ENERGY AND RESOURCE SAVING DURING SUPERCRITICAL DRYING

ABSTRACT

The relevance of the work. In accordance with the presidential decree «On national goals and strategic objectives for the development of the Russian Federation for the period up to 2024», one of the most important tasks for GDP is the growth in production of high-tech and science-intensive industries. Aerogel is a highly porous solid material with a developed specific surface area and low density. Aerogels are used in various fields, it can be used as materials for sound and thermal insulation, energy storage, sensitive materials in gas sensors, gas sorbents, sorbents for oil spill response. Aerogel is obtained in some countries on an industrial scale as a highly effective thermal insulation material. It should be noted that a similar production exists on the territory of the Russian Federation. Scientists from D. Mendeleev University were engaged in the technology development for the production of heat-insulating material based on aerogel and transferred the technology to Niagara LLC. To obtain aerogels, it is necessary to carry out a supercritical drying process. This process is technologically complex. One of the most important tasks is the intensification of the supercritical drying process to reduce both capital and operating costs aerogels production.

This work is devoted to theoretical and experimental studies of energy and resource saving, intensification of the process of supercritical drying of aerogels. To intensify the process, various operating-technological and instrumental-constructive methods are used. Such methods can be used to solve problems of both the optimization of existing production facilities and the design of new enterprises.

The work was carried out with the financial support of the Ministry of Science and Higher Education of the Russian Federation within the framework of the state assignment, agreement No. 075-03-2020-232/3 (FSSM-2020-0003).

The purpose of the work is theoretical and experimental studies of energy and resource saving, intensification of the process of supercritical drying of aerogels. To achieve the purpose it was necessary to solve the following **tasks:**

1. Investigation of the preparation of alumina aerogel and their characteristics.
2. Intensification of the supercritical drying process on equipment with a volume of 22 ml.
3. Intensification of the supercritical drying process on equipment with a volume of 250 ml.
4. Mathematical modeling the supercritical drying kinetics.
5. Mathematical and CFD modeling of ultrasound in supercritical fluid.
6. Development of a method and software for calculating the economic efficiency of supercritical drying.

Scientific novelty. The three-component system «epichlorohydrin – ethanol – water», which is formed during the preparation of alumina gels, at a temperature of 298 K and atmospheric pressure has been investigated. The process of gelation is investigated and the mechanisms of structure formation of alumina aerogels are proposed. The study of the structural characteristics of the obtained aerogels has been carried out. Regularities that affect the properties of aluminum gels and aerogels have been established.

The following methods of intensification of the supercritical drying process have been investigated: optimization of operating and technological parameters (carbon dioxide consumption, temperature, pressure), pulse change of process parameters (pressure), superposition of fields (ultrasonic vibrations), intensification in accordance with phase diagrams. The intensification influence methods on the following stages of the supercritical drying process is analyzed: pressurization of the apparatus, exchange of solvent from the apparatus volume, diffusion solvent exchange, which made it possible to give recommendations on intensification of the supercritical drying process.

A mathematical model for describing the kinetics of the supercritical drying process has been developed. The model considers mass transfer inside the gel, in the boundary layer of the gel and in the free volume of the apparatus. The mathematical description is applicable to various types of gels in the form of cylinders, spheres and plane-parallel bodies.

A mathematical model to describe hydrodynamics, heat and mass transfer processes in supercritical fluids under ultrasonic action has been developed. It allows obtaining velocity diagrams, concentration distributions at each point of the apparatus.

A method for calculating the economic efficiency of the supercritical drying process, which includes a mathematical model for describing the kinetics of the supercritical drying process has been developed.

Practical significance. Experimental studies on the production of alumina aerogels have been carried out. The structural characteristics of aerogels can be changed depending on the synthesis parameters, which makes it possible to use this material in various applications.

Experiments were carried out to intensify supercritical drying on equipment with a volume of 22 and 250 ml. The results obtained can be used for supercritical drying equipment of various scales.

Equipment for the supercritical drying process under ultrasonic action, which was carried out for the first time for drying aerogels has been developed.

A software to describe the kinetics of the supercritical drying process has been developed. This software can be used to investigate the effect of gel characteristics and process parameters on supercritical drying.

A software to assess the economic efficiency of the process has been developed. This sofware was used to optimize the supercritical drying process on a 70-liter pilot plant.

The main provisions for the defense. Research results of the processes of obtaining alumina aerogels using the sol-gel technique. Results of the ternary system «epichlorohydrin – ethanol – water» phase diagram experimental studies, which is formed during gelation at a temperature of 298 K and atmospheric pressure. Influence of the preparation parameters on the gelation process and the structural characteristics of aerogels.

Equipment with a volume of 22 ml designed for the supercritical drying process under ultrasonic action.

Intensification of the supercritical drying process on equipment with a volume of 22 and 250 ml using the following methods: optimization of operating and technological parameters (carbon dioxide consumption, temperature, pressure), pulse change of process parameters (pressure), superposition of fields (ultrasonic vibrations), intensification in accordance with phase diagrams.

A mathematical model and a software developed in Python (Jupyter Notebooks) describing the kinetics of the supercritical drying process.

The results of the influence of ultrasonic vibrations on the velocity distribution of supercritical carbon dioxide and on the mass transfer in the «carbon dioxide – isopropanol» mixture in a 22 ml high-pressure apparatus using Ansys Fluent.

Development of a method and a software for calculating the assessment of the economic efficiency of the supercritical drying process. Optimization of the supercritical drying process on equipment with a volume of 70 liters.