. 1 – Experimental setup for studying the thermodynamic conditions for obtaining a nanostructured porous surface layer on the granules: 1 – vortex granulator; 2 – electric air heater; 3 – steam generator; 4 – gas blowers; 5 – capacity with recessed pump; 6 – atomizer; 7 – electromagnetic vibrator; 8 – electronic regulator; 9 – frequency meter; 10 – compressor; 11 – hopper; 12 – segment dispenser

Conventional notation:

- $T_c$  – coolant temperature, °C;
- $T_0$  – initial temperature of the granule, °C;
- $r$  – granule radius, m (mm);
- $d$  – granule diameter, m (mm);
- $U_{in}, U_{fin}$  – initial and final moisture content, respectively, moisture kg/material kg;
- $\tau$  – time, s.

Devices and equipment:

- thermo-anemometer TES-1340 for the determination of the hydrodynamic characteristics of flow motion;
- thermocouple TC10-C, recording potentiometer KSP-3 for measuring temperature in the air heater;
- thermal imager Fluke Ti25, pyrometer Victor 305B to measure temperature in the granulator working space;
- multimeter DT-838 for measuring moisture content of granules and air;
- microscope KONUSPIX-450X KONUS, scanning electron microscope SEM-100U, X-ray spectrometer with energy dispersion to study the granule microstructure.

3. RESULTS AND DISCUSSION

In Fig. 2 we present the typical calculation results of the change kinetics of the granule temperature along the diameter ( $d = 1-3$  mm) in a certain time period and the granule temperature in a given time period ( $\tau = 2-6$  s).

Granules of different diameters are heated with different intensities that gives grounds for determining the total heating time of the granules in a polydisperse system at the level of the maximum heating time for the granule of the greatest diameter. Despite the fact that, as shown by the results of experimental studies, the size distribution of wet granules should be blurred in the lower part of the vortex granulator, the uniform heating of the entire polydisperse system is possible only under the above time.

The calculations presented are valid for a constant-temperature coolant and do not take into consideration the influence of the granule temperature (for example, when introducing the product) and the liquid phase (wetting agent), which are introduced into the device, on the change of coolant temperature. Thus, the granule will be heated along with the coolant to a certain temperature, and only then an intense moisture removal will occur. This factor increases the necessary total residence time of the granules in the device. The calculations based on the mathematical model showed that it takes 8 seconds to completely heat the granule of  $d = 2$  mm from  $20$  °C to  $120$  °C in the coolant flow with a temperature up to  $120$  °C. If a stepwise calculation is performed taking into account the fact that the coolant is cooled when introducing the granules or wetting agent, then the gradual heating of the granule to a temperature of  $120$  °C will take 3-3.5 times longer than in the previous case. The results of experimental studies of the heating kinetics of the vortex granulator working space under various conditions, which will be presented in the second part of the given work, is the basis for determining the coolant temperature with gradual heating of the granule simultaneously with the coolant flow.

In Fig. 3 we illustrate the change kinetics of the relative mass of the ammonium nitrate granule of various diameters under different moistening conditions and requirements to the moisture content of the final product. It should be noted that up to 60 % of the desired moisture is removed in the region of intense vortex motion of the granules in the first drying period with a constant speed. The amount of residual moisture is successively removed mainly in the region of combined ascending and vortex motion of the granules. In the third zone – of the prior ascending motion – moisture is removed slightly that is confirmed by the results of experimental studies

of the effect of the gas flow rotationality on the relative intensity of moisture removal from the granule, which will be presented in the second part of the work.

Estimating the impact of the thermodynamic characteristics of the coolant on the porous surface layer structure, we should note the following:

1. The number of pores increases and the number of cracks and faults decreases in the porous layer structure with increasing granule diameter at an arbitrarily chosen coolant temperature. This is explained by the fact that heating of the granules of smaller diameter is more intense. In this case, there is a sharp release of moisture and ammonia from the granule core that is accompanied by the formation of cracks and faults because of a sudden change in the humidifier volume (its transition to the vapor state) and the incipient internal stresses. The surface layer structure of granules of different diameter is illustrated in Fig. 4-Fig. 6. The investigation results of the properties of granules showed a slight decrease in their strength with decreasing diameter.

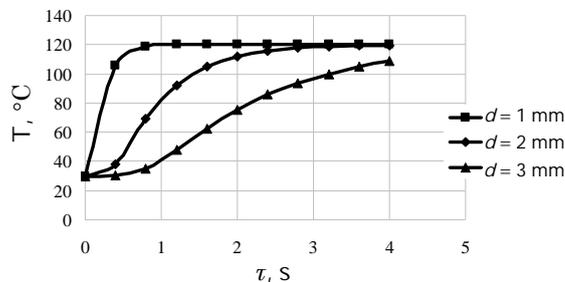


Fig. 2 – Heating kinetics of the ammonium nitrate granule along the radius at  $T_c = 120$  °C,  $T_0 = 30$  °C and  $\tau = 4$  s

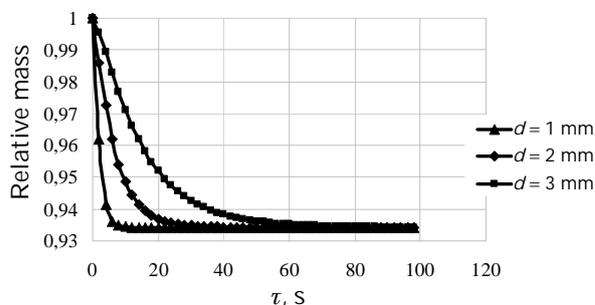


Fig. 3 – Change kinetics of the relative mass of the ammonium nitrate granule at  $T_c = 100$  °C,  $U_{in} = 0.01$  moisture kg/material kg,  $U_{in} = 0.003$  moisture kg/material kg

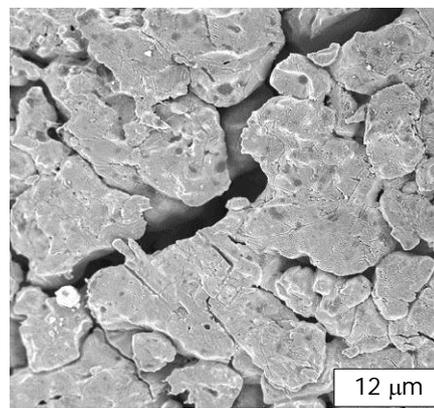


Fig. 4 – Surface layer on the granule of 1 mm diameter

2. The surface layer structure has a different form at an arbitrarily chosen granule diameter with increasing coolant temperature. At a temperature of 90-110 °C, the granule surface has a certain amount of shallow pores; a predominantly non-porous structure is observed (Fig. 7). In this case, the retention capacity of the granule with respect to diesel fuel is lower than the standard indicator at sufficient strength of the granule core.

At a temperature of 120-140 °C, cracks are predominantly formed on the granule surface due to the rapid evaporation of moisture (Fig. 8); these cracks increase the absorption capacity of the granule, but significantly reduce the strength of its core.

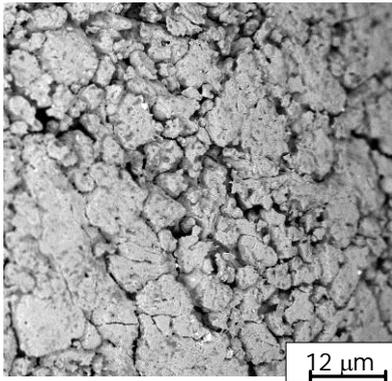


Fig. 5 – Surface layer on the granule of 2 mm diameter

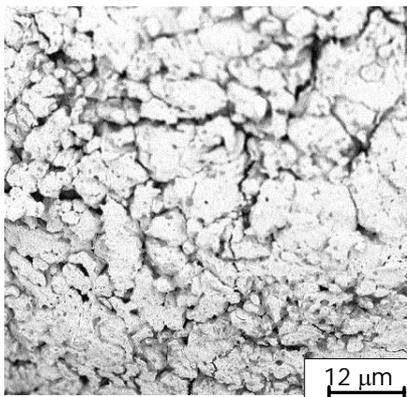


Fig. 6 – Surface layer on the granule of 3 mm diameter

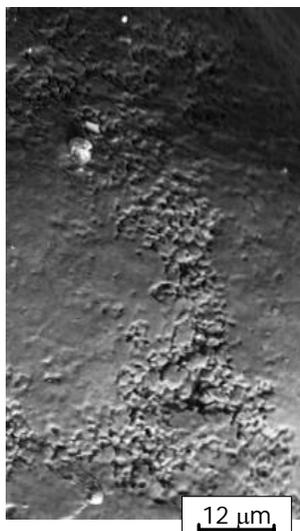


Fig. 7 – Sample dried at a temperature of 90-110 °C

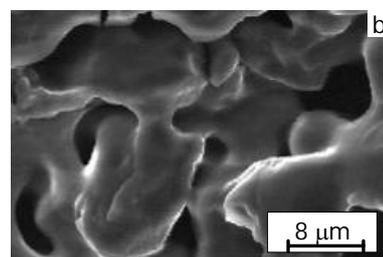
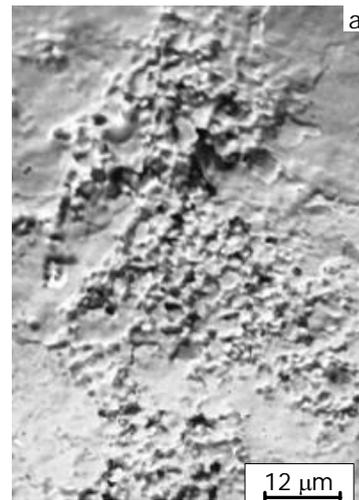


Fig. 8 – Sample dried at a temperature of 110-120 °C: a – surface; b – cleavage

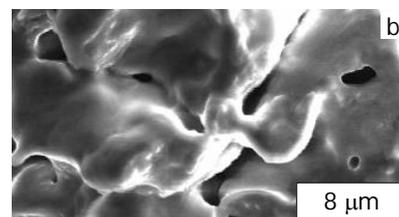
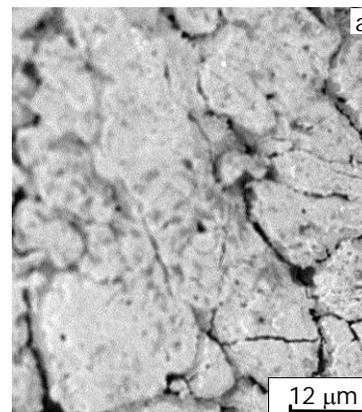


Fig. 9 – Sample dried at a temperature of 120-140 °C: a – surface; b – cleavage

At a temperature of 110-120 °C, a layer with a developed porous structure is formed on the granule surface; the pores are sufficiently deep, the cracks in the structure are almost absent (Fig. 9). The retention capacity of the granules in this case reaches standard indicators, when integrity and strength of the granule core are constant.

The research data are the basis for the optimization calculation of the technological process of producing a porous nanostructured layer in vortex-type granulators.

Of further interest is the study of the consolidation of the thermodynamic characteristics of the coolant and the hydrodynamic parameters of its motion (in particular, the swirling intensity) on the quality of a porous surface layer on the ammonium nitrate granule. The second part of the work will be devoted to the solution of this problem.

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