

## Analysis of The Mature "Pokrovskiy" Cheese Microstructure Before Drying, After Freeze Drying and After Vacuum Drying

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### Abstract

The microstructure of the mature "Pokrovskiy" cheese samples was analysed before drying, after freeze-drying and vacuum drying treatment. The obtained micrographs made it possible to analyse the cheese microstructure, to measure the size of fat globules and micro-capillaries. The inner structure of the "Pokrovskiy" cheese samples after freeze drying is more acicular than the one of the cheese after vacuum drying treatment. It is certain that vacuum and freeze drying treatment will not shrink the cheese. The structure of dry cheese samples after vacuum treatment is more vivid in comparison with the structure of freeze-dried cheese samples, which is determined by the peculiarities of the drying processes. Profiles of the elemental composition of the "Pokrovskiy" cheese samples before drying, after vacuum and freeze drying treatment were obtained. The dry cheese showed, that calcium phosphate was in the form of characteristic formations, similar in size and shape to formations in the cheese samples before drying treatment.

### Introduction

The structure, texture and pattern of the cheese samples give characteristics of the bio-chemical and physicochemical processes correctness during the cheese production and, consequently, the quality of the finished product.

The texture of a dense product is determined as the size and spatial arrangement of individual particles or components. The sizes of structure components and their distribution are analysed by various methods [1, 2].

Each variety of cheese samples has its own characteristic microstructure, but in general, for all rennet cheeses, it consists of the same structural elements. Macrograins contain various inclusions – micro-grains. These include fatty micro-grains, crystalline deposits of calcium salts and colonies of microorganisms [3].

Angular and oval micro-grains are often found in cheeses. They are usually located at the junction of some macrograins. Their appearance is explained by gases'

formation during the ripening period of the cheese production. The accumulation of gases in micro-grains provokes the formation of pores [4].

The nature of the cheese sample texture is determined primarily by its structure patterns - the size and distribution of macro- and micro-grains, and layers as well. Other factors influencing the texture of cheese are the protein decay rate and degree, the composition of the non-decomposed paracasein complex (calcium content in cheese samples), the state and amount of such elements as moisture, fat, etc. in the cheese mass structure.

The composition of the paracasein complex determines the curd ability to bind and retain moisture. The higher it is, the more calcium is in the complex and vice versa. The calcium content in the complex depends on the amount of lactic acid accumulated in the cheese mass. With a significant amount of acid, the process of calcium elimination from the complex is active, the mass swells poorly and the cheese acquires a prickly and crumbly consistency.

## Experimental Section

This section considers the result of freeze- and vacuum-drying treatment impact on the "Pokrovskiy" cheese microstructure with a ripening period of 30 days.

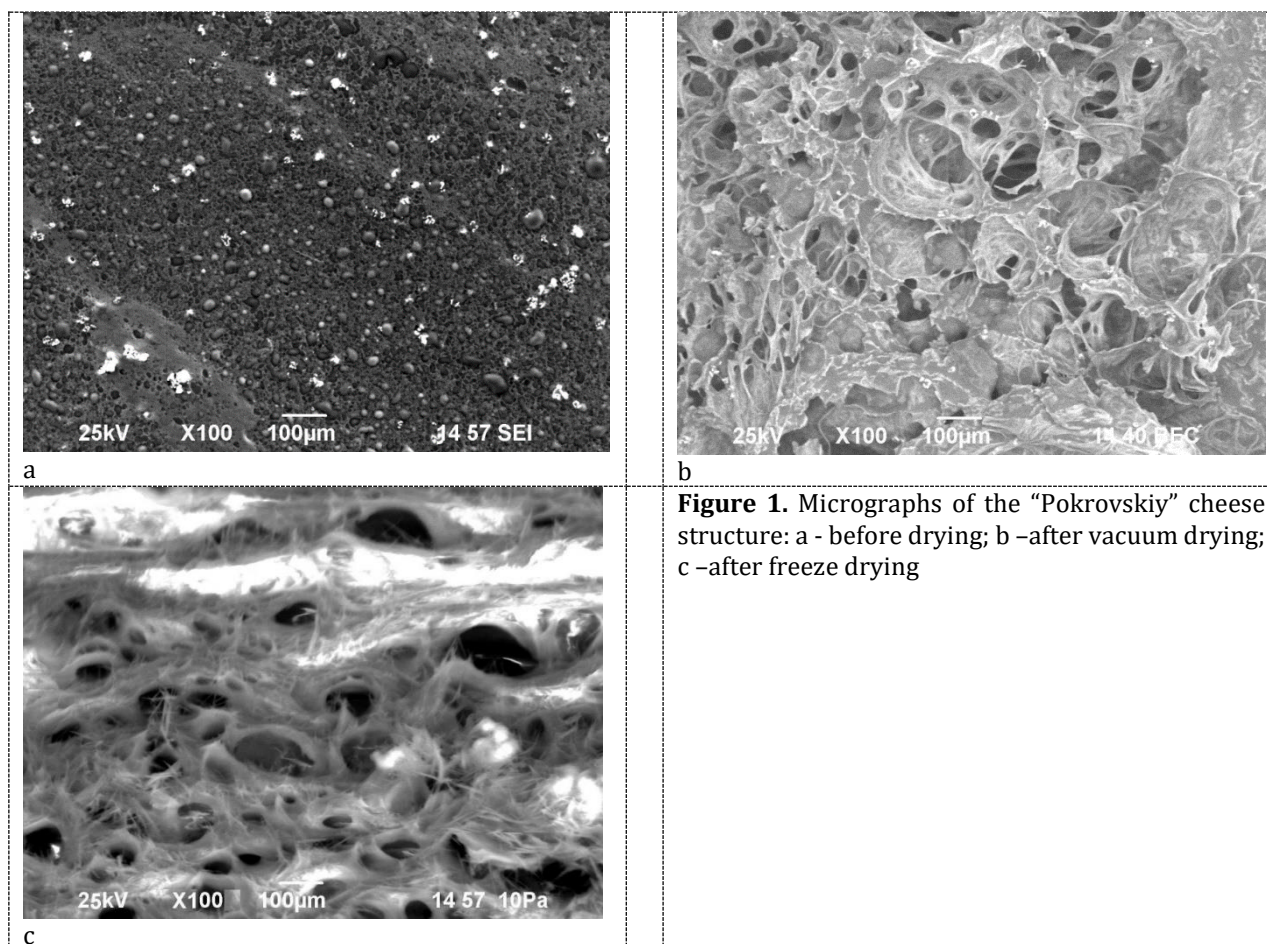
Many researchers performing the research of the cheese structural elements, rennet samples are frozen with solid carbon dioxide and sublimated [5, 6]. In these studies, the freeze drying treatment of the "Pokrovskiy" cheese was organized in the following sequence: self-freezing in a sublimator and subsequent sublimation of ice. Vacuum cheese drying treatment was performed at a residual pressure of 2-3 kPa; during the entire drying process period, the cheese had a positive temperature.

## Result Section

Fig. 1 shows micrographs of the cheese "Pokrovskiy" structure before drying, after vacuum and freeze drying treatment. The given micrographs are of a fairly high quality. The cheese surface layer before drying treatment

(Fig. 1 a) is flat and closed, which indicates the presence of a sufficiently large amount of moisture content. Content of moisture in the cheese "Pokrovskiy" is (45-47)%. Globules of fat (10-30) microns in size and micro-capillaries (2-7) microns in size in which moisture is distributed are evenly distributed over the cheese surface.

After vacuum and freeze drying, the cheese structure patterns become "expanded" (Fig. 1 b, c). The structure and capillaries become visible, which were not visible in the micrographs of ordinary cheese. It should be established that the structure of the "Pokrovskiy" cheese after vacuum drying treatment is more developed than the structure of the freeze-dried cheese. This is due to the peculiarities of the moisture removal process. During freeze drying treatment, moisture is removed from frozen cheese by phase transition "ice-vapor"; the intensity of moisture removal is high, but significantly lower than during vacuum drying treatment. Moisture is removed evenly with minimal deformation effects on the structure.



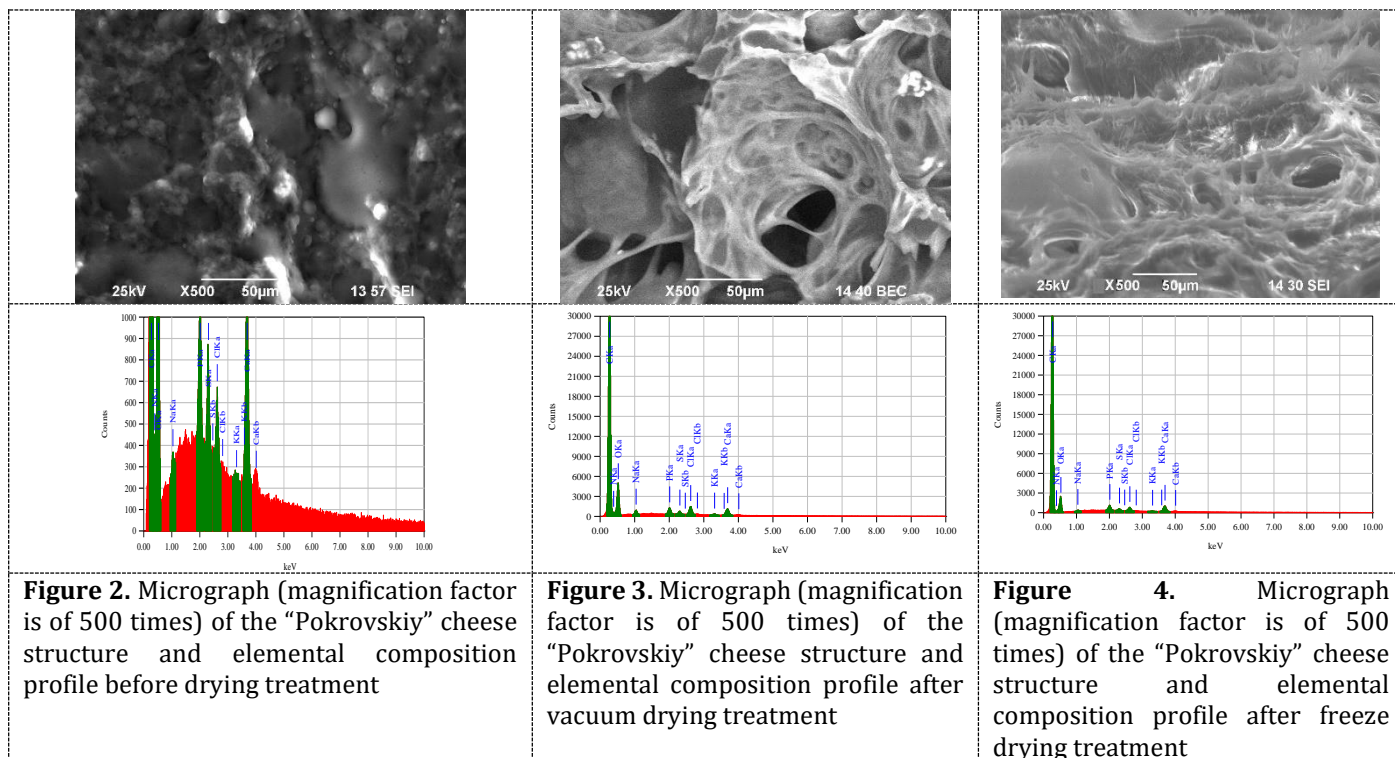
**Figure 1.** Micrographs of the "Pokrovskiy" cheese structure: a - before drying; b -after vacuum drying; c -after freeze drying

Vacuum drying treatment takes place at a positive temperature, when the unit reaches the residual pressure mode and the heat supply is switched on, moisture boils up rather quickly under reduced pressure and tends to leave the protein mass of the cheese. Intensive vaporization and diffusion of moisture from the cheese surface occurs. Due to the rapid release of moisture to the outside, the structure

of the cheese is deformed. Regardless of the way the cheese was dried, the capillary size ranges from 5 to 100 microns. Capillaries of these sizes are not visible in the "Pokrovskiy" cheese before drying treatment due to the fact that its structure is closed, and the capillaries themselves are filled with liquid. Moreover, large capillaries are located in the thickness of the cheese.

Figures 2, 3 and 4 show micrographs (magnification factor is of 500 times) and profiles of the elemental composition

of the "Pokrovskiy" cheese before drying, after vacuum and freeze-drying treatment, respectively.



With a multiplicity of magnification of 500 times, as well as with 100 times magnification, it is seen that the structure of the cheese before drying treatment is closed and contains a large amount of moisture (Fig. 2). There are fat globules on the cheese surface.

In vacuum-dried cheese, the protein matrix and the capillaries structure of the cheese mass are clearly visible. The structure of freeze-dried cheese is more acicular compared to vacuum-dried cheese. In the dry "Pokrovskiy" cheese after vacuum and freeze-drying treatment, fat is distributed in a thin film over the surface and is also inside the protein structure. During the drying process, fat globules that are less than 2 microns in size are dispersed.

The presented micrographs of the "Pokrovskiy" cheese structure confirm that fat is not melted out during vacuum and freeze-drying treatment. This, in turn, testifies to the correct choice of operating parameters and high-quality indicators of dry cheeses. When the operating parameters of the drying process deviate from the rational ones (an

increase in the drying temperature or heat load), destabilization and melting of fat occurs. Free fat is susceptible to oxidation, which leads to premature deterioration of the product.

According to micrographs of dry cheeses, the thickness of the protein layers was determined to be from 5 to 15 microns. In large capillaries, the thickness of interlayers is greater than in small ones.

Comparison of the "Pokrovskiy" cheese micrographs before drying, after freeze-drying and vacuum drying treatment, prove that the cheese does not shrink during the process of vacuum and freeze-drying treatment. Since during freeze-drying, moisture is removed from the frozen product, and during vacuum drying treatment, the rate of moisture removal is intense and uniform.

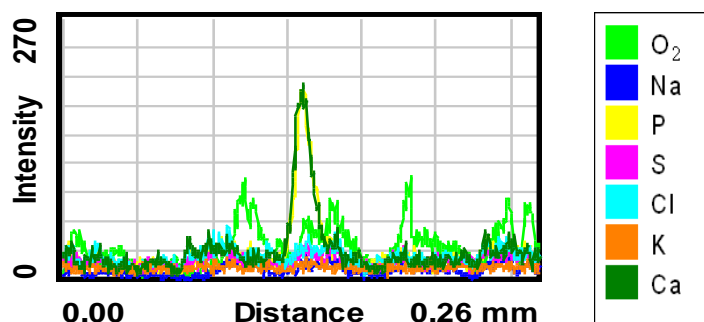
Based on the elemental composition profiles, table 1 was compiled; it shows the elemental composition of mature "Pokrovskiy" cheese - ripening period 30 days.

Element	"Pokrovskiy" cheese		
	before drying	after drying	after vacuum drying
C	83,01	83,69	83,21
N	10,44	10,72	8,15
O <sub>2</sub>	2,81	1,77	3,55
Na	0,08	0,15	0,48
P	0,93	0,93	1,04
S	0,59	0,37	0,52
Cl	0,47	0,76	1,43
K	0,04	0,11	0,25
Ca	1,62	1,49	1,35
Total	100,0	100,0	100,0

**Table 1:** Elemental composition of mature "Pokrovskiy" cheese before drying, after freeze and vacuum drying treatment, %

The sodium, chlorine and potassium content of dry cheese increases. This is due to the concentration of salts in the dry cheese. Calcium phosphate is well detected in dry cheese in

the form of characteristic formations, similar in size and shape to formations in the cheese before drying treatment (Fig. 5).



**Figure 5:** Linear spectrum of the element distribution in the dry "Pokrovskiy" cheese samples.

Three characteristic peaks are highlighted in the linear spectrum confirming the presence of calcium phosphate in the dry cheese. Distribution of oxygen, sodium, sulfur, chlorine, potassium is even throughout the entire thickness of the cheese.

## Conclusions

Basing on the data analysis of the mature "Pokrovskiy" cheese microstructure before and after freeze-drying and vacuum drying treatment, the following results were established:

- micrographs of the "Pokrovskiy" cheese structure were made before drying, after vacuum and freeze drying, which made it possible to study the microstructure of the cheese, determine the size of fat globules and micro-capillaries. The structure of the "Pokrovskiy" cheese after freeze-drying is more acicular in comparison with vacuumdried cheese;
- according to the "Pokrovskiy" cheese micrographs before drying, after freeze-drying and vacuum drying treatment, it is certain that vacuum and freezedrying do not cause cheese shrinkage. The structure of dry cheese after vacuum drying is more developed in comparison with the structure of freezedried cheese, which is explained by the peculiarities of the drying processes;

- the capillary size of dry cheese samples after vacuum drying treatment is set from 40 to 100 microns; after freeze drying- from 5 to 60 microns;
- based on micrographs of dry cheeses, the protein thickness layer was determined to be from 5 to 15 microns. The thickness of the protein layers between large capillaries is greater (10-15 microns) than between small ones (5-10 microns);
- profiles of the elemental composition of the "Pokrovskiy" cheese was analysed before drying, after vacuum and freeze-drying treatment. According to the profiles obtained, it is determined that the mass fraction of carbon, phosphorus, sulfur and calcium after freeze-drying and vacuum drying treatment practically does not change. The content of sodium, chlorine and potassium in dry cheese of vacuum and freeze-drying treatment increases compared to cheese before drying treatment;
- in dried samples of cheese, Calcium phosphate was detected as particular formations, similar in size and shape to formations in the cheese before drying treatment.

## References

1. Rubinskienė M., Viškėlis P., Dambrauskienė E., Viškėlis J., Karklelienė R., Effect of drying methods on the chemical composition and colour of peppermint (*Mentha × piperita* L.) leaves, *Zemdirbyste-Agriculture*, 102 (2), 223–228, 2015.
2. Golchin, A.; Farahany, T.Z. Biological Products: Cellular Therapy and FDA Approved Products. *Stem Cell Reviews and Reports* 2019, 15, 166-175, <https://doi.org/10.1007/s12015-018-9866-1>.
3. Golchin, A.; Shams, F.; Kangari, P.; Azari, A.; Hosseinzadeh, S. Regenerative Medicine: Injectable Cell Based Therapeutics and Approved Products. *Adv Exp Med Biol* 2020, 1237, 75-95, [https://doi.org/10.1007/5584\\_2019\\_412](https://doi.org/10.1007/5584_2019_412).
4. Mungure, T.E.; Roohinejad, S.; Bekhit, A.E.; Greiner, R.; Mallikarjunan, K. Potential application of pectin for the stabilization of nanoemulsions. *Curr Opin Food Sci* 2018, 19, 72-76, <https://doi.org/10.1016/j.cofs.2018.01.011>
5. Zdravko M., Aleksandra N., Stela D., Radomir V., Optimization of frozen wild blueberry vacuum drying process, *Hemijskaindustrija*, 69 (1), 77–84, 2015.
6. Moreno, M.S. Engenharia de Tecidos nas substituição de tecido ósseo. Dissertação (Mestrado). Universidade Fernando Pessoa, Porto, 2014. 5. Dai, Z.; Ronholm, J.; Tian, Y.; Sethi, B.; Cao, X. Sterilization techniques for biodegradable scaffolds in tissue engineering applications. *Journal of Tissue Engineering* 2016, 7, <https://doi.org/10.1177/2041731416648810>.
7. Yang L.Q. Dry sliding behavior of a TiZr-based alloy under air and vacuum conditions, *Journal of Materials Engineering and Performance*, 28, 3402-3412, 2019.
8. Fernandez-Yague, M.A.; Abbah, S.A.; McNamara, L.; Zeugolis, D.I.; Pandit, A.; Biggs, M.J. Biomimetic approaches in bone tissue engineering: Integrating biological and physicochemical strategies. *Advanced Drug Delivery Reviews* 2015, 84, 1-29, <https://doi.org/10.1016/j.addr.2014.09.005>.
9. Gautam, S.; Dinda, A.K.; Mishra, N.C. Fabrication and characterization of PCL/gelatin composite nanofibrous scaffold for tissue engineering applications by electrospinning method. *Materials Science and Engineering: C* 2013, 33, 1228-1235, <https://doi.org/10.1016/j.msec.2012.12.015>.
10. Couto, R.O.; Araujo, R.R.; Tacon, L.A.; Conceição, E.C.; Bara, M.T.; Paula, J.R.; Freitas, A.P. Development of a phytopharmaceutical intermediate product via spray drying. *Drying Technol.* 2011, 29, 709-718, <http://dx.doi.org/10.1080/07373937.2010.524062>
11. Wendel, S.; Celik, M. An overview of spray-drying applications. *Pharm Tech*. 1987, 27, 125-156.
12. Lopej, Vega-Galvez A., Bilbao-Sainz C., Chiou Bor-Sen, Uribe E., Quispe-Fuentes I., Influence of vacuum drying temperature on: Physico-chemical composition and antioxidant properties of murta berries, *Journal of Food Process Engineering*, 40 (6), UNSP e12569, 2017.
13. Kang, H.W.; Tabata, Y.; Ikada, Y. Fabrication of porous gelatin scaffolds for tissue engineering. *Biomaterials* 1999, 20, 1339-1344, [https://doi.org/10.1016/s0142-9612\(99\)00036-8](https://doi.org/10.1016/s0142-9612(99)00036-8).
14. Han, J.; Zhou, Z.; Yin, R.; Yang, D.; Nie, J. Alginate-chitosan/hydroxyapatite polyelectrolyte complex porous scaffolds: Preparation and characterization. *International Journal of Biological Macromolecules* 2010, 46, 199-205, <https://doi.org/10.1016/j.ijbiomac.2009.11.004>.
15. Monsoor, M.A.; Kalapathy, U.; Proctor, A. Improved method for determination of pectin degree of esterification by diffuse reflectance Fourier transform infrared spectroscopy. *Journal of Agricultural and Food Chemistry*. 2001, 49, 2756-2760, <https://doi.org/10.1021/jf0009448>
16. Xie L., Mujumdar Arun S., Fang Xiao-Ming, Wang Jun, Dai Jian-Wu, Du Zhi-Long, Xiao Hong-Wei, Liu Yanhong, Gao Zhen-Jiang, Far-infrared radiation heating assisted pulsed vacuum drying (FIR-PVD) of wolfberry (*Lycium barbarum* L.): Effects on drying kinetics and quality attributes, *Food and Bioprocess Processing*, 102, 320-331, 2017.
17. Rabeta M., Lin S., Effects of different drying methods on the antioxidant activities of leaves and berries of *Cayratia trifolia*, *Sains Malaysiana*, 44 (2), 275-280, 2015.
18. Ermolaev V.A., Development of technology for vacuum drying of fat-free cottage cheese: thesis on competition of a scientific degree of candidate of technical sciences. *Kemerovo Technological Institute of Food Industry*, 168, 2008.
19. Ermolaev V.A. Kinetics of the vacuum drying of cheeses, *Foods and Raw Materials*, 2, 2, 2014.
20. Temerbayeva M. Using of creamy bioaditives in the production of melted cheese, 7 (4.38), 2018.
21. Ermolaev V.A., Cheese as a Tourism Resource in Russia: The First Report and Relevance to Sustainability, *Sustainability*, 11, 5520, 2019.
22. Ermolaev V.A. Missions of Russian Cheese Producers: Principal Components and Relevance for Rural Communities, *Agriculture*, 10, 68, 2020.
23. Rebelo, M.; Chaud, M.; Balcão, V.; Vila, M.; Aranha, N.; Hanai-Yoshida, V.; Alves, T.; Oliveira Jr, J. Chitosan-based scaffolds for tissue regeneration: Preparation and microstructure characterisation. *European Journal of Biomedical and Pharmaceutical Sciences* 2016, 3, 15-24.
24. Flores-Ruiz, E.; Miranda-Navales, M.G.; Villasís-Keever, M.A. The research protocol VI: How to choose the appropriate statistical test. *Inferential statistics. Rev Alerg Mex*. 2017, 64, 364-370, <https://doi.org/10.29262/ram.v64i3.304>.
25. Einhorn-Stoll, U.; Kunzek, H.; Dongowski, G. Thermal analysis of chemically and mechanically modified pectins. *Food Hydrocolloids*. 2007, 21, 1101-1112, <http://dx.doi.org/10.1016/j.foodhyd.2006.08.004>
26. Lv, Q.; Feng, Q. Preparation of 3-D regenerated fibroin scaffolds with freeze drying method and freeze drying/foaming technique. *Journal of Materials Science: Materials in Medicine* 2006, 17, 1349-1356, <https://doi.org/10.1007/s10856-006-0610-z>.

27. Garcia Pelizaro, T.A.; Tolaba, A.G.; Rodriguez-Chanfrau, J.E.; Veranes-Pantoja, Y.; Guastaldi, A.C. Influence of the Application of Ultrasound During the Synthesis of Calcium Phosphates. *Journal of Bionanoscience* 2018, 12, 733-738, <https://doi.org/10.1166/jbns.2018.1585>.
28. Mu Yanqiu, Zhao Xinhuai, Liu Bingxin, Liu Chenghai, Zheng Xianzhe., Influences of microwave vacuum puffing conditions on anthocyanin content of raspberry snack, *International Journal of Agricultural and Biological Engineering*, 6 (3), 80-87, 2013.
29. Ermolaev V.A. Missions of Russian Cheese Producers: Principal Components and Relevance for Rural Communities, *Agriculture*, 10, 68, 2020.
30. Nile S., Park S., Edible berries: bioactive components and their effect on human health, *Nutrition*, 30 (2), 134-144, 2014.
31. Kellogg J., Wang J., Flint C., Lila M., Ribnicky D., Kuhn P., Raskin I., De Mejia E., Alaskan wild berry resources and human health under the cloud of climate change, *Journal of Agricultural and Food Chemistry*, 58 (7), 3884-3900, 2010.
32. Bowen-Forbes C., Nair M., Zhang Y., Anthocyanin content, antioxidant, anti-inflammatory and anticancer properties of blackberry and raspberry fruits, *Journal of Food Composition and Analysis*, 23 (6), 554-560, 2010.
33. Afrin S., Gasparrini M., Forbes-Hernandez T., Reboredo-Rodriguez P., Giampieri F., Battino M., Mezzetti B., Varela-López A., Promising health benefits of the strawberry: a focus on clinical studies, *J. Agric. Food Chem*, 64 (22), 4435-4449, 2016.
34. Artnaseaw A., Theerakulpisut S., Benjapiyaporn C., Development of a vacuum heat pump dryer for drying chilli, *Biosystems Engineering*, 105 (1), 130-138, 2010.
35. Zecchi B., Clavijo L., MartínezGarreiro J., Gerla P. Modeling and minimizing process time of combined convective and vacuum drying of mushrooms and parsley, *Journal of Food Engineering*. 104 (1), 49-55, 2011.
36. Mannanov U., Mamatov Sh., Shamsutdinov B., Research and study mode vacuum infrared drying vegetables, *Austrian Journal of Technical and Natural Sciences*, 3-4, 38-41, 2016.
37. Dalvi-Isfahan M. Review on identification, underlying mechanisms and evaluation of freezing damage, *Journal of Food Engineering*, 255, 50-60, 2019
38. Li T. Effect of E-polysine on K-carrageenan gel properties: phenology, water mobility, thermal stability and microstructure, *Food Hydrocolloids*, 95, 212-218, 2019.
39. Nicolai T. Gelation of food protein-protein mixtures, *Advances in Colloid and Interface Science*, 270, 147-164, 2019.
40. Amy Y.Xu, Effects of polysaccharide charge pattern on the microstructures of  $\beta$ -lactoglobulin-pectin complex coacervates, studied by SAXS and SANS, *Food Hydrocolloids*, 77, 952-963, 2018.