ТЕХНОЛОГІЯ ЛІКАРСЬКИХ ПРЕПАРАТІВ

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The study of the kinetics of drying natural zeolite granules in a tray dryer

Determination of the main drying parameters for choosing the optimal ones is obligatory when developing the drug technology, which uses wet granulation operations.

Aim. To study the drying process of natural zeolite granules obtained by the wet granulation in a tray dryer for substantiation of its optimal modes.

Materials and methods. The objects of the study were granules of natural zeolite with the size of 0.5-1.0 mm obtained by wet granulation using a NG-12 laboratory granulator made by Mariupol factory of technological equipment. As a binder 7 % starch paste added to the granulated mass in the amount of 25 % was used. The drying process was carried out in a tray dryer at the temperatures of 50, 60, 70, 80, 90 and 100 °C. Duration of the drying process was 5, 10, 15, 20, 25, 30, 40, 50, 60, 70, 80 and 90 min. Determination of the moisture content of granules was carried out by the weight loss when drying. The moisture content (gram of water/gram of a dry material) was determined taking into account the difference in mass before and after drying. The drying rate was determined by the amount of moisture removed from unit surface per unit time.

Results and discussion. The study of kinetics of drying natural zeolite granules has shown that the rate of the process depends on the moisture content of the material, the level and duration of the temperature effect on the experimental objects. The optimal drying time for natural zeolite granules in a tray dryer and similar apparatuses by design and functionality is 25-30 min, and the optimal temperature mode is 60-80 °C.

Conclusions. The kinetics and basic parameters of drying of natural zeolite granules obtained by the wet granulation method in a tray dryer have been studied. The optimal temperature and time modes of drying have been determined. The data obtained can be used when developing the technology of granules and tablets with natural zeolite.

***Key words*:** natural zeolite; granules; drying; tray dryer

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Вивчення кінетики сушки гранул цеоліту природного в сушарці поличній

Визначення основних параметрів сушки з метою вибору найбільш оптимальних є обов'язковим при розробці препарату, технологія якого передбачає проведення операцій вологого гранулювання.

Мета. Вивчення процесу сушіння гранул цеоліту природного, отриманих шляхом вологого гранулювання в сушарці поличній, для обґрунтування оптимальних режимів сушки.

Матеріали та методи. Об'єктом дослідження були гранули цеоліту природного розміром 0,5-1,0 мм, отримані шляхом вологого гранулювання з використанням лабораторного гранулятора НГ-12. Зволожувач – 7 % крохмальний клейстер, доданий в кількості 25 %. Температура сушки – 50, 60, 70, 80, 90 та 100 °С. Тривалість процесу сушіння: 5, 10, 15, 20, 25, 30, 40, 50, 60, 70, 80, 90 хв. Визначення вологовмісту гранул проводили за втратою маси при висушуванні. Вміст вологи (грам води/грам сухої речовини) визначали за різницею маси до та після висушування. Швидкість сушки визначали за кількістю видаленої вологи з одиниці поверхні за одиницю часу.

Результати та їх обговорення. Дослідження кінетики сушки гранул цеоліту природного показало, що швидкість процесу залежить від вологості матеріалу, рівня та тривалості впливу температури на експериментальні об'єкти. Оптимальна тривалість сушки гранул цеоліту природного в сушарці поличній та у подібних до неї за конструкцією та способом функціонування апаратах становить 25-30 хвилин, а оптимальна температура – 60-80 °С.

Висновки. Досліджені кінетика та основні параметри сушки гранул цеоліту природного, отриманих методом вологого гранулювання в сушарці поличній. Визначені оптимальні температурні та часові режими сушки. Отримані дані можуть бути враховані при розробці технології гранул і таблеток з цеолітом природним.

***Ключові слова*:** *цеоліт природний; гранули; сушка; сушарка полична*

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Изучение кинетики сушки гранул цеолита природного в сушилке полочного типа

Определение основных параметров сушки с целью выбора наиболее оптимальных является обязательным при разработке технологии препарата, которая предусматривает проведение операций влажного гранулирования.

Цель. Изучение процесса сушки гранул цеолита природного, полученных путем влажного гранулирования в сушилке полочного типа, для обоснования оптимальных режимов сушки.

Материалы и методы. Объектами исследования служили гранулы цеолита природного размером 0,5-1,0 мм, полученные путем влажного гранулирования с использованием лабораторного гранулятора НГ-12. Увлажнитель – 7 % крахмальный клейстер, добавленный в массу в количестве 25 %. Температура сушки 50, 60, 70, 80, 90 и 100 °С. Продолжительность процесса сушки – 5, 10, 15, 20, 25, 30, 40, 50, 60, 70, 80 и 90 мин. Определение влагосодержания гранул проводили по потере массы при высушивании. Содержание влаги (грамм воды/грамм сухого вещества) определяли с учетом разницы массы до и после высушивания. Скорость сушки определяли по количеству удаленной влаги с единицы поверхности в единицу времени.

Результаты и их обсуждение. Исследование кинетики сушки гранул цеолита природного показало, что скорость процесса зависит от влажности материала, уровня и продолжительности воздействия температуры на экспериментальные объекты. Оптимальная продолжительность сушки гранул цеолита природного в сушилке полочного типа и в подобных ей по конструкции и способу функционирования аппаратах составляет 25-30 минут, а оптимальный температурный режим– 60-80 °С.

Выводы. Исследована кинетика и основные параметры сушки гранул цеолита природного, полученных методом влажного гранулирования, в сушилке полочного типа. Определены оптимальные температурные и временные режимы сушки. Полученные данные могут быть использованы при разработке технологии гранул и таблеток с цеолитом природным.

Ключевые слова*: *цеолит природный; гранулы; сушка; сушилка полочная

Drying is the process of mass transferring, and it is in removing moisture (water or other solvent) by evaporation from a solid, semi-solid or liquid matter. In the broadest sense, the moisture contained in the solid material can be classified as free and bound moisture. The moisture retained by the material only as a result of a mechanical bond is called free. The superficial moisture and the moisture filling pores and large capillaries in the material when it is wetted are bound mechanically with the solid material. This moisture is easily removed from the material and is usually called external moisture. The bound moisture is the moisture retained by the material by any additional (except for a mechanical bond) forces, such as physical adsorption, chemical interaction, forces of capillary or hygroscopic coupling, etc. When drying this moisture should be torn off from the material, consuming extra energy. Physically and chemically, solids are bound with the moisture absorbed by the surface of small capillaries (adsorption moisture) or penetrated into cells of an organic material due to diffusion (structural and osmotic moisture). Chemically, solid materials are bound with the hydrated moisture (crystallized moisture), which during the drying process is usually not removed. Unlike the external moisture, the moisture firmly bound with the material is called hygroscopic [1].

During the drying process of granules at first free moisture evaporates due to the heat transfer from the environment. Then the bound moisture moves to the surface of the solid and then evaporates from the surface of the material. The removal of the surface moisture depends on several conditions, such as temperature, relative humidity, the flow rate of the air, the contact area and pressure. The transfer of the bound moisture is a function of temperature, humidity and primarily the physical nature of solids [2].

In the process of wet granulation certain bonds providing plasticity of the material and allowing the change in the shape of the granules without their destruction form between the particles. To obtain the finished product it is necessary to strengthen these bonds, giving rigidity to the structure obtained during the granulation process. This is achieved by removing the liquid phase or transforming it into the solid one in the process of drying granules, which leads to the intense crystallization of solid components inside the granule. The final moisture content of the product largely determines its technological properties (hardness, compressibility, hygroscopicity, etc.) [3-4].

The process of removing the liquid phase proceeds at a certain rate, which depends on the form of the moisture bond with the material. Furthermore, duration of the drying process is determined by the structure of the material, the size of the initial and the final moisture of the material, as well as its temperature. For granules that are further tableted, transported and stored the drying process is very important [4-7]. Determination of its main parameters for choosing the optimal ones is obligatory when developing the drug technology, which uses wet granulation operations.

The aim of the work was to study the drying process of natural zeolite granules obtained by the wet granulation in a tray dryer for substantiation of its optimal modes.

Materials and methods

The objects of the study were granules of natural zeolite with the size of 0.5-1.0 mm obtained by wet granulation using a NG-12 laboratory granulator made by Mariupol factory of technological equipment. As a binder 7 % starch paste added to the granulated mass in the amount of 25 % was used. The drying process was carried out in a tray dryer at the temperatures of 50, 60, 70,

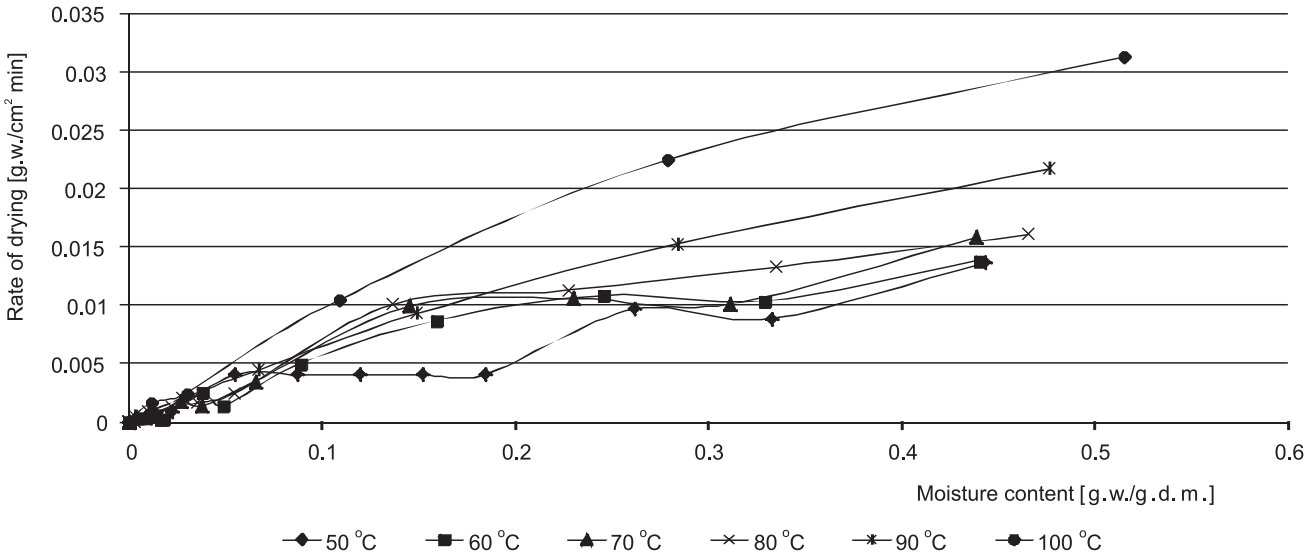

Fig. 1. The dependence of the drying rate on the moisture content of the material and the temperature

80, 90 and 100 °C. The method of the experiment was as follows: first, granules were made by the standard technology of wet granulation, weighed and put in trays, then the trays were placed into a drying chamber previously heated up to the experimental temperature. Duration of the drying process was 5, 10, 15, 20, 25, 30, 40, 50, 60, 70, 80 and 90 min. After each time period some granules were sampled, and their moisture content was determined. In each experiment 5 parallel measurements were performed, among them the average value was found.

Determination of the moisture content of granules was carried out by the weight loss when drying using the method of the State Pharmacopoeia of Ukraine (SPhU), paragraph 2.2.32 [8]. The sample of approximately 2 g was placed in a dry weighing bottle, weighed, dried in a drying chamber at 105 °C to constant weight, and weighed again. The moisture content (gram of water/gram of a dry material) was determined taking into account the difference in mass before and after drying.

The rate of drying "R" was calculated by the formula:

$$R = -\frac{S}{A} \cdot \frac{dX}{dt},$$

where: R – is the drying rate, g.w./cm^2; S – is the weight of a dry solid, g; A – is the exposed surface area for drying, cm^2; X – is the solid moisture content, g.w./g.d.m.; t = time, min.

Results and discussion

The study of the kinetics of drying natural zeolite granules showed that the rate of the process depended on the moisture content of the material, the level and duration of the temperature effect on the experimental objects (Fig. 1-2). According to the experimental data the most intense evaporation of moisture occurred within the moisture content of the material of 0.5-0.3 g.w/g.d.m. for the first 20 min of the experiment. It should be also noted that the rate of evaporation was different for almost all samples from the first minute of the experiment.

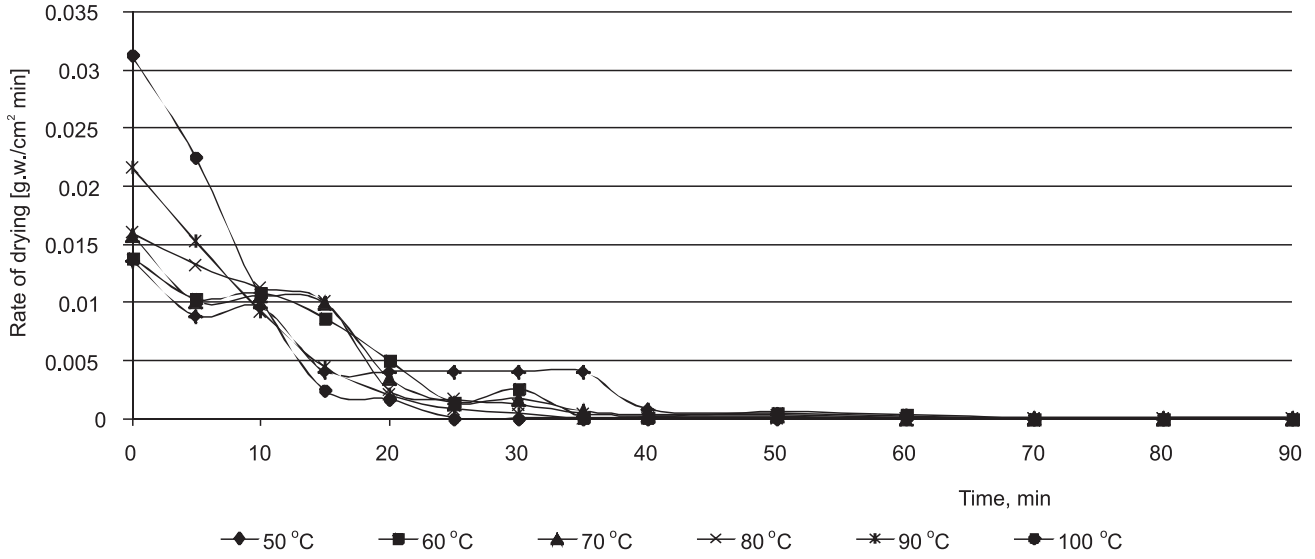

Fig. 2. Dynamics of change of the drying rate in time

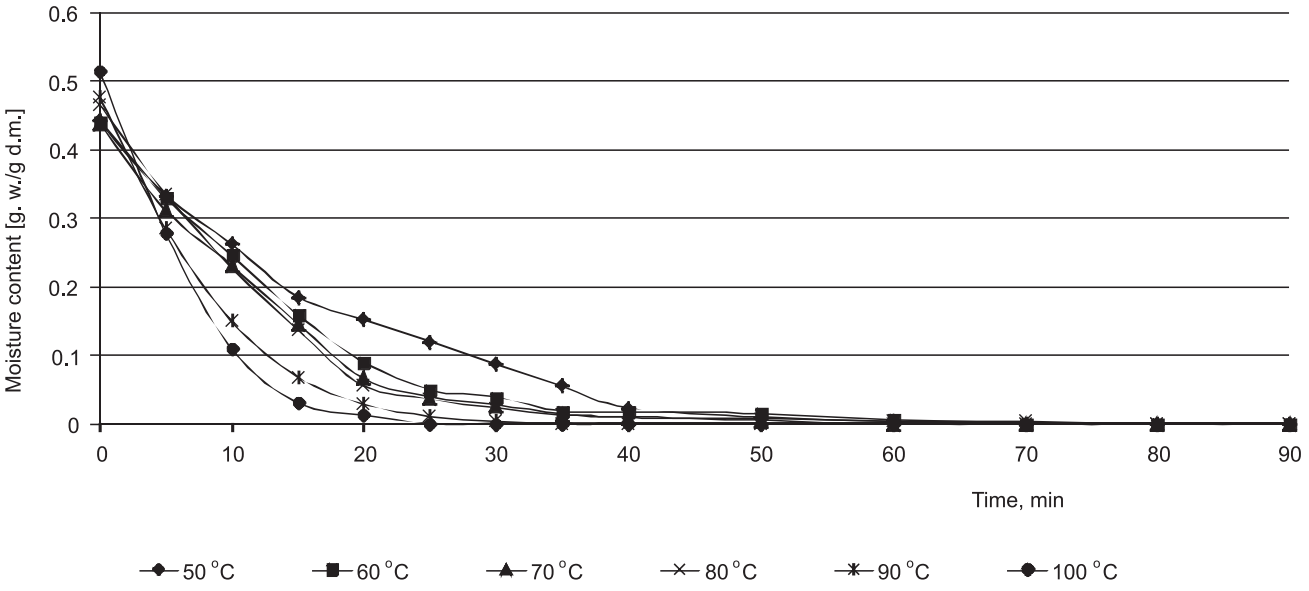

Fig. 3. The dependence of the moisture content of granules on the time and temperature of drying

With the exception of heating modes of 90 °C and 100 °C, the curves of dependence of the rate changes on the moisture content were characterized by several periods of the rate variation: decrease (the moisture content of 0.48-0.3 g.w/g.d.m. and 0.15-0.05 g.w/g.d.m.) and the relative stability (the moisture content of 0.3-0.15 g.w/g.d.m. and 0.05-0.03 g.w/g.d.m.). It corresponds to evaporation of moisture from the surface of the material and from its deep layers, respectively. The maximum drying rate was observed for the heating modes of 90 °C and 100 °C, the minimum rate was when heating at 50 °C and 60 °C.

Analyzing the dependence of the change in the moisture content of the material on the drying time and temperature (Fig. 3) it is possible to distinguish two main periods of the moisture evaporation. The first period of 20-25 min is characterized by the most rapid and significant decrease in the moisture content from 0.5 to 0.15 g.w/g.d.m.; during it mostly free moisture, which is on the surface and in the upper layers of the material, is removed. After reaching the critical moisture content (approximately 0.15-0.1 g.w/g.d.m.) the second period begins; within it the moisture is removed from the inside of the material. For the first conditional period the dependence of the average rate on temperature is typical. The drying rate during the second period with the temperature change is almost unchanged (Fig. 4).

Taking into account the data of the optimal moisture content (0.03-0.05 g.w./g.d.m.) for granules and tablets containing natural zeolite given in our previous work [9] it should be concluded that the optimal drying time for natural zeolite granules in a tray dryer and similar apparatuses by design and functionality is 25-30 min. In addition, taking into account the properties of active substances and excipients, the optimal temperature mode is 60-80 °C. Decrease in the heating temperature of the drying chamber results in an unjustified increase in the drying time, and an increase above 80 °C leads to destruction of the excipients of the composition, in particular potato starch.

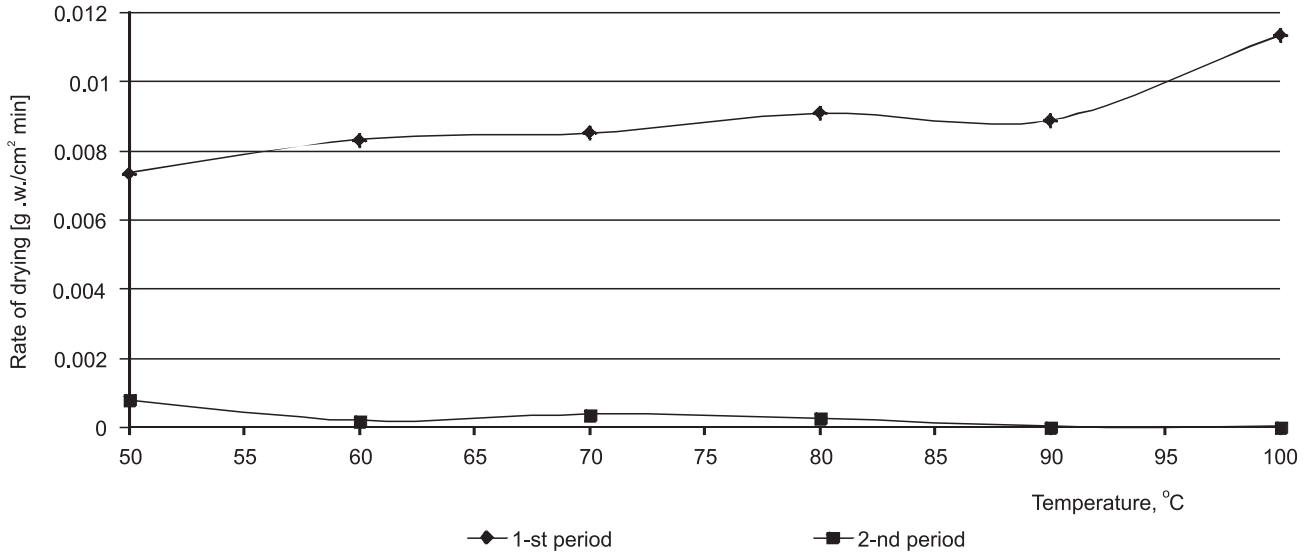

Fig. 4. The average drying rate curves

CONCLUSIONS

The kinetics and basic parameters of drying of natural zeolite granules obtained by the wet granulation method in a tray dryer have been studied. The effect of the moisture content of the material, the temperature range and duration of the process on the rate of the moisture removal has been studied. Based on the data obtained and our previous studies the optimal level of the moisture content for natural zeolite granules has been determined as 0.03-0.05 g.w./g.d.m. Based on the analysis of the data obtained duration of the drying process of natural zeolite granules in a tray dryer and similar apparatuses by design and functionality has been substantiated; it is 25-30 min at the temperature mode of 60-80 °C. The data obtained can be used when developing the technology of granules and tablets with natural zeolite.

Conflict of Interests: authors have no conflict of interests to declare.

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