EFFECT OF VACUUM DRYING ON MICROSTRUCTURE OF SEMI-SOLID CHEESE

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Abstract: Both theoretical and applied studies on microstructure of cheese immediately after a technological cycle and dry cheese obtained by vacuum drying are described. The aim of the microstructure studies is a more comprehensive evaluation of the product quality. Images of cheese upon high- and low-temperature secondary heating at different magnification were studied. The effect of drying on cheese microstructure was investigated. Analysis and identification of various components in the cheese mass using microstructure studies were performed. Calcium phosphate depositions were detected with electron microscopy. Calcium lactate was detected in mature cheese.

Keywords: microstructure, vacuum drying, cheese, microvoids, macrograins, residual pressure, temperature, shrinkage, pores

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INTRODUCTION

Vacuum drying, performed at residual pressure above the water triple point, is one of the most advanced methods of food products dehydration. In Russia, it has been widely used in chemical, pharmaceutical, medical, food, and other industries, that is, in the areas where materials are especially sensitive to the effects of high temperatures [1–4].

Vacuum dryers allow for a product of high purity and quality. In function of the material properties and requirements to the final product, time of drying and temperature modes vary. Vacuum drying proceeds in two stages. During the first stage, drying rate is constant and the material temperature is close to the temperature of water saturation at given pressure. During the second stage, the rate of drying decreases and the material temperature increases approaching the temperature of the heat transfer medium. Intensity of heat transfer in the second stage decreases sharply. Increase in the rate of water evaporation in vacuum dryer may be achieved by increase in the temperature of the heat transfer medium or decrease of pressure [2, 5].

Considering cheese as a subject of vacuum drying, it should be noted that changes in cheese properties in the process of drying depend on both physicochemical properties, structure, and forms of moisture deposition in the material on one hand and thermophysical characteristics accounting for mass and energy transfer features on the other. Major structural elements of cheese are macrograins, layers between them, microvoids, and micrograins [6, 7]. Protein network forms the basis of each macrograin, while micrograins are embedded into its cells as lipid or lipoid drops or depositions of crystals [8, 9].

All structural components of cheese undergo deep changes in the course of maturation, producing texture and pattern typical of the cheese variety [5, 10–12].

Knowledge on changes in capillary structure of cheese mass in the course of ageing and further storage is required for correct design of the drying process.

In connection with the entry of large volumes and assortment of domestic and imported dairy products, including the concentrated ones, in the market, thorough and comprehensive control of the source composition and how it meets the requirements of the current standards is needed. Today, the main document regulating the dairy products, including cheese, is the Technical regulations of milk and dairy products, according to which evaluation of quality and identification of milk processing products proceed basing on the major physicochemical, organoleptic, and microbiological parameters. For the most thorough control of the dairy products source materials, different methods are used.

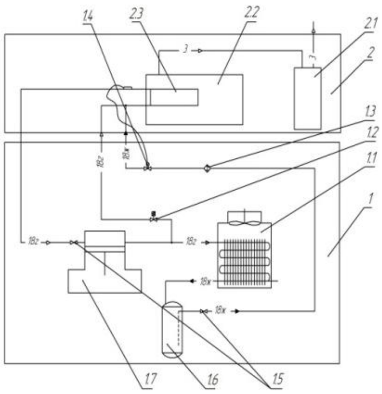
Internationally, histology methods of composition identification are used to control quality and exclude the possibility of adulteration of dairy products [13–16].

Today, one of the modern methods is scanning electron microscopy, allowing for investigation of structure of aged cheese prior to realization and dry cheese, upon vacuum drying [17, 18].

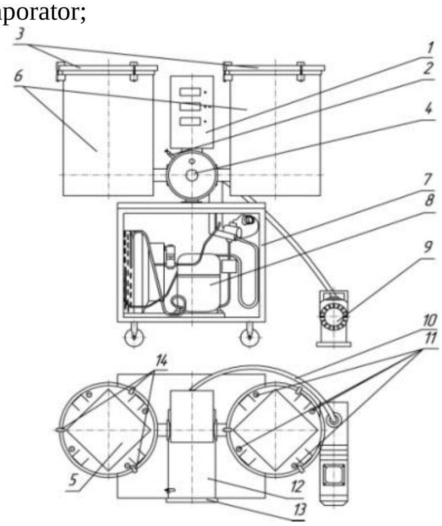
MATERIALS AND METHODS

The semi-solid rennet cheese varieties Sovetskii, Gollandskii, and Ozernyi were the subjects of the study. Vacuum drying of cheese was performed on an INEI-6M freeze-dryer (Laboratory of methods and instruments for biochemical analysis, Institute for Biological Instrumentation, Russian Academy of Sciences) (Fig. 1).

(a), outside appearance;



(b), principal scheme of the freeze-dryer:
1, freezer; 1.1, condenser; 1.2, solenoid valve; 1.3, drying filter; 1.4, thermostatic expansion valve; 1.5, cut-off valve; 1.6, receiver; 1.7, compressor; 2, vacuum unit; 2.1, vacuum pump; 2.2, desublimator; 2.3, evaporator;



(c), sublimation unit design:
1, control panel; 2, pressure release valve; 3, drying chamber lid; 4, evaporator; 5, product shelf; 6, drying chamber; 7, refrigerator unit bucket; 8, refrigerator unit; 9, vacuum pump; 10, vacuum pump flexible tubing; 11, incandescent lamp; 12, desublimator; 13, desublimator lid; 14, fixator of the drying chamber lid.

Fig. 1. «INEI-6M» freeze-dryer.

The refrigerator unit of the sublimation dryer is designed to remove moisture evaporating from the product in the process of drying through condensation on the surface of evaoprator, since the temperature of the latter one is much lower than the dew point temperature (–(35–45)°C). Because of this, partial pressure over the surface of evaporator is lower than the partial pressure of water vapor in the drying chamber under conditions of residual pressure of 10–100 Pa in the system.

The process of drying consists of the following stages. The product is placed on the shelfs 5, which are put into the drying chambers 6. The chambers are closed with the lids 3. The refrigerator unit 8 is turned on using the control panel 1; then, the instrument takes approximately 10–15 min to reach the freezing out mode. The freezing out mode is detected by the evaporator temperature 4 (the temperature should not be higher than –35°C), then the vacuum pump 9 is turned on, and the drying mode starts. Due to the low pressure in the chambers, the product is freezed and sublimation starts. Then, incandescent lamp 11 is turned on and the product is heated to remove residual water [19, 20].

Microstructure studies and elaboration of the electron microscopy techinque application to cheese samples were performed on a JEOL JSM-6390 LA (JEOL, Japan) scanning electron microscope.

RESULTS AND DISCUSSION

Quality of the material being dried depends on the preparation of the material, rate, and evenness of drying. For maximum retaining of all the initial properties of the product, the material should be of high quality, since high-quality products are more appropriate by their physicochemical composition, and their structure is undamaged; therefore they would be more fully-featured when dried. In this connection, only the high-quality cheese was used for drying. Figure 2 presents cross section of freeze-dryed cheese.

Upon vacuum drying, cheese dimensions practically did not change and it became porous. Prior to drying, linear dimensions of Sovetskii and Gollandskii cheese samples were 117 and 120 mm, and after drying, 114 and 115 mm, respectively. Diameter of the Ozernyi cheese prior to drying was 130 mm, and after drying, 124 mm. Linear shrinkage of cheese in the process of vacuum drying occurs only by 3–5%. Mass fraction of water in cheese samples did not exceed 5%. Vacuum drying was performed at 2–3 kPa.

Figures 3–8 present images of microstructure of Sovetskii, Gollandskii, and Ozernyi cheese samples before and after the vacuum drying. For convenience of microstructure analysis and comparison of capillary structure of the cheese mass, the same magnification, that is, 50, 200, and 2000-fold magnification, was used for all samples.

(a)

(b)

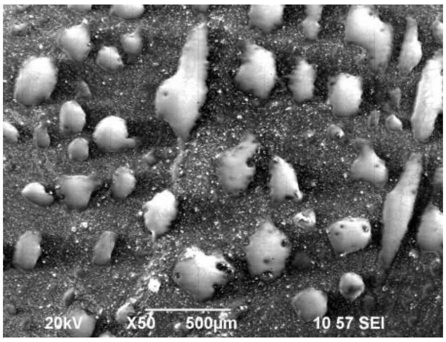
(c)

(d)

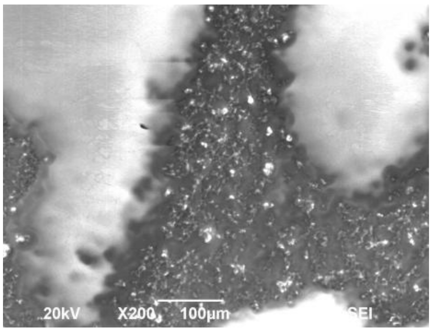
(e)

(f)

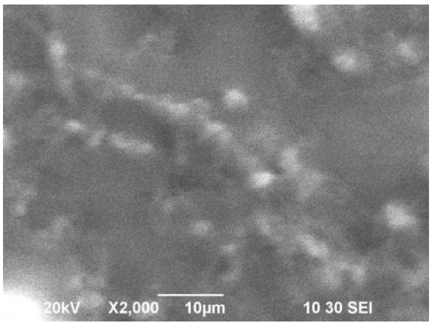
Fig. 2. *End.* Cross sections of cheese of different varieties before (a, c, and e) and after (b, d, and f) freeze-drying: (a, b) cheese variety Sovetskii; (c,d) Gollandskii; and (e, f) Ozernyi.



(a)



(b)



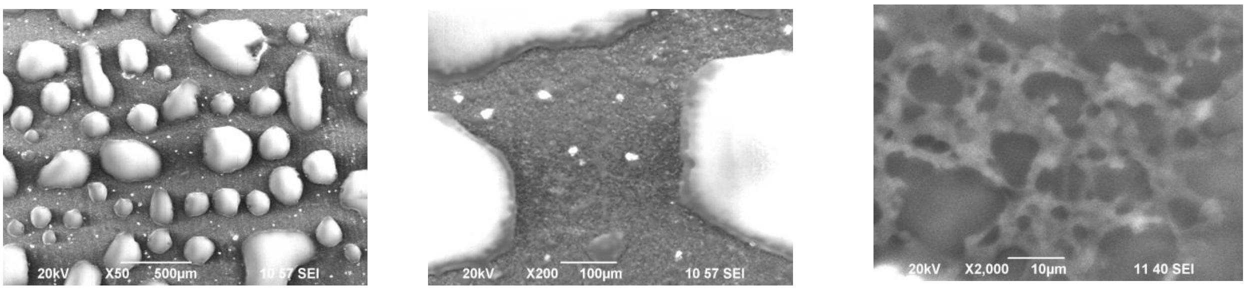
(c)

Fig. 3. Microstructure of the Sovetskii cheese sample prior to drying: (a) magnification of 50×; (b) 200×; and (c) 2000×.

Structure, texture, and pattern of cheese characterize the correctness of the development of biochemical and physicochemical processes upon cheese production. The structure of the dense product is the size and spatial arrangement of individual particles or components.

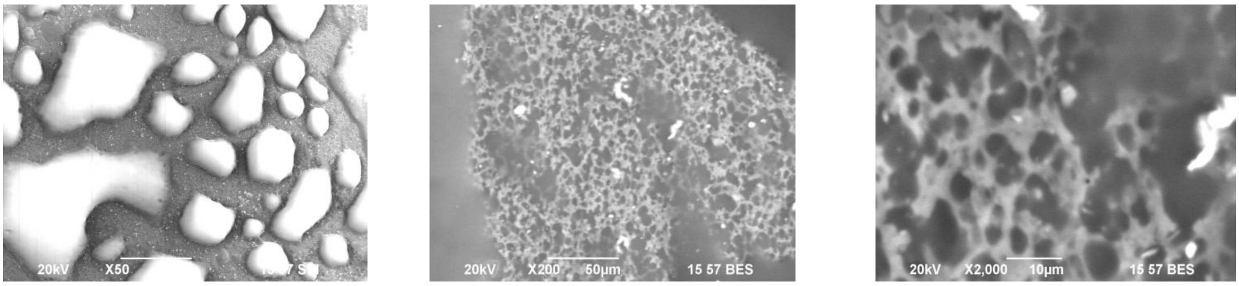
Each type of cheese has its own microstructure, but in general, the structure of all rennet cheese varieties contains the same structural elements. Its macrograins contain various inclusions, or micrograins.

Fat globules from 50 to 300 μm in size are evenly spread over the cheese surface. At magnification of 1000× and 2000×, cell-like structure of the cheese mass is observed. The cell-like structure is formed by protein matrix with capillaries of 10–12 μm retaining moisture. After vacuum drying, structure of cheese unfolds. In dry cheese, due to low water content of 4–7 %, structure and capillaries, which were not detected in the microimages of cheese before drying, are better seen.



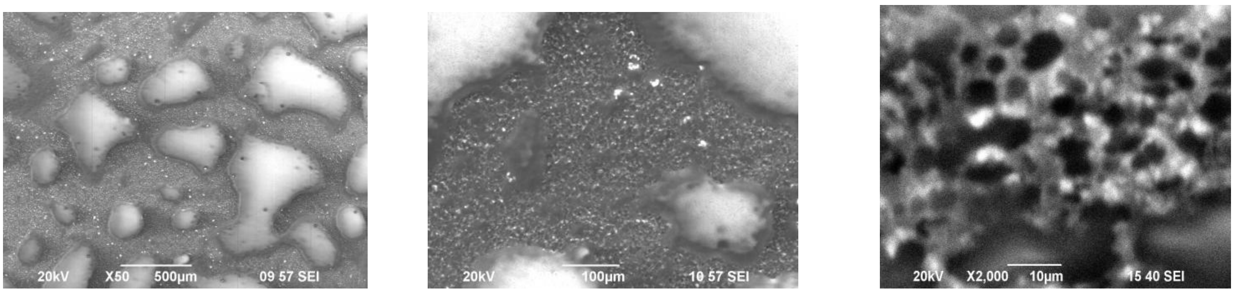
(a) (b) (c)

Fig. 4. Microstructure of the Gollandskii cheese sample prior to drying: (a) magnification of 50×; (b) 200×; and (c) 2000×.



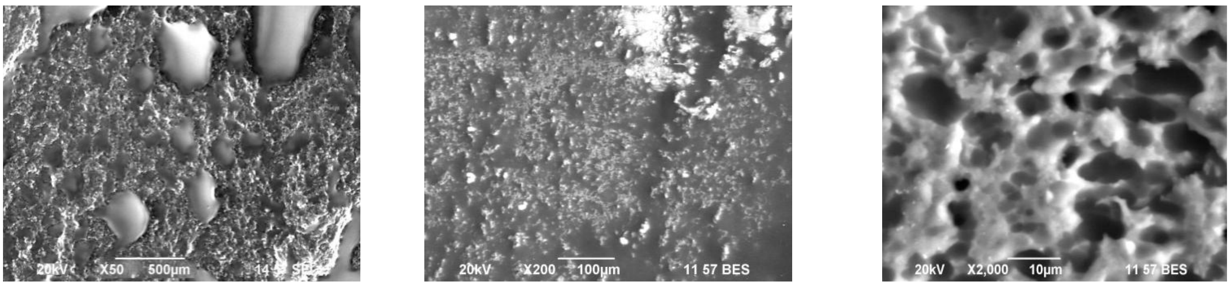
(a) (b) (c)

Fig. 5. Microstructure of the Ozernyi cheese sample prior to drying: (a) magnification of 50×; (b) 200×; and (c) 2000×.



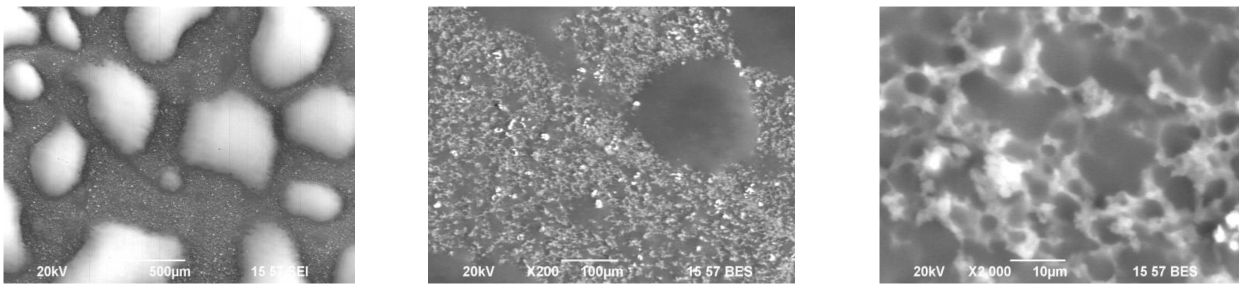
(a) (b) (c)

Fig. 6. Microstructure of the dried Sovetskii cheese sample: (a) magnification of 50×; (b) 200×; and (c) 2000×.



(a) (b) (c)

Fig. 7. Microstructure of the dried Gollandskii cheese sample: (a) magnification of 50×; (b) 200×; and (c) 2000×.



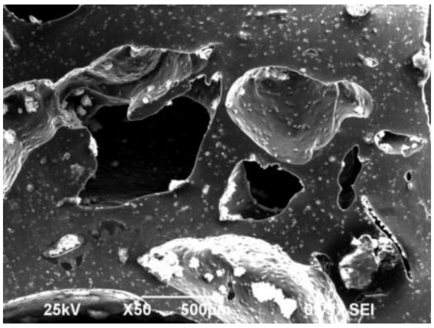
(a) (b) (c)

Fig. 8. Microstructure of the dried Ozernyi cheese sample: (a) magnification of 50×; (b) 200×; and (c) 2000×.

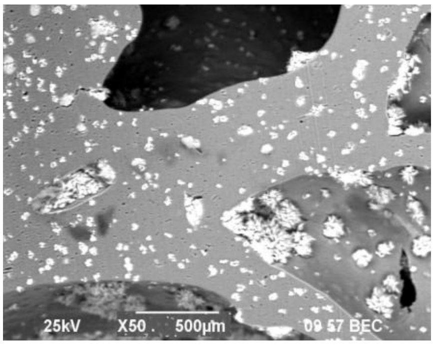
After the dryer has reached the residual pressure mode, heating is turned on, water boils rather soon at the low pressure, and leaves the protein mass of the cheese. Intensive vapor formation and moisture diffusion from the cheese surface occur. Size of capillaries containing the moisture do not change after drying and even become larger, reaching 5–15 μm.

After drying, fat globules merge into larger shapes with size reaching from 100 to 700 μm, that is, fat globule size in the dry cheese is almost twice as large as in cheese before drying. Studies on microstructure of different solid cheese varieties proved that no shrinkage of cheese occurs upon vacuum drying.

Electron microscopy revealed calcium phosphate depositions in cheese. In the course of microstructure studies of cheese sample before and after drying, individual particles contained in cheese ionized in the electron beam and produced g by low on the microimages (Fig. 9).



(a)



(b)

Fig. 9. Depositions of calcium salts in Kostromskoi cheese sample: (a) before and (b) after drying.

To identify the particles, analysis of element distribution over the cheese mass was performed. Maps of element distribution in cheese before and after drying were obtained. In the maps of element distribution, sites of phosphate and calcium localization are distinctly seen. Locations of phosphate and calcium overlap. Also, these sites overlap with the glowing particles in the images.

Calcium phosphate shows weak conductivity. Under the effect of electron beam it is ionized and produces glowing in the images. In dry cheese, calcium phosphate concentrates and the particles increase in size. Particles of calcium phosphate in Kostromskoi cheese sample before drying were of 10–12 μm and evenly spread. After drying, calcium phosphate particles increased in size to 20–30 μm. In dry cheese, calcium phosphate is concentrated and the particles are aggregated. It concentrates the most in pores and microvoids of the cheese.

Calcium phosphate is present in cheese in the form of accumulations of crystal micrograins. Crystal micrograins are round-shaped formations (Fig. 10), comprising wedge-like crystals, 20–30 μm in diameter.

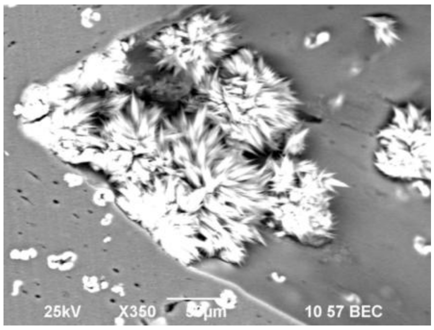
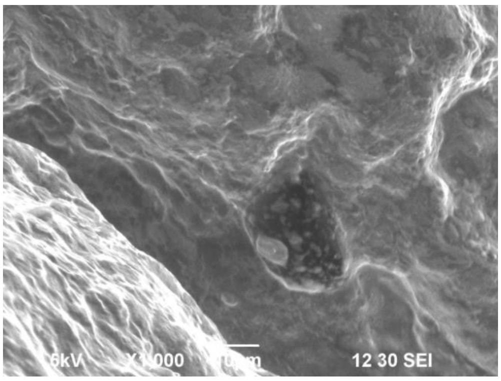
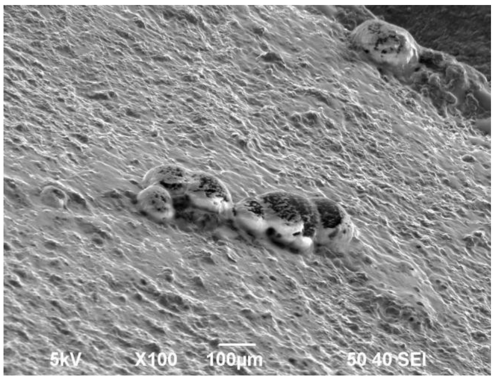


Fig. 10. Calcium phosphate in a sample of dry Kostromskoi cheese.

In mature cheese of the Kostromskoi or Rossiiskii variety, calcium lactate was detected (Fig. 11). Formation of calcium lactate should be associated with the process of cheese ripening, since it only occurs in mature cheese. Size of calcium lactate particles in Kostromskoi and Rossiiskii cheese samples was 200 × 150 μm. Figure 12 presents images of calcium lactate in Rossiiskii cheese sample.

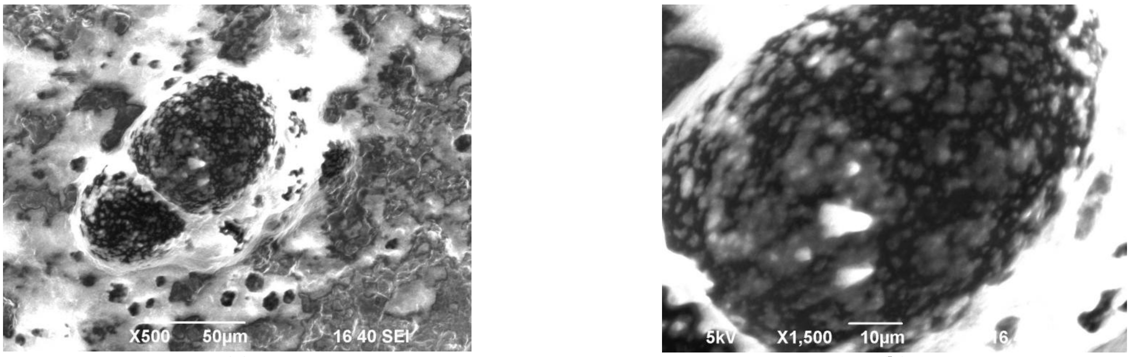


(a)



(b)

Fig. 11. Calcium lactate in mature cheese samples: (a) Kostromskoi and (b) Rossiiskii.



(a) (b)

Fig. 12. Calcium lactate in Rossiiskii cheese sample at magnification of a, 500×, and b, 1500×.

Therefore, images of cheese microstructure obtained with a scanning electron microscope allowed investigation of cheese structure before and after drying and their comparison. Structure of cheese is a protein matrix pierced with capillaries containing moisture; fat globules are contained in the protein matrix and on surface of cheese. Capillaries are of round and oval shape. The number and size of the capillaries influence the cheese pattern, which is characterized by shape and arrangement of holes and voids. Calcium phosphate depositions were detected using the electron microscopy. Calcium phosphate particles in cheese before drying are 10–12 μm big. After drying, they increase to 20–30 μm. Calcium phosphate particles in the dry cheese concentrate and agglomerate into larger particles. The highest concentration of calcium phosphate is reached in pores and microvoids of the dry cheese. In mature cheese samples, calcium lactate was detected.

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