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КРІОГЕННА ТЕХНОЛОГІЯ ДРІБНОДИСПЕРСНИХ ПОРОШКОПОДІБНИХ ДОБАВОК ІЗ ТОПІНАМБУРА

Р.Ю. Павлюк, В.В. Погарська, О.С. Бессараб, А.О. Борисова, К.С. Балабай

Запропоновано та розроблено кріогенну технологію дрібнодисперсних порошкоподібних добавок із топінамбура, яка відрізняється від традиційних застосуванням кріогенного «шокового» заморожування, низькотемпературного механічного подрібнення та сублімаційного сушіння, що дозволяє більш повно вилучити біологічно активні речовини із зв'язаного із нанокомплексами стану у вільний та зруйнувати значну частину полісахариду інуліну до окремих його мономерів – фруктози.

Ключові слова: *кріогенне заморожування, низькотемпературне подрібнення, сублімаційне сушіння, топінамбур, інулін, дрібнодисперсні добавки.*

КРИОГЕННАЯ ТЕХНОЛОГИЯ МЕЛКОДИСПЕРСНЫХ ПОРОШКООБРАЗНЫХ ДОБАВОК ИЗ ТОПИНАМБУРА

Р.Ю. Павлюк, В.В. Погарская, А.С. Бессараб, А.А. Борисова, Е.С. Балабай

Предложена и разработана криогенная технология мелкодисперсных порошкообразных добавок из топинамбура, которая отличается от традиционных применением криогенного «шокового» замораживания, низкотемпературного механического измельчения и сублимационной сушки, что позволяет более полно извлечь биологически активные вещества из связанного с нанокомплексами состояния в свободное и разрушить значительную часть полисахарида инулина до отдельных его мономеров – фруктозы.

Ключевые слова: *криогенное замораживание, низкотемпературное измельчение, сублимационная сушка, топинамбур, инулин, мелкодисперсные добавки.*

CRYOGENIC TECHNOLOGY OF FINE-DISPERSED POWDERED ADDITIVES FROM TOPINAMBOUR

R. Pavlyuk, V. Pogarskaya, A. Bessarab, A. Borysova, K. Balabai

Cryogenic technology of fine-dispersed powdered additives based on topinambour, which differs from traditional technologies with the use of cryogenic

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"shock" freezing, low-temperature mechanical grinding and sublimation drying is proposed and developed. It allows both to save all biologically active substances and liberate them from the bound with nanocomplexes condition into free, and destroy large part of polysaccharide inulin into its separate monomers – fructose.

It differs from traditional technologies because it excludes heat treatment of product fully and it is based on the refrigerating treatment of raw materials during the preparation of topinambour, freezing and low-temperature fine-dispersed grinding with the following sublimation drying.

New technology makes it possible to obtain additives from topinambour in the form of fine-dispersed powders with a particle size ten times smaller than during traditional grinding. Their quality by content of fructose in free condition and biologically active substances extracted from bound condition surpasses domestic and foreign analogues.

Amino acid composition of proteins in free and bound conditions in original inulin-containing raw materials (sublimation-dried topinambour) and fine-dispersed powdered additives from topinambour was compared. It is established that significant mechanical destruction of protein molecules into separate amino acids, their transformation from bound condition to free, occur during the fine-dispersed grinding of frozen inulin-containing raw materials in comparison with raw materials. So, mass fraction of bound amino acids in fine-dispersed powdered additives from topinambour decreased 2 times in comparison with the original raw materials. At the same time mass fraction of amino acids in free condition increased 1,7 – 10 times.

Keywords: *cryogenic freezing, low-temperature grinding, sublimation drying, topinambour, inulin, nano-powders.*

General formulation of problem. This scientific work is devoted to the development of cryogenic technology of fine-dispersed powdered additives from topinambour, revealing the influence patterns of cryogenic freezing and cryogenic mechanical destruction to biopolymers inulin and protein, studying its mechanolysis to separate monomers – fructose and amino acids and saving biologically active substances during the obtaining of fine-dispersed additives in the powder form.

Nowadays, the global problem all over the world is unbalanced nutrition and lack of complete proteins, vitamins, minerals and other biologically active substances (BAS) in daily ration. At that time the need of Ukraine population in these components is satisfied for about 50%. Beside it, there is immunity reduction of population in the whole Earth. In this regard, functional healthful products (especially fruits and vegetables and combined dairy and plant products aimed to health promotion) are very popular in many countries. This issue is given much attention in the works of both domestic and foreign scientists [1–10].

Analysis of recent research works and publications. Inulin-containing raw materials, particularly topinambour, occupies a special place among the plant materials, used for making functional healthful products [1–3].

In Ukraine, the traditional source of inulin is topinambour and chicory root. Inulin is a natural linear polysaccharide, the main structural monomer of which is fructose remains connected with β-fructoside bonds. It is commonly believed that the use of food fortified with inulin-containing raw materials, is able to reduce general carbohydrate load to a human body, which leads to reduction of glycemic index and calorie concentration and great increase of their biological value. The assimilation degree of topinambour inulin in a human body is not fully clear.

It is well known that during the consumption of fresh or processed (in the form of powders, flour, sauce, syrup, etc.) topinambour, inulin and oligofructoses, which are included to it and built from fructose, remains connected with β-fructoside bonds, are not splited by a human body's enzymes (either in stomach or in small intestine) to fructose. Therefore, inulin goes a way from mouth cavity to colon almost unchanged. This fact has been proved by many scientists from around the world. In other words, plant inulin is poorly-digestible by a human body. It is also known that existing technologies of getting different additives from topinambour in the form of powders, pastes, flour, puree, extracts with the use of steam-thermal processing, drying, do not allow to transform the part of inulin into easily digestible fructose.

There is another significant problem during the getting of powders, syrups, puree from topinambour – product darkening due to oxidative enzymes. The technological methods that allow inactivate oxidative enzymes and obtain high quality end products are in search. In addition, during the getting of different products from topinambour with the use of thermal processing methods, the destruction of fructose is 10 to 20%.

It is known that the most effective way of plant raw materials processing during the obtaining of puree is fast "shock" freezing, which provides the highest preservation of vitamins and other biologically active substances [1–3; 9]. However, there are the losses of cell sap and vitamins during the unfreezing of frozen products and the guarantee shelf life of frozen products are limited to 6 months. Cryogenic "shock" freezing which means freezing with the use of cryogenic liquids (liquid nitrogen, liquid carbon dioxide, etc.) is widely adopted abroad [1–3; 9]. In Ukraine this way of freezing has not found its application yet, cryogenic technologies have not designed and biochemical and physical-chemical processes during the obtaining of frozen puree are not studied. Literature analysis has shown that cold technologies of processing of topinambour (such as cryogenic "shock" freezing, sublimatic drying, cryogenic grinding) are absent [1–3; 9].

According to the authors of the article, one of innovative directions of the development of science and technology in international practice is use of the methods of fine-dispersed grinding, particularly food plant raw materials that lead to mechanical destruction (including cryodestruction), mechanical activation and mechanical chemistry, which were demonstrated during the increase of a degree of chopped materials dispersion. As a result, the product acquires new properties and nanostructured or nanoscale form. Currently, advanced methods of fine-dispersed grinding are widely used in metallurgical, textile, aviation, chemical, construction industries and others. In the food industry these processes almost have not been studied [2; 3; 7; 8].

In this research during the development of cryogenic technology of fine-dispersed powdered additives from topinambour as innovation, it is suggested to use cryogenic "shock" freezing with the use of liquid and gaseous nitrogen and low-temperature fine-dispersed grinding, which accompanies cryogenic destruction, mechanical activation and mechanical chemistry. Complex application allowed to develop a new method of getting canned additives in the form of fine-dispersed powder of sublimation drying with new quality characteristics and chemical composition (in comparison with raw materials), which cannot be obtained by traditional methods.

The purpose of the article is the development of cryogenic technology of fine-dispersed powdered additives from topinambour with the use of cryogenic "shock" freezing with the use of liquid and gaseous nitrogen as innovation, fine-dispersed mechanical low-temperature grinding and sublimation drying that allows to save maximally and extract the bound forms of BAS with biopolymers from raw materials and maximally realize cryogenic mechanical destruction of biopolymers – inulin, protein, cellulose into its composite monomers for better use of biopotential of plant materials and better digestibility by the organism.

To achieve this goal it is necessary to solve the following problems:

– to develop cryogenic technology of getting fine-dispersed powdered additives from topinambour with the use of cryogenic "shock" freezing (with the use of liquid and gaseous nitrogen), low-temperature grinding and sublimation drying as innovation;

– to study the influence of cryogenic "shock" freezing, fine-dispersed grinding and sublimation drying to BAS and polysaccharides, including inulin, during the getting of fine-dispersed powdered additives from topinambour;

– to study mechanolysis of topinambour's protein and transformation of bound amino acids into the free form during the getting of fine-dispersed powdered additives by cryogenic technology, and reveal the mechanism of this process;

– to study infrared spectrum of samples of fine-dispersed powders from topinambour with the size of particulate solids nearly about a few microns and traditionally grinded powders with the size of particulate solids 50 ... 250 microns;

– to study chemical composition of fine-dispersed powdered additives from topinambour in comparison with the analogs and raw materials.

Main materials of the research work. The cryogenic technology of fine-dispersed powdered additives from topinambour in the powder form of sublimation drying with the use of low temperature processing, including cryogenic "shock" freezing and low temperature mechanical grinding was proposed and developed by Kharkiv State University of Food Technologies and Trade in collaboration with experts of NUFT. The research was realised at the Department of Technology of fruits, vegetables and milk processing of Kharkiv State University of Food Technologies and Trade on the base of 2 research laboratories "Innovative cryogenic and nanotechnologies of herbal additives and healthful products" and "Technology and Biochemistry of phyto-concentrates". The work was realized with the use of modern equipment such as cryogenic program freezer with computer software, low-temperature chopper (France), cryogenic chopper, binocular microscope with program software, video-camera and calibration scale in the micrometer and nanometer diapason.

The scientific research described in this article is a continuation of the works of authors, connected with the development of cryogenic technologies of the processing of various fruits and vegetables and fine-dispersed additives in the form of frozen purees and powders that were included into the work, which was awarded the State Prize in Science and Technology of Ukraine in 2006 [2].

The main thing in the development of cryogenic technology of fine-dispersed powdered additives from topinambour with the use of freezing, cryogenic destruction and mechanical destruction is to exclude thermal processing of raw materials completely, to transform inulin into soluble form (free fructose), inactivate oxidative enzymes, to preserve BAS maximally and increase the degree of their extraction from nanocomplexes into free condition from raw materials maximally, to exclude the use of synthetic food additives completely and to obtain the high quality products.

It is established that during the "shock" freezing with the use of different speeds of freezing to different end-freezing temperatures in the product (– 18; – 20; – 25; – 30; – 40° C), sublimation-drying and fine-dispersed grinding of topinambour significant part of inulin (45…55%) is transformed to the soluble free fructose (its quantity increases 9 – 10 times comparing with initial fructose in fresh topinambour due to non-enzymatic and non-acid destruction of β-fructose bonds in inulin. This process is realized due to mechanical destruction – mechanical cracking. For example, raw materials (topinambour) contain 52,2…56,0% of inulin on an oven-dry basis, and after low-temperature processing 25,8…28,7% of inulin on oven-dry basis, and 26,4…27,3% of it is transformed into free fructose. It is established that simultaneously the destruction and degradation of cellulose and protein occur: 43…45% of cellulose is transformed to its monomers – glucose, and 50% of protein break to separate free amino acids.

It is also established that "shock" freezing and fine-dispersed grinding of topinambour, accompanying mechanical and cryogenic destruction, mechanical activation, allow both to save all biologically active substances such as phenolic compounds, ascorbic acid, tannins etc., and liberate them from the bound condition with biopolymers of nanocomplexes or nanoassociates and transform them to free condition (their amount increases 1,7 – 2,2 times comparing with the original raw materials). It allows to get a product with new chemical composition and excellent consumer properties.

The amino acid composition of proteins in free and bound conditions in the original inulin-containing raw materials (sublimation-dried topinambour) and fine-dispersed powdered additives from topinambour compared. It is established that significant mechanical destruction (mechanolysis) of protein molecules to separate amino acids, their transformation from bound condition to free condition occurs in the process of fine-dispersed grinding of frozen inulin-containing raw materials in comparison with raw materials (Table. 1). So, mass fraction of bound amino acids in fine-dispersed powdered additives from topinambour decreased 2 times in comparison with original raw materials (mass fraction of bound amino acids is 5,59 mg/100 g, fine-dispersed powdered additive from topinambour – 2,44 mg/100 g). Reduction of mass fraction of amino acids of protein after fine-dispersed grinding is 56,4%. At the same time, mass fraction of amino acids in free condition increases 1,7 – 10 times.

Therefore, it is established that disaggregation, destruction and mechanolysis of protein biopolymers occur during fine-dispersed grinding of frozen inulin-containing raw materials in comparison with the original raw materials (sublimation-dried topinambour). These processes are manifested in the reduction of approximately 45…50% of mass fraction of bound amino acids of protein due to transformation of amino acids into their free form.

Thus, the total content of amino acids is 6,8 mg in 100 g of dried topinambour, 5,59 mg of which are amino acids in a bound condition and 1,21 mg – in a free condition. The total content of amino acids in 100 g of fine-dispersed powdered additive is 6,8 mg. Among them 2,44 mg are amino acids in a bound condition and 4,36 mg – in a free condition.

New cryogenic nanotechnology of processing topinambour to fine-dispersed additives in the form of powders is developed based on the experimental data. It differs from traditional technologies because it absolutely excludes heat treatment of a product and is based on refrigeration of raw materials during the preparation of topinambour, freezing and low-temperature fine-dispersed grinding with the following sublimation drying.

Table 1

Influence of cryogenic freezing and fine-dispersed grinding to transformation of amino acids from a bound to free condition during the reception of inulin-containing fine-dispersed powdered additives

Amino acid	Mass fraction of amino acids					
	in a bound condition			in a free condition		
	Original material (dried topinambour), mg/100 g	Fine-dispersed powdered additive from topinambour, mg/ 100 g	% relatively to original material	Original material (dried topinambour), mg/100 g	Fine-dispersed powdered additive from topinambour, mg/ 100 g	% relatively to original material
Asparagine acid	0,47	0,24	51,1	0,33	0,56	169,7
Threonine	0,20	0,10	50,0	0,09	0,19	211,1
Serine	0,32	0,16	50,0	0,04	0,2	500,0
Glutamine acid	1,33	0,14	10,5	0,27	1,46	540,7
Proline	0,34	0,16	47,1	0,02	0,2	1000,0
Cystine+Glycine	0,31	0,18	58,1	0,05	0,18	360,0
Alanine	0,40	0,22	55,0	0,04	0,22	550,0
Valine	0,30	0,17	56,7	0,06	0,19	316,7
Methionine	0,12	0,08	66,7	0,03	0,07	233,3
Isoleucine	0,42	0,25	61,0	0,06	0,22	366,7
Leucine	0,40	0,21	52,5	0,04	0,23	575,0
Tyrodine	0,12	0,07	58,3	0,03	0,08	266,7
Phenylalanine	0,20	0,11	55,0	0,02	0,11	550,0
Histidine	0,11	0,06	54,5	0,04	0,09	225,0
Lysine	0,48	0,25	52,1	0,06	0,29	483,3
Arginine	0,07	0,04	57,1	0,04	0,07	175,0
Total	5,59	2,44	43,6	1,21	4,36	360,3

New technology makes it possible to obtain additives from topinambour in the form of fine-dispersed powders with a particle size ten times smaller than during traditional grinding. Their quality by the content of fructose in a free condition, and biologically active substances extracted from bound condition, surpasses domestic and foreign analogues.

It is established that chemical composition of new additives from topinambour, obtained by refrigeration technologies, significantly differs from raw topinambour (Table. 2).

Table 2

Content of biologically active substances and food nutrients in fine-dispersed powdered additive from topinambour

Name of factor	Raw topinambour		Fine-dispersed powdered additive from topinambour	
	Component mass fraction on an air-dry basis	Component mass fraction on an oven-dry basis	Component mass fraction on an air-dry basis	Component mass fraction on an oven-dry basis
Carbohydrates, including:	17,1±0,5	72,5±2,1	73,6±0,5	77,9±0,5
Inuline, %	12,8 ±0,5	54,2±2,1	25,6±1,5	27,1±1,6
Total sugar, %	4,4±0,1	18,6±0,4	22,4±1,4	23,7±1,5
fructose, %	–	–	25,6±1,5	27,1±1,6
Protein, %	1,2±0,01	6,8±0,04	6,8±0,5	6,8±0,5
Cellulose, %	2,0±0,1	8,5±0,4	3,6±0,1	3,8±0,1
Pectin, %	0,4±0,01	1,7±0,04	3,8±0,2	4,0±0,2
L-ascorbic acid, мg/100 g	10,3±0,1	43,6±0,4	78,2±2,4	82,8±2,5
Phenolic compounds (for chlorogenic acid) mg/100 g	350,0±5,7	1483,1±24,2	2800,0±15,8	2963,0±16,7
Flavone glycosides (for rutin) mg/100 g	240,0±4,8	1016,9±20,3	1800,0±12,4	1904,8±13,1
Tannins (for tannin), mg/100 g	300,0±6,4	1271,2±27,1	2160,0±14,0	2285,7±14,8
Ash-content, %	1,6±0,1	6,8±0,4	6,8±0,2	7,2±0,2
Dry substances, %	23,6±1,2	–	94,5±0,1	–
Organic acids, %	0,3±0,01	1,3±0,04	1,5±0,1	1,6±0,4

They differ by high content of fructose in a free condition. Thus, mass fraction of fructose in 100 g of a new additive from topinambour in the form of powder is 25…26% and the amount of biopolymers, such as inulin, protein, cellulose decrease almost half in comparison with raw materials (calculated on dry basis). It is also shown that the additives differ with a high content of biologically active substances, such as phenolic compounds, tannins and others with P-vitamin activity and antioxidant properties. So, mass fraction of low-molecular phenolic compounds (for chlorogenic acid) in raw topinambour is 350±5,7 mg in 100 g, flavone glycosides (for rutin) – 240±4,8 mg in 100 g, and in a fine-dispersed powdered additive from topinambour – 2800,0±15,8 mg in 100 g and 1800,0±12,4 mg in 100 g appropriately. The similar common factors concern polyphenolic compounds (appropriately 300±6,4 in raw material and 2160,0±14,0 mg in 100 g in fine-dispersed powdered additive from topinambour. Therefore, fine-dispersed powdered additives, obtained with the use of such innovations as cryogenic freezing, low-temperature grinding and sublimation drying during the processing of topinambour allow getting a new product that cannot be obtained with the use of traditional methods of processing of plant material.

The obtained results are the basis for the development of new technologies of fine-dispersed powders from topinambour for healthful nutrition with inulin in easily digestible form (up to 50…55% in the form of free fructose). According to chemical composition, new products have immunomodulatory, antineoplastic and detoxic potential. New technologies have been tested in a production environment in "CRYAS" Scientific industrial Corporation. Normative documentation for fine-dispersed powdered additives from topinambour is developed. New kinds of healthful products (cheese desserts, nano-drinks, bioyoghurts, biokephirs, new types of nano-ice-cream, fast-dissolving fruit "Instant" nano-drinks, juices, pastries, etc.) are developed on their base.

Conclusions. Cryogenic technology of fine-dispersed powdered additives based on topinambour, which differs from traditional technologies with the use of cryogenic freezing with help of liquid and gaseous nitrogen, low-temperature mechanical grinding and sublimation drying is proposed and developed. A unique new technology allows both to save all biologically active substances and liberate them from abound with biopolymers nanoassociates or nanocomplexes into free condition (almost twice more in comparison with raw materials) and destroy a large part of polysaccharide inulin into its separate monomers – fructose.

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Павлюк Раїса Юріївна, д-р техн. наук, проф., лауреат Державної премії України, заслужений діяч науки і техніки України, кафедра технологій переробки плодів, овочів і молока, Харківський державний університет харчування та торгівлі. Адреса: вул. Клочківська, 333, м. Харків, Україна, 61051. E-mail: ktppom@mail.ru.

Павлюк Раиса Юрьевна, д-р техн. наук, проф., лауреат Государственной премии Украины, заслуженный деятель науки и техники Украины, кафедра технологий переработки плодов, овощей и молока, Харьковский государственный университет питания и торговли. Адрес: ул. Клочковская, 333, г. Харьков, Украина, 61051. E-mail: ktppom@mail.ru.

Pavlyuk Raisa, doctor of technical sciences, professor, the State Prize laureate of Ukraine, Honored figure of Science and Technology in Ukraine, Department of Technology of fruits, vegetables and milk processing, Kharkov State University of Food Technology and Trade**.** Address: Klochkivska str., 333, Kharkov, Ukraine, 61051. E-mail: ktppom@mail.ru.

Погарська Вікторія Вадимівна, д-р техн. наук, проф., лауреат Державної премії України, кафедра технологій переробки плодів, овочів і молока, Харківський державний університет харчування та торгівлі. Адреса: вул. Клочківська, 333, м. Харків, Україна, 61051. E-mail: ktppom@mail.ru.

Погарская Виктория Вадимовна, д-р техн. наук, проф., лауреат Государственной премии Украины, кафедра технологий переработки плодов, овощей и молока, Харьковский государственный университет питания и торговли. Адрес: ул. Клочковская, 333, г. Харьков, Украина, 61051. E-mail: ktppom@mail.ru.

Pogarskaya Viktoriya, doctor of technical sciences, professor, the State Prize laureate of Ukraine, Department of Technology of fruits, vegetables and milk processing, Kharkov State University of Food Technology and Trade. Address: Klochkivska str., 333, Kharkov, Ukraine, 61051. E-mail: ktppom@mail.ru.

Бессараб Олександр Семенович, канд. техн. наук, проф., заслужений працівник освіти України, кафедра технології консервування, Національний університет харчових технологій**.** Адреса: вул. Володимирська, 68, м. Київ, Україна, 01601. E-mail: tk_nuft@i.ua.

Бессараб Александр Семенович, канд. техн. наук, проф., заслуженный работник образования Украины, кафедра технологии консервирования, Национальный университет пищевых технологий. Адрес: ул. Владимирская, 68, г. Киев, Украина, 01601. E-mail: tk_nuft@i.ua.

Bessarab Alexandr, candidate of technical sciences, professor, Honored Worker of education in Ukraine, Department of Technology preservation, National University of Food Technologies. Address: Volodimirovska str., 68, Kyiv, Ukraine, 01601. E-mail: tk_nuft@i.ua.

Борисова Аліна Олексіївна, доц., кафедра іноземних мов, Харківський державний університет харчування та торгівлі. Адреса: вул. Клочківська, 333, м. Харків, Україна, 61051. E-mail: inter-dep62@mail.ru.

Борисова Алина Алексеевна, доц., кафедра иностранных языков, Харьковский государственный университет питания и торговли. Адрес: ул. Клочковская, 333, г. Харьков, Украина, 61051. E-mail: inter-dep62@mail.ru.

Borysova Alina, docent, Department of foreign languages, Kharkov State University of Food Technology and Trade. Address: Klochkivska str., 333, Kharkov, Ukraine, 61051. E-mail: inter-dep62@mail.ru.

Балабай Катерина Сергіївна, асист., кафедра технологій переробки плодів, овочів і молока, Харківський державний університет харчування та торгівлі. Адреса: вул. Клочківська, 333, м. Харків, Україна, 61051. E-mail: ktppom@mail.ru.

Балабай Екатерина Сергеевна, асист., кафедра технологий переработки плодов, овощей и молока, Харьковский государственный университет питания и торговли. Адрес: ул. Клочковская, 333, г. Харьков, Украина, 61051. E-mail: ktppom@mail.ru.

Balabai Katerina, assistant, Department of Technology of fruits, vegetables and milk processing, Kharkov State University of Food Technology and Trade. Address: Klochkivska str., 333, Kharkov, Ukraine, 61051. E-mail: ktppom@mail.ru.

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