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SOME TECHNOLOGICAL PECULIARITIES OF CO_2 – EXTRACTS OBTAINED FROM VEGETATIVE AND ANIMAL RAW MATERIAL

The carbon dioxide in subcritical and supercritical states is used to extract valuable components from vegetative and animal raw material. The unique properties of this solvent in subcritical make it possible to extract selectively a complex of flavour and gustative substances with a comparatively small molecular mass. Proteins, carbohydrates and components with a bigger molecular mass can not be extracted in this state. New schemes of supercritical installations which make it possible to extract the valuable components not only from both dry and from moist raw material have been worked out.

Keywords: subcritical extraction, CO_2 – extracts, supercritical extraction, carbon dioxide, chemical content, extraction installations.

Углекислота в подкритических и суперкритических государствах используется, чтобы извлечь ценные компоненты из вегетативного и животного сырого материала. Уникальные свойства этого растворителя при определенных условиях делают возможным извлечение выборочно комплекса аромата и вкусовых субстанций со сравнительно маленькими молекулярными массами. Белки, углеводы и компоненты с большей молекулярной массой не извлекаются. Представлены новые схемы суперкритических установок, которые делают возможным извлечение ценных компонентов не только из высушенного, но и из влажного сырого материала.

Ключевые слова: подкритическая вытяжка, CO2 - выдержки, суперкритическая вытяжка, углекислота, химическое содержимое, экстракционные установки.

INTRODUCTION

Sub- and supercritical fluidic extraction of components from vegetative and animal raw material is an area which considers the nature and regularities of liquefied and suppressed gases movement in selfcontained contour and is a part of continuous media mechanics. It has been formed in the past years as an independent section of applied physical chemistry and has integrated the achievements of gas dynamics, hydraulics and high temperature chemistry.

At present the extraction methods of obtaining aroma and gustative substances from vegetative raw material have been widely applied. The use of extracts is more effective. The well-known periodic and continuous extraction installations work at pressure close to atmospheric and use carbohydrates, spirits, ethers and ketons as a solvent. The disadvantage of this method and installations is that extraction by organic solvents does not always provide a complete withdrawal of aromatic and gustative substances. Besides, when the solvent is evaporated thermolabile substances of extracts are destroyed. It may be avoided when extraction of aromatic components from vegetative raw material is made by liquefied gases - argon, butane, propane, liquefied carbon dioxide, khladons etc. In this case the pressure in extraction installations may significantly exceed the atmospheric pressure, however, the process of extraction is carried out at the temperature of the environment, sparing termolabile components and produce high quality extracts.

Traditional and innovative methods of extraction, properties of extracts and their application are of great interest for the greate number of food industry specialists. However, a lot of data that are important for practical application available in a broad variety of sources have not been systematized. This article is aimed to fill, in some degree, the saps in production technology and application of CO_2 – extracts in food industry.

In Russia research in the area of volatile oil bearing and spicy-aromatic extracts production and application technology with the use of Carbon Dioxide as a solvent started in the sixties of the twentieth century.

It has been shown, that the most effective method of extracting valuable components from spicy and aromatic raw material consists in application of liquefied and suppressed gases and liquids, overheated in comparison with the environment. The liquefied carbon dioxide has become dominant in food industry as a solvent.

As a liquid carbon dioxide can exist at pressure from 73,8 * 10^2 (critical pressure) up to 5,18 * 10^2 kPa (triple point) and corresponding temperatures from +31,1 up to – 56,6 ^{0}C.

When this solvent is applied a most complete extraction of ether oils and other aromatic and gustative substances is achieved and most of disadvantages, typical of organic solvent extraction and evaporation are removed.

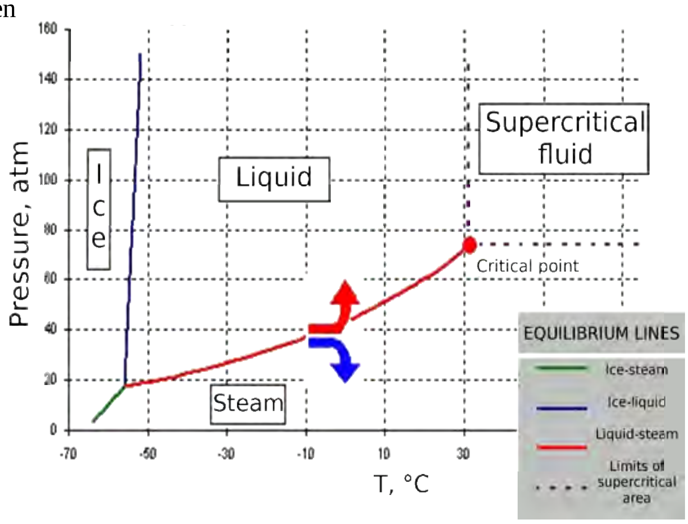

Fig. 1. The diagram of CO_2 phase state

Fig. 1 shows the diagram of CO_2 phase state.

During the process of extraction it is necessary to maintain process temperature to achieve a more complete extraction during the working period.

The strict control of temperature is necessary at distillation as the thermolabile substances are destructed at high temperatures and the appearance of extracts

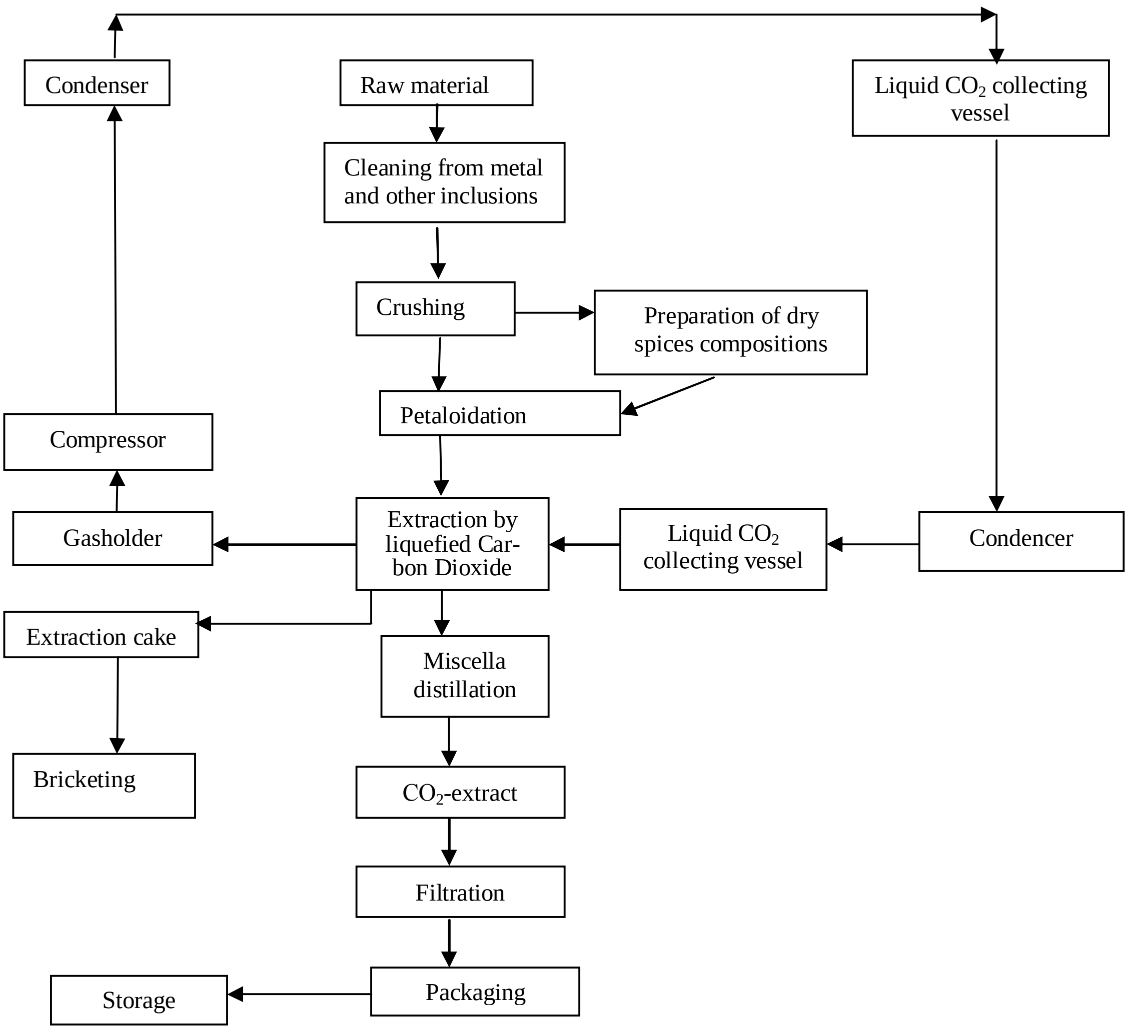

Fig. 2. The blow sheet of CO_2 – extracts production

deteriorates. The most acceptable temperatures of miscella distillation process are 40-500C. At these temperatures the extracts keep nativity and have organoleptic and biological properties of the original raw material. Subcritical CO2 – extraction is carried out at a sparing mode and is ecologically clean. The absence of strong mineral acids in technological process makes it possible to do without any water purifying installations.

The blow sheet of CO_2 – extract production is given in fig. 2.

The content of extractive substances in vegetative raw material is determined by exhausting extraction using the installation given in fig. 3.

Nowadays the express method of extractive substances determination by using liquefied gases as solvent has been worked out. This method allows to cut the time of analysis significantly.

The preliminary treatment of the air-dry raw material is very important for the production of extracts for various purposes. The preparation of vegetative raw material is necessary to carry out the extraction process at optimal modes that include the degree of the raw material crushing up to the sizes from 100 to 500 mkm, depending on the kind of the raw material, with consequent flatten on the roll machine tool to make a petal of 150 up to 300 mkm.

Commercial application of CO_2 – extracts makes possible to receive the products of higher quality.

A market analysis has shown that there is a significant demand for CO_2 – extracts among food enterprises, pharmaceutical enterprises, biologically active food supplement producers, perfumery-cosmetic manufactures.

Any extract is practically a replica of initial vegetative raw material and is not analogous to traditional extracts. A chromatographic analysis has shown that the content of valuable substances of CO_2 - extracts is ten times bigger than traditional ones. Now production of extracts from spices and medicinal herbs; fruit aroma; α & β – acids from hop; antioxidants, carotenoids and licopins (including these tomato raw material); natural colouring substances (from red pepper); lanolin from wool; natural vegetative wax; sea buckthorn oil; dogrose; ginseng has been implemented on industrial scale. Technology and equipment for extraction of vegetative raw by liquefied gases has attracted more and more attention.

In the course of research a laboratory installation has been made to study the extraction kinetics.

The laboratory installation (figure 3) is used to determine extractive substances. It consists of an extractor (6) and miscella collectors (1). The extractor and miscella collectors are placed in the main body (3) equipped with a hatch (4) and a viewing window (2).

The miscella collectors are placed on a platform (8) that can rotate periodically in such a way as to place under the extractor tube only one miscella collector.

To control the pressure the installation is equipped with a manometer. The lower part of the extractor is heated by an electric heater and the upper part is cooled down by cold water or antifreeze. A feeding pipeline to supply carbon dioxide is also designed.

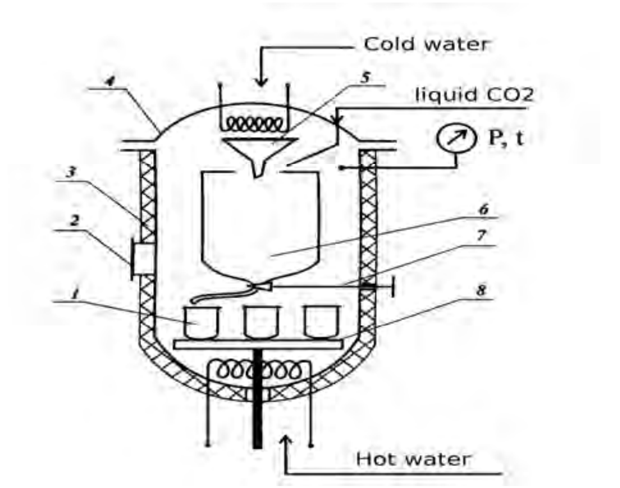

Fig. 3. A drawing of a laboratory installation for CO_2 – extract production: 1 – vessel for miscella, 2 – viewing window, 3 – extractor, 4 – selfpacking manhole, 5 – funnel for condensate, 6 - glass extractor with raw material, 7 – handle manipulator, 8 – rotary platform

In Table 1 the modes of CO_2 – extraction are given.

A drawing of the subcritical commercial installation used in the extraction plant of the Karavan Co. is given in fig. 4

The research into quantitative and qualitative content of food products and CO_2 – extracts in particular, by method of IR-spectroscopy, in combination with other methods, has made it possible not only to cut the analysis time, but also to control the technological process with sufficient accuracy.

The application of supercritical carbon dioxide has some advantages: energy saving process; high mass exchange characteristic due to low viscosity and high penetration capacity of the solvent; high degree of extraction of components, high quality of product, the absence of CO_2 in the final product, use of inert solution media at temperature preventing thermal degradation, absence of wastes, recycling (the CO_2 after decompression can be collected and reused), the unique microbiological cleanness, absence of multistage process.

Supercritical carbon dioxide (SC-CO_2) is a good solvent for non-polar and mid-polar substances and can extract substances with molecular mass up to 2000 and more dalton with high selectivity. The regulation of thermodynamic conditions allows to change significantly the physical properties of extragent (density, diffusion capacity etc.) and the selectivity of component extraction from mixes of organic substances. Extraction by CO_2 in supercritical state allows to avoid the undesirable influence of oxygen and organic solvents, to remove unnecessary substances and enrich ether oil by volatile aromatic substances. In Russia SC- CO_2 – extraction is implemented in Krasnodar (Kuban State University of Technology), in Rostov (the STC “Goro”), in Kasan (the Kasan State Technological University), in Moskow (Moskow State University), Tomsk (the “Siberian plant of extracts and biotechnologies Ltd. co”) and other cities.

Table 1

Technological modes of CO_2 – extraction and vegetative raw material characteristics

Raw material	Sheet thickness, мм	Density, g/dm^3	Extraction time, min	Output of extract, %
1	2	3	4	5
Sweetflag	0,16-0,20	320	90	5,0-6,0
Anise	0,14-0,18	350	90	4.0-4,5
Anise-tree	0,12—0,16	400	90	9,0—11,0
Basil	0,14—0,18	210	145	2 0—2,5
Grape seeds	0,18—0,20	380	70	4,0-4,8
Pink	0,16-0,20	400	90	18,0-20,0
Jointweed	0,10-0,10	190	120	2,5-3.0
St. John’s wort	0,14-0,18	280	120	2,5-3,0
Sweet-grass	0,40	250	145	1,5-2,0
Ginger	0,12-0,16	450	90	3,5-4,0.
Calendula	0,12-0,16	180	100	1,7-2,3.
Inula	0,12-0,16	300	120	4,0-5,O
Angelica	0,16-0,18	300	130	2,5-3,0.
Cardamom	0,12-0,16	260	90 120	5,0—7,0
Cinnamon	0,12-0.16	350		2,0-3,0.
Nettle	0,25	210	84	3,2-3,8
Coriander	0,15-0,18	260	120	3,0.-3,5
Laurel	0,8	250	100	2,5-3,0
Curcuma	0,18-0,20	420	40	3,0-3,0
Juniper	0,14-0,18	320	120	2,0-3,0
Carrot (seeds)	0,12-0,16	280	120	3,0-4,0
Muscat	0,16-0,18	100	80	10,0-12,0
Red pepper	0,12-0,16	200	120	6,0-5,0
Black pepper	0,14-0,18	300	I80	6,5-7,5
Fragrant pepper	0,12—0,16	400	90	4,5-5,0
Parsley	0,12-0,16	350	90	4,0-5,0
Sage-brush	0,18-0,26	220	60	3,0-3,6
Barley sprouts	0,10-0.12	280	120	0.8-1,2
Chamomile	0,12-0.16	260	210	2.0-5,0
Selery	0,12-0.16	260	120	5,5-7,0
Yarrow	0,12-0,16	200	120	1,2-1,7
Caraway seeds	0,12-0,14	350	120	5,0-7,0
Fennel	0,12-0,16	- 350	90	3,5-4,5
Fennel (seeds)	0,12-0,16	280	120	5,0-7,0
Fir needle	0,76	360	95	5.0-6.8
Hop	0,5	220	120	8,0-10,0
Sage	0,3	220	120	4,0
Tarragon	0,10-0.16	200	120	3,8

SC-CO_2 is a good solvent for volatile aromatic substances of rose oil. The produced SC-CO_2- extracts were saturated by minor components, enriching the content and depth of rose oil aromatic spectrum. It is especially explicit in comparison with the traditionally obtained rose oil.

The variation of extraction thermodynamic conditions makes it possible to change the ratio between the components in the extracted products. At the increase of pressure from 13,0 to 18,0 the extracts are enriched by more “heavy” components (nerol, geraniol, citronellol, elemol, hexadecane etc.), meanwhile the relative content of more volatile components is diminished with growth of pressure (benzaldehyde, linalool etc.). The undesirable changes in the range of natural aromatic substances do not take place.

MATERIALS AND METHODS

A research on CO_2 – extract is aimed at determining

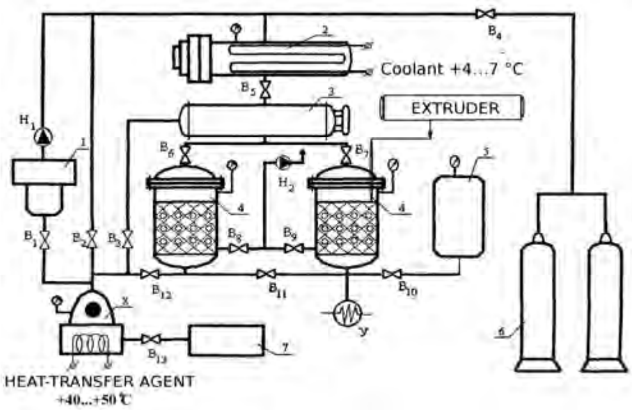

Fig. 4. The drawing of a commercial subcritical extraction installation

its naturalness, the absence of admixtures and number of parameters - density, angle of rotation, acid number, ethereal number before and after acetylation.

The naturalness of the extract is determined analyzing colour, smell and taste.

Colour and transparency are determined by placing 10 ml of the extract into a cylinder of transparent colourless glass with a diameter of 2-3 cm and observing in the light.

The smell is determined in the following way: 0,1 ml (two drops) of the extract are placed on the ribbon of filtering paper of 12 cm long and 5 cm wide avoiding the edges of the paper and then compare the smell of the test sample with the smell of the control sample placed in the same way on the filtering paper. During one hour the smell must be similar to the smell of the control sample.

The taste is determined by touching the filtering paper ribbon with the drop of the extract placed on it or with the mixture of 1 g of sugar powder with 1 drop of the extract with the tongue.

The content of water is determined by means of distillation. 1-2 ml of the extract is placed on the test glass and heated up to 120^0C. If there is some water the glass is misted up. The amount of water can be determined by evaporating 10 ml of the extract at 120^0C with a cooler. After the water has ceased to evaporate (the steam thermometer shows less than 100^0C) it is weighed.

The density of the extract is determined by a picnometer. To determine this parameter the sign *d* is used, indicating the conditions, usually 20^0C, related to water density at 4^0C: d_4^{20}.

The angle of polarization plane rotation is defined by polarimeter. Depending on the nature of the substance the rotation of polarization plane may have the various directions and quantity.

For the ethereous oil of *Mentha piperita* (pepper mint) the angle of rotation must be no less than – 18^0C, for ethereous oil of eucalypt – from 0 up to + 10^0C.

The refraction parameter is determined in a refractometer.

For the ethereous oil of *Mentha piperita* (pepper mint) the refraction parameter must be 1.459-1.470, for ethereous oil of eucalypt – 1.458-1.470.

The temperature of solidification is determined in a special device consisting of a vessel of a cooling mixture where the test glass is placed in. The height of the extract layer must be no less than 5 cm. The highest temperature that remains for a short time constant since the moment of substance solidification is determined by a thermometer and defined as solidification temperature.

Acid number is determined by a standard methodic using KOH and phenolphthalein.

Ethereous number is determined in the solution obtained after determination of acid number by heating the test glass with KOH using air cooling and subsequent titration with H_2SO_4 (indicator - phenolphthalein)

Etherous number (E_n) after acetylation is determined by boiling the extract in the presence of acetic anhydride and anhydrous sodium acetate and subsequent washing with NaCl solution and water.

The content of complicated ethers or bound spirits X_1 is calculated by equation:

$$X_1 = \frac{E_n * M}{561}$$

where *M* – the molecular mass of ether or spirit

The content of phenols is determined by using NaOH solution.

Division and analysis of extracts into classes of chemical substances are carried out according to the diagram given in Fig. 5.

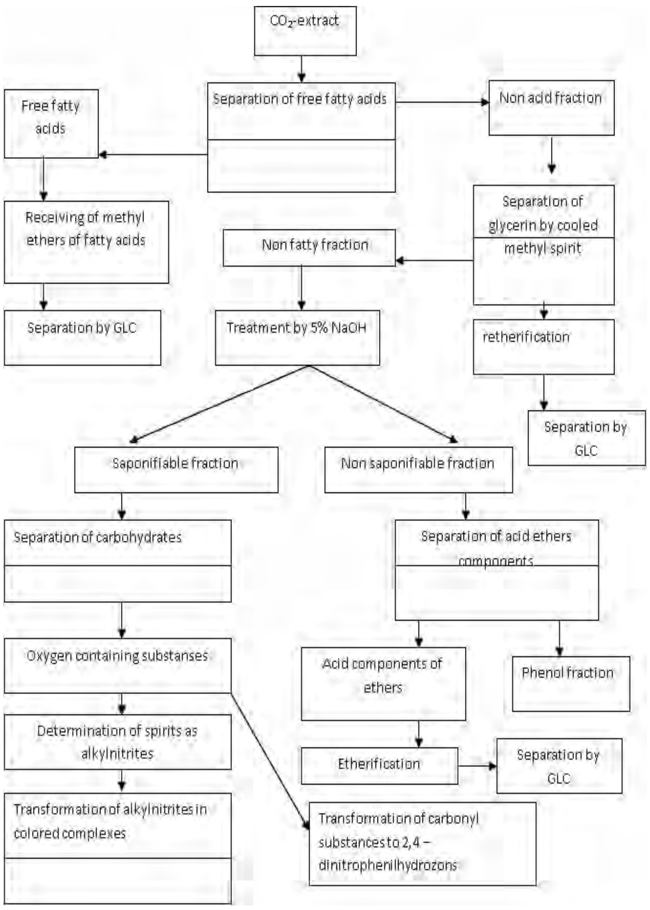

Fig. 5. The diagram of CO_2 – extracts division into classes of organic substances

RESULTS AND DISCUSSION

The researches on physical and chemical parameters of CO2 – extracts conducted at the experimental installation have been the base of making technical documentation for CO2 – extracts.

Documentation determines the production of CO_2 – extracts obtained both from separate spicy raw material and from a mix of dry spices, used for aromatization of various products.

Most of extracts from spicy and aroma vegetative raw material are oily liquid of yellow, green, brown color with more dark or light shades.

The qualitative content of CO_2 – extracts from domestic spices is given in Tab. 2.

The physical and chemical parameters of some main components of CO_2 – extracts are given in Tab. 3.

Table 2

The qualitative content of CO_2 – extracts from domestic raw material

Extracts	Oily substances	Titrable acids	Non-fat fraction						Other sub-stan-ces
			Saponifiable substances		Non-saponifiable substances				
			Total	Phenols	Total	Carbonyl substan-ces	Spirits	carbohydrates	
Anise	33,30	7,35	7,07	0,9	52,30	-	-	20,70	
Sweetflag root	1,10	0,80	16,20	4,6	82,00	5,3	66, 60	10,10	-
Basil	8,90	8,90	29,20	20, 3	34,80	-	33, 00	1,80	18,2
Angelica	7,32	2,00	50,20	6,2	40,50	13,20	15, 70	11,60	-
Sweet-grass	34,10	0.40	40,00	3,4	25,50	3,90	20, 23	1,37	-
Galangal	11,17	0,60	48,20	12,0	40,00	3,40	32, 60	4,10	-
Coriander	61,21	1,10	2,17	0,4	34,38	3,10	27,22	4,02	-
Laurel leaf	22,16	2,40	27,80	9,2	47,60	6,70	18,56	22,34	-
Juniper berry	27,90	11,20	43,00	10,6	29,10	13,80	15,20	0,10	-
Red pepper	69,00	0,80	15,10	2,4	15,10	3,60	9,33	1,17	-
Caraway seeds	36,20	0.84	19,10	5,0	44,70	30,20	14,50	-	-
Sage	12,80	17,80	22,70	8,3	31,50	13,00	18,50		15,2

Table 3

Physical and chemical parameters of some main components of CO_2 – extracts (at 20^0C)

Components	Appearance	Molecular mass	Temperature, °C		Density, kg/м³	Refraction coefficient
			Boiling	Melting		
Apiole $C_{12}H_{14}O$	Crystals look like long colourless needles with gentle smell of parsley and chilly taste	222,20	292	28,0—30,0	1,1788	1,5380
Anetole $C_{10}H_{12}O$	White crystal mass with the smell of anise	148 ,20	233—234	22,5—23,0	0,9840-0,9860	1,5590-1,5610
d -Borneole $C_{10}H_{18}O$	White crystal product with the smell of camphor or ambra	154,20	214	207,8—209,3	1,0110	
d -Karvone $C_{10}H_{14}O$	Colorless liquid with the smell of omum plant	150,21	224-225	—	0,9659	1,4949
Cinnamun aldehyde C_5H_8O (β-phenilacroleine)	Transparent liquid with the smell of cinnamun	132,10	252	-	1,0520	1,6194
Coumarin $C_9H_60_2$	Colorless crystals with the smell of fresh hay	146,14	301	71,0	1 ,0148	—
Linalool $C_{10}H_{18}O$	Colorless transparent liquid with the smell of lily	154,2.	I85— 199	-	0,8607	1 ,4614 1
1,8-Cineole $C_{10}H_{18}O$	Colorless syrup like oil with the smell of camphor, taste is cooling	154,2	177—178	1,0-1,5	0,9262	1,4616
Azarone $C_{12}H_{10}O_3$	Crystals without smell and taste	208,2	296	62—63	1 ,0910	1,571-9
eugenol $C_{10}H_{12}O_2$	Transparent liquid of yellow color with strong smell of clove	164.2	250—255	-	1 ,0630	1,5445

components.

The substitution of dry spicy substances by the analogous CO_2 – extracts in various ratios provides high quality of tomato sauces, vegetable and fruit marinades, meat, vegetable and fish snack canned products, sausages, meat semifinished products. CO_2 – extracts have already found a broad application in public catering and home culinary products.

Using the methods of system analysis, a conception

CONCLUSIONS

It has been determined that sub- and supercritical CO_2 – extracts from spicy-aromatic, medicinal, volatile oil bearing vegetative raw material are absolutely similar to the native aroma and taste of plants. The content of CO_2 – extracts is represented by volatile oils, vitamins (E, F, C), provitamins (A, D), phytohormones, phytoncides, cumarins, alkaloids, tarry substances, waxes and other

of a new scientific direction – the uniform system of carbon dioxide application in food industry for creation of principally new high technologies of CO_2 – treatment of agricultural raw material has been formulated.

1. Peculiarities of interaction, functioning and development of technological processes based on interaction of food products with carbon dioxide in stable or changing phase states that made it possible to design the scien-

tific bases of engineering solutions in the area of technique and technology of CO_2 – processing have been revealed.

2. The full-scale chemical and biological researches on more than 100 kinds of domestic and imported fruit-vegetable, spicy, aromatic, oily and other kinds of raw material, including nontraditional kinds of raw material and byproducts of food raw material have been carried out, the adaptability of this raw material for industrial application has been revealed, the raw material most suitable for the CO_2 – technology has been tested.

3. The scientific basis for new areas of food and cooling techniques, such as selective extraction and line-spray treatment in gasdynamic chillers has been elaborated and the peculiarities of substance crystallization in the "CO_2 – component" complex systems have been revealed.

4. Using the methods of system analysis, a conception of a new scientific direction in the application of CO_2 – technologies in food industry for processing various kinds of food raw material has been formulated.

5. The scientific basis of selective extraction has been worked out and the conditions of "coextraction" effect have been described.

6. The combined methods of process thermodynamic effectiveness analysis (directed line spray crystallization, selective extraction etc.) have been worked out.

7. The methodology of selecting and determining the sequence of designing have been worked out to estimate the interdependent mode and technological characteristics of the new equipment for CO_2 – extraction.

8. The generalized approaches to the processes have been worked out, which made it possible to find out the interactive factors in the development of new technological solutions, to formulate the directions of scientific researches and to sum up the ways of the solving problems.

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Одеська національна академія харчових технологій

ВПЛИВ ЕЛЕКТРОМАГНІТНОГО ОБРОБЛЕННЯ НА МОЛОЧНОКИСЛІ БАКТЕРІЇ

Показано вплив електромагнітного оброблення молока на молочнокислі бактерії *Lactobacillus acidophilus* та *Lactobacillus plantarum*. Встановлено, що даний вид оброблення не пригнічує розвиток корисної мікрофлори, а при силі струму 0,6 А (93 кА/м) і 1 А (165 кА/м) навіть виявляє незначну стимулюючу дію.

Ключові слова: електромагнітне оброблення, молочнокислі бактерії, накопичення біомаси, ліпіди молока.

It is showed influence of electromagnetic treatment on lactobacilluss of *Lactobacillus acidophilus* and *Lactobacillus plantarum.* It is set that the type of treatment is given not represses development of useful microflora, but at strength of current 0,6A (93 kA/m) and1A (165 kA/m) even finds out an insignificant stimulant an action in relation to some lactobacillus.

Keywords: electromagnetic treatment, lactobacillus, biomass accumulation, milk fats.

Людина і середовище, що її оточує, знаходяться у постійній біологічній рівновазі. Загальновідомо, що здоров'я людини значною мірою залежить від якості води та напоїв, рівня їх мінералізації та величини кристалогідратів.

Сьогодні однією із найважливіших проблем є вдосконалення методів оброблення води і харчових систем. Постає питання про необхідність впровадження нових способів і технологій.

Одним із найбільш реальних напрямів оброблення напоїв з метою поліпшення засвоєння мінеральних речовин є застосування електрофізичних методів. Розвиток цього напряму зумовлюється електричною природою продуктів споживання. Відомо, що найактивніше на електрично заряджені частинки можна впливати за допомогою електричних, магнітних і електромагнітних полів [1]. Електромагнітне оброблення застосовується в багатьох галузях промисловості з метою інтенсифікації технологічних процесів. Воно технологічне, просте в апаратурному оформленні, екологічне, не вимагає значних енергетичних витрат.

Відомо [1], що при електрофізичному обробленні використовують електромагнітні поля різної частоти, електричні поля високої напруженості, постійні і пульсуючі магнітні поля. Так, наприклад, в Національному університеті харчових технологій під керівництвом професора Українця А.І. проводились роботи по дослідженню і впровадженню у виробництво електроіскрових і магніто-імпульсних методів оброблення харчових продуктів, також в окремих випадках використовувались ультразвукові установки [2].

Всі перераховані чинники в реальних умовах діють на біологічні об'єкти в тому або іншому поєднанні спільно, і тому важко, а часом неможливо роздільно досліджувати процеси, що мають різну фізичну природу. Об'єктивною оцінкою впливу різних факторів на біологічні об'єкти може бути дослідження стійкості культур мікроорганізмів до їх дії.

Дослідники [2] вивчали вплив електроіскрового оброблення на дріжджі роду Saccharomyces cerevisiae; оцтовокислі бактерії Acetobacter aceti і молочнокислі