

Intensification of supercritical drying process

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The main and most technologically complicated stage of aerogels production is supercritical drying. A feature of such drying is the use of a substance in a supercritical state, as drying agent. Which is miscible with an organic solvent contained within the dried material at appropriate process parameters. This feature allows to get highly porous materials without significant shrinkage maintaining of their structural characteristics. The process of supercritical drying is batch process and drying of monoliths with large geometric dimensions takes a considerable time. Therefore, an important task is the intensification of mass transfer processes that occur during supercritical drying in order to reduce its time. It is important to note that the supercritical drying process can be divided into a number of main steps: (1) pressurization, (2) solvent displacement from the free volume of the apparatus, (3) diffusional exchange of the solvent in the medium of the porous body. During the first step, due to a significant change in the mutual solubility of the organic solvent and carbon dioxide, a spillage phenomenon occurs when a significant part of the organic solvent leaves the dried porous material. At the second step, due to the organization of a carbon dioxide continuous flow through the apparatus, the solvent is displaced mainly from the free volume of the apparatus. Within the framework of this step, the intensity of drying is determined by the intensity of convective transport, that is, the higher the flowrate of carbon dioxide and the more efficiently the geometry of the apparatus for drying is organized, the less time is required to carry out this step. The third step of the process is the longest one, and only diffusional transport occurs. The intensification of this particular step is the most interesting, since it will significantly reduce the total time of the supercritical drying process.

In the course of research, it is proposed to use two approaches for supercritical drying process intensification: (I) a pulsed change in the external parameters and the drying agent flowrate; (II) application of high-frequency ultrasonic vibrations. The application of the first approach is based on the formation of density, speed, composition gradients within the system inside the apparatus for supercritical drying. Due to this, a more active hydrodynamic situation can be organized, the intensity of flow separation at the interface “porous body - free volume of the apparatus” can be increased. In the course of the work, the effect of pulsed changes in pressure and carbon dioxide flowrate was studied. The second approach is to organize high-frequency oscillations in the working volume of the apparatus for supercritical drying. Such vibrations make it possible to increase the intensity of mass transfer, including in the volume of the dried porous body. An experimental setup of own design was built to implement this approach. It was shown that the superposition of the field of high-frequency ultrasonic vibrations at a frequency of 35 kHz and a power of 100 W in an apparatus with a volume of 22 ml, the duration of the supercritical drying process can be significantly reduced.

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