Intensification and modeling of vacuum freeze drying of materials of various structures (using the example of biopolymer matrices and suspensions)

Abstract

Relevance of the research. In accordance with the Decree of the Government of the Russian Federation dated October 22, 2021 No. 1814 on the approval of the state program “Scientific and Technological Development of the Russian Federation”, as well as in accordance with the forecast of the Higher School of Economics on the scientific and technological development of Russia until 2030, the development of technologies for producing new biodegradable, composite and dispersed polymer materials is a current direction in the development of medicine and related fields of science. These types of materials include matrices and dry particles as carriers of active pharmaceutical ingredients (APIs). Matrices are highly porous materials, usually consisting of biopolymers and having a number of properties that determine their use in medicine. Due to their developed surface, matrices are widely used as substrates for cell culture, which is especially important in the field of regenerative medicine when developing materials for the restoration of damaged tissues and organs. Due to the properties of biocompatibility and biodegradation, matrices are widely used as local dressing materials. In turn, dry particles, as API carriers, before drying can be obtained in the form of a suspension - a dispersed system in which solid particles are initially dispersed in a continuous liquid phase. Dried micropowders are widely used as delivery vehicles for drugs in the treatment of socially significant diseases: tuberculosis, asthma and others. The listed types of materials are obtained by the method of vacuum freeze drying (VFD), which allows preserving the original properties of the dried objects, without losing their structural integrity and biological activity. However, the VFD method is energy- and resource-intensive, because occurs at low temperatures. In accordance with the above-mentioned decree of the government of the Russian Federation, in 2019 the volume of internal expenditures on research and development on scientific topics “Energy Efficiency” and “Energy Saving” amounted to 110.37 billion rubles. Due to high energy costs, research and intensification of the VFD process is a relevant and important direction in order to improve energy efficiency and increase productivity in the field of obtaining biopolymer materials.

The dissertation work is devoted to experimental studies on the production of materials of various structures and the study of their characteristics, as well as theoretical

and experimental studies of the intensification of VFD at each stage of the process: from pre-freezing to drying.

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The purpose of the work – intensification and mathematical modeling of the VFD process, taking into account the structural features of materials.

Tasks:

1. Experimental studies of processes for obtaining materials of various structures.

2. Intensification of the VFD process at various stages: development of an installation for carrying out the freezing process with ultrasonic influence; study of the effect of pressure on the kinetics of VFD; modernization of the design of the VFD installation; development of a VFD process control system.

3. Mathematical modeling of VFD stages taking into account the structural features of materials.

4. Analysis of energy and economic efficiency of the WSS process.

Scientific novelty. Types of materials are identified depending on the physical structure and technology of their production. The influence of ultrasonic vibrations applied at the pre-freezing stage on the morphology of ice crystals and the subsequent structure, and the morphology of materials after VFD, was studied.

Methods for intensifying VFD at various stages of the process have been studied: the influence of ultrasound on the kinetics of freezing and subsequent VFD has been studied; The influence of pressure gradients, as well as infrared radiation and ultrasonic influence on the kinetics of VFD was studied.

A mathematical model has been developed to describe the kinetics of freezing of polymer materials, including taking into account ultrasonic influence. In the model, using optimization methods, the coefficients of the empirical equation describing the size distribution of ice crystals are selected.

An approach is proposed to take into account the structural features of the material when calculating the rate of heat and mass transfer in the VFD process, which consists of different arrangements of material components and the distribution of physicochemical properties in the computational domain. A mathematical model has been developed for calculating the VFD of materials of various structures, taking into account the uneven distribution of water vapor throughout the volume of the working chamber. The influence

of water vapor pressure on the drying kinetics when organizing the process in laboratory and industrial installations was studied. A mathematical model of the kinetics of VFD materials of various structures has been developed, taking into account ultrasonic and infrared exposure.

Practical significance. A large amount of experimental and analytical data has been obtained for polymer materials of various structures. Methods for obtaining materials have been tested, in the amount of 17 different samples, which can be used as matrices or substrates for cell cultivation, as well as drug delivery systems.

A design of an installation for carrying out the pre-freezing process with ultrasonic influence has been proposed. A design of an installation for carrying out VFD with simultaneous infrared and ultrasonic exposure in the volume of the working chamber has been proposed in order to intensify the process, which was carried out for the first time for VFD of polymer matrices and suspensions.

An automated control system for the VFD process has been developed with simultaneous infrared and ultrasonic exposure, as well as a software module for determining the moisture content and temperature of the material during the drying process. The software module was used to carry out experimental work to study the kinetics of VFD.

A large amount of data on the kinetics of freezing and VFD of materials of various structures was analyzed (81 experiments). Recommendations are given for conducting the VFD process with ultrasonic and infrared exposure.

A number of computer programs have been developed and registered to calculate the kinetics of freezing and VFD. The software can be used to select process modes.

An analysis of the economic efficiency of the VFD process was carried out. A reduction in energy costs has been confirmed when organizing a process with infrared radiation and ultrasonic influence in laboratory and industrial scale installations.

The main provisions for the defense.

– Results of experimental studies of processes for obtaining materials of various structures. Results of a study of freezing kinetics. Results of analytical studies of the obtained materials.

– Method and device for carrying out the freezing process with ultrasonic influence. Results of a study of the influence of ultrasonic vibrations on the kinetics of freezing and the morphology of formed ice crystals in various polymer solutions.

– A device for intensifying the VFD process with simultaneous regulation of infrared heating and a replaceable ultrasound source in the volume of the working chamber. Process control system and software module for studying drying kinetics.

– Results of the study of the intensification of the VFD process: study of the influence of water vapor pressure on the kinetics of the drying process; study of the influence of ultrasound applied at the pre-freezing stage on the kinetics of the drying process; study of the influence of infrared radiation and ultrasound on the kinetics of the drying process.

– Mathematical model for calculating the freezing kinetics and size distribution of ice crystals for materials of various natures.

– Study of the influence of water vapor pressure in the volume of the working chamber on the kinetics of drying agent, when organizing the process in laboratory and industrial installations, using an iterative approach based on the transfer of calculated data between a program for calculating drying kinetics, written in the Python programming language, and the Ansys Fluent software package for calculating gas dynamics and distribution of water vapor in the volume of the working chamber.

– Mathematical model for calculating the kinetics of VFD with infrared and ultrasonic influence.

– Study of the influence of VFD modes on economic and energy efficiency when organizing the process in laboratory and industrial installations.