ECONOMICS COLLEGE IN STALOWA WOLA

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DEVELOPMENT OF MODELING THEORETICAL STUDIES ON FRACTIONATING DISTILLATION PARAMETERS OF NATURAL SOURCES OF AROMA

Frolova N. E., Yushchenko N. M.

INTRODUCTION

Formulation of the problem you ask: "What is aroma?" Then remind, how smell sweet cream or freshly baked fancy cakes at your place and you won't help the pleasant feeling of satisfaction and upraise. Aromatic fugitive fraction is a compound of air upon a nutrition product. During an inhale of such air aromatic substances interact with one or more man olfactory receptor and a man feels product's smell.

Ability to percept smell is connected with a deep biological nature of man[1]. Individuality of aromatic preferences is hidden not only in genetic code of a person, but also in upbringing, lifestyle[2].

A positive impact of aroma on a human body should be mentioned. Aroma calms, notifies, balances, relieves cramps, relieves pain and prevents inflammatory processes. Some aromas control processes of body's cellular generation and turnover and thanks to this stimulate biological processes in cells[3].

No matter how the theory of smell and the nature of its perception is interpreted, for food manufacturers, the problem of available sources of natural aromatic substances, the preservation of natural flavor in nutrition products remains important. Ecological changes, intensification of production reduce a part of natural aromatic substances in our food. Furthermore purposeful decrease of content of fat, salt, sugar, increase of

[1] Reed D.R., Knaapila A. Genetics of taste and smell: poisons and pleasures. *Prog. Mol. Biol. Transl. Sci.* 2010. № 94. P. 213–240.

[2] Jeremy F. McRae, Sara R. Jaeger, Christina M. et al. Identification of Regions Associated with Variation in Sensitivity to Food-Related Odors in the Human Genome. *Current Biology.* 2013. Vol. 23. Issue 16. 19 August. P. 1596–1600.

[3] Influence of lemon essential oils composition on their antioxidant properties and stability of components / T.A. Misharina, M.B. Terenina, N.I. Krikunova, M.A. Kalinichenko. *Chemistry of vegetable raw materials.* 2010. Vol. 1. P. 87–92.

content of food fibers in the health-improving products also significantly changes taste and aroma of a product.

Existing production of food flavors in the country isn't able to meet the growing needs of interested producers. An average annual import of natural flavors into the Ukrainian market is estimated at more than 96%[4].

There are three crucial criteria of choosing a way of processing natural sources of aromatic substances (AS). Firstly, taking into account that a loss of AR even in the smallest quantities leads to a change in the aroma of a product[5]. Secondly, complete exclusion of chemical or enzymatic adverse reactions that may alter the natural composition[6]. And, thirdly, absence of toxic substances in industrial raw material and products of its processing.

Natural sources of aroma, first of all, include essential oils derived from domestic herbs. On the territory of Ukraine dominate coriander, mint, cumin, lavender, rose, sage, and fennel. During the last 20 years, Lemon Artemisia (Artemisia balchanorum Kpach.), catnip (Nepeta cataria), Moldavian dragonhead (Dracocephalum moldavicum L.) have been introduced to the industrial culture[7].

In recent years, in Ukraine, about 100–120 tons of essential oils are produced per year for a value of 60–70 million UAH. However, despite the significant experience of processing essential oils, most of them are not processed in the country and are exported to other countries. At the same

[4] Hasuha L.O. Contemporary content of food safety and its regional manifestation. URL: http://www.nbuv.gov.ua/portal/soc_gum/en_re/2011_8_1/85.pdf. 5 (Access date: 01.11.2018).

[5] Fractionation of Citrus Oils Using a Membrane-Based Extraction Process / D.J. Brose, M.B. Chidlaw, D.T. Friesen, Ed.D. Lachapelle, P. Eikeren. *Biotechnology progress*. 1995. Vol. 11. № 2. P. 214–220.

[6] Isolation and characterization of bioactive compounds from plant resources: The role of analysis in the ethnopharmacological approach / G. Brusotti, I. Cesari, A. Dentamaro, G. Caccialanza, G. Massolini. *Journal of pharmaceutical and biomedical analysis*. 2014. Vol. 87. P. 218–228.

[7] Frolova N.E., Silka I.M. Technology of natural flavors oriented on the domestic raw material and equipment base [Text]. *Collection of research papers "Bulletin of the NTU "HPI": New solutions in modern technologies*. 2015. № 46. URL: Bulletin of the NTU "HPI" (Access date: 01.11.2018).

time it returns to Ukraine in a form of thousands of items of flavors, pharmaceuticals, cosmetics, but at a price of 30–50 times higher[8].

More often Ukrainian essential oils are bought by the countries of the European Union, and in the first place France, England, Holland and Germany [4].

Topical and timely are theoretical and practical developments on excretion and concentration of a complex of aromatic substances from essential oils raw material and production of concentrated natural flavors of long-term storage.

1. Analysis of recent researches and publications

In the world to increase the range, natural sources of aromatic substances are subject to different processing. For this purpose, both traditional technologies that are well-proven, and new technological processes are used. In this analysis it is not possible to analyze a large variety of special methods of processing sources of aromatic substances, listed in modern scientific reviews[9]. However, in the analysis published by Mc Clements [10], it is stated that in the methods of processing sources of aromatic substances, described in 234 randomly selected works, 78% primary use distillation. In other cases it is extraction. At the same time, in many cases, extraction is only a preliminary stage for which distillation is used. According to a well-known scientist, researcher and practitioner A. Kiss, the technology of distillation is still young and full of opportunities for breakthrough[10].

[8] Kasyanenko M.K. Reviving the aroma industry. Den. 2009. 13 August. URL: http: //www.day.kiev.ua/uk/article/ekonomika/vidrodzhuyuchi-industriyu-aromativ (Access date: 01.11.2018).

[9] Hüsnü Can Baser K., Buchbauer Gerhard. Handbook of essential oils: science, technology, and applications. Boca Raton: CRC Press. 2010. 843 p.

Kiss A.A. Advanced distillation technologies: design, control and applications. John Wiley & Sons. 2013. 416 p.

McClements D.J. Food emulsions: principles, practices, and techniques. CRC press. 2015. 690 p.

[10] Kiss A.A. Distillation technology–still young and full of breakthrough opportunities. *Journal of Chemical Technology and Biotechnology*. 2014. Vol. 89. №4. P. 479–498.

Along with distillation, selective adsorption is common[11], fractional extraction and condensation[12], ultrasound, microwaves treatment[13], turbohydrodistillation[14], steam- and hydro- distillation[15], extraction of the aromatic complex by preparative chromatography[16]. Selective fractionation through inclusion in deoxycholic acid is proposed[17].

For obtaining fractions liquefied gases are successfully used – hydrocarbon (Ca), chladones (halogenated hydrocarbons C2, C4), that boil at temperatures below 0 C and do not require special heating during evaporation[18]. Fractionation with supercritical carbon dioxide allows obtaining aromatic products at low temperatures[19].

Under the classical laws of distillation, when the complex mixture is heated to boiling, components whose elasticity of steam is greater than of the others, will first of all transfer into a steam phase. Fractional distillation is essentially the opposite of distillation Through fractional distillation, the processes of evaporation and condensation are repeated many times, and

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[11] Essential oil terpeneless by extraction using organic solvents or ionic liquids / A. Arce, A. Marchiaro, O. Rodriguez, A. Soto. *AIChE journal*. 2006. Vol. 52. № 6. P. 2089–2097.

[12] Ascending bubble extraction of terpenes from freshly squeezed orange juice / P. Komthong et al. *Food research international*. 2006. Vol. 39. № 1. P. 53–58.

[13] Valorization of citrus by-products using Microwave Steam Distillaton (MSD) / N. Sahraoui, M.A. Vian, E.V. Maataoui, C. Boutekedjiret, F. Chemat. *Innovative Food Science & Emerging Technologies*. 2011. Vol. 12. № 2. P. 163–170.

[14] Process for he rectification of mixtures of high-boiling air and temperature-sensitive useful products: Patent № US 8747621 B2. Date of Patent: Jun. 10, 2014. URL: https://patents.google.com/patent/US8747621/de. (Access date: 01.11.2018).

[15] Selection and evaluation of adsorbents for capturing aromatic substances during concentration of juices and extracts / N.E. Frolova, A.I. Ukrainets, V.O. Usenko, K.A. Naumenko. *Scientific paper of NUFT*. 2010. № 33. P. 68–72.

Stichlmair J. Distillation and Rectification. Ullmann's *Encyclopedia of Industrial Chemistry*. 2010. P. 456–484.

[16] Tzamtzis N.E., Liodakis S.E., Parissakis G.K. The deterpenation of orange and lemon oils using preparative adsorption chromatography. *Flavour and Fragrance Journal*. 1990. Vol. 5. № 1. P. 57–67.

[17] Fantin G., Fogagnolo M., Maietti S., & Rossetti S. Selective removal of monoterpenes from bergamot oil by inclusion in deoxycholic acid. *Journal of agricultural and food chemistry*. 2010. № 58 (9). P. 5438–5443.

[18] Gironi F., Maschietti M. Continuous countercurrent deterpenation of lemon essential oil by means of supercritical carbon dioxide: Experimental data and process modeling. *Chemical Engineering Science* 2008. Vol. 63. № 3. P. 651–661.

[19] Hu Z. Fractionation of Ligusticum Chuanxiong by Adsorption in Supercritical Carbon Dioxide. *Industrial & Engineering Chemistry Research*. 2012. Vol. 51. № 44. P. 14496–14502.

distillate every time becomes a starting material for the next process. As a result, even low-boiling components condense[20].

Fractional distillation is a long process, even for binary separation. For bigger quantity of components there are more difficulties that can be eliminated by distillation in the column for precise fractionation[21]. As a result of fractional distillation a number of separated fractions, which boil in the narrow temperature limits, are formed[22].

Fractional distillation of natural sources of aromatic substances is complicated by the multiple component composition, with wide limits of individual boiling temperatures (T_{boil}), as well as the possibility of chemical modification.

To the main governing parameters of fractional distillation belong operating temperature and pressure[23].Data on dependence of these governing parameters is limited. Fractional distillation is suggested to be carried out at close to T_{boil} of essential oils components. Herewith, the difference in T_{boil} of components in a vacuum can be much higher than under atmospheric pressure. According to[24], a significant amount of substances that boil at atmospheric pressure at a temperature of 250°C and above with decomposition are dispersed without changing the composition at a pressure of 1.33 kPa and a temperature of 160 ... 210°C, or within the temperature range from 100 to 130°C at a pressure of 0.00133 kPa, or at temperatures from 40 to 60°C in a vacuum. At the same time, there are some difficulties connected to lowering the pressure of the process. First of all tangible fluctuations of temperature within the limits of 10 ... 15°C at the beginning

[20] Distillation applied to the processing of spirits and aromas / A.J. Meirelles, A.C. Batista Eduardo, F.A. Scanavini Helena, R.M. Batista Fábio, Ceriani Roberta, F.L. Luz Luiz. *Extracting Bioactive Compounds: Theory and Applications*. 2008. P. 75–136.

[21] Feoktistov D.V. Experimental researches of an efficiency of the distillation process of binary substances. *Industrial energetics*. 2013. № 10. P. 35–39.

[22] Stichlmair J. Distillation and Rectification. *Ullmann's Encyclopedia of Industrial Chemistry*. 2010. P. 456–484.

[23] Sidorov Y.I., Chuyesho V.I. Processes and equipment of the chemical and pharmaceutical industry: manual for farm. and chem. specialized Universities. Kyiv: *Nova Knyha*, 2009. 290 p.

[24] Gryta M., Waszak M. Application of vacuum membrane distillation for concentration of organic solutions. *Chemical Papers*. 2016. Vol. 70. № 6. P. 737–746.

of distillation, decrease of the general speed of mass transfer and, accordingly, increase of distillation time[25].

From modern scientific publications on the methods of fractional distillation of aromatic substances sources informative is the distillation of essential oil of Italian mandarin into fraction. The process is realized in a column (30x2.5 cm) with receipt of 4 fractions: saturated and unsaturated monoterpenes, oxygen-containing substances, mixture of nitrogen-containing substances with terpenes. The most unstable fraction of unsaturated monofilaments is removed. All other fractions are combined and used as a persistent flavor of an orange smell[26]. Fractional distillation of lavandin allows extract three fractions[27] [28]. The first fraction contains 50% of terpenes, in addition 18…20% of rosoxide, which has a distinctive geranic smell and 17% of menthol and isomenthone, which have a mint aroma. The second fraction − carbonyl-oxide consists of 25% of terpenes, 30% of rosoxide, 25% of menthol and isomenthone, as well as 20% of linalool, which contributes to the general flavor a lily of the valley aroma. Composition of the carbonyl (third) fraction consists of 10% terpene hydrocarbons, 10% rosoxide, 40 x 50% menthol and isomenthone, 10 x 20% linalool, geraniol and citronellol with distinctive rose aroma. Out of carbonyl fraction isomenthone oxime, which degrades the aromatic properties of this fraction is exerted. The obtained fractions are natural flavors.

Fractional distillation is also used to obtain a water essence of citrus oils with high content of aromatic components and low content of terpenes[28].

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[25] Wiles S. Phase equilibrium in chemical technology: in 2 v. / trans. from English A.B. Bespalovaya, ed. C. Beskov. Moskow: Mir, 1989. 304 p.

[26] Fractionation of citrus essential oil using liquid–liquid extraction: Equilibrium data for model and real systems at 298.2 K / D. Gonçalves, C.C. Koshima, M.E. Teschke, C.E.C. Rodrigues. *Fluid Phase Equilibria*. 2015. Vol. 399. P. 87–97.

[27] A comparison of essential oils obtained from lavandin via different extraction processes: ultrasound, microwave, turbohydrodistillation, steam and hydrodistillation / S. Périno-Issartier, C. Ginies, G. Cravotto, F. Chemat. *Journal of Chromatography*. 2013. Vol. 1305. P. 41–47.

[28] Ascending bubble extraction of terpenes from freshly squeezed orange juice / P. Kornthong, T. Katoh, N. Igura, M. Shimoda et al. *Food research international*. 2006. Vol. 39. № 1. P. 53–58.

The analysis of recent researches and publications has indicated narrowness of data on theoretical modeling of the influencing parameters of fractional distillation of natural aroma sources.

The aim of the submitted paper is representation of scientific researches on the development of theoretical modeling of parameters of controlled essential oils distillation into fractions of given aromatic characteristics.

2. Present of the main material

The peculiarity of classical essential oil production is that fresh essential oil (EO) may not be immediately realized, as it does not comply with international regulatory documentation. It takes extra time and operations after which oil becomes marketable. This increases material production costs.

A viable alternative to the processing essential oils of the original quality is the controlled distillation into fractions. As it is a physical process, the resulting fractions are natural flavors. As a result, from a single essential oil a series of natural flavors of different flavors may be obtained.

The operating scheme of modeling included:

1. Determination of valid T_{boil} of EO components under atmospheric pressure.

2. Establishing the equilibrium distillation pressures of an EO and relative fugitiveness of α as the ratio of pressures at constant temperature.

3. Construction of the equilibrium curves of composition of the liquid and steam Y-X for the key components of EO.

4. Calculation of a minimum number of theoretically perfect plates and operationing reflux ratio of EO distillation.

Scientific researches were carried out on options of obtaining the specified parameters through classical works on distillation of complex mixtures[29], modern foreign publications[30], and original domestic articles[31].

[29] Optimization of alcohol distillation process / Y.V. Buliy, P.L. Shiyan, A.P. Dmytruk, A.M. Kuts. *Chemistry and technology of food*. Kaunas: PICTU. 2015. V. 49. № 1. P. 20–28.

Fomikhin O.V., Leontiev S.A., Galiciev R.M. Calculations of the constants of phase equilibrium. SPb: Nadra, 2010. 105 p.

[30] Lai Y., Dilidaer M. D., Chen B. et al. In vitro studies of a distillate of rectified essential oils. *American journal of rhisology & allergy*. 2014. Vol. 28. №. 3. P. 244–248.

[31] Korchagin V.I., Agapov M.A. On the issue of separation of binary systems. *Bulletin of the Kursk State Agricultural Academy*. 2015. № 2. P. 73–75.

Components of EO have different fugitiveness, which depends on their boiling temperatures (T_{boil}). This is the indicator that connects structure of EO components, their physicochemical properties with thermodynamic distillation parameters. The values of T_{boil} of aromatic substances given in scientific publications have divergences at the level± 10…45 °C[32].

To determine the valid T_{boil} of essential oils components logarithm correlation of rectified holding time (Kovac index – KI) with T_{boil} was used.

It should be noted that certainty of IK values of aromatic substances can be achieved only under own researches, subject to the conditions of GC analysis on existing equipment, the correct choice of standard substance, stationary phase mark. The following sequence of actions for determining IK of EO components is suggested.

Primarily a chromatograph of a base matrix of six n-paraffins is obtained (Picture 1a). Out of the EO chromatograph, for example coriander (Picture 1b), relative maintenance time of its key components is determine and lgt'R logarithm value for each component is rated. Then, under the system of coordinate gird the chromatograph of a base matrix is imposed on the essential oil chromatograph (Picture 1c) and n-paraffins between the components are situated are set (On Picture 1c these are component 5, it is situated between C10 (decane) and C11 (hendecane) – $t'z10 < t5 < t'z11$). Such an operation allows set reference points (n-paraffins) to calculate the values of KI. Calculation of the KI is recommended to be conducted on a computer program in the Visual Basic environment[33].

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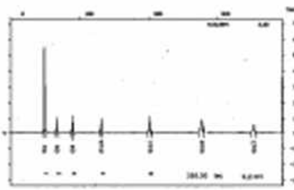
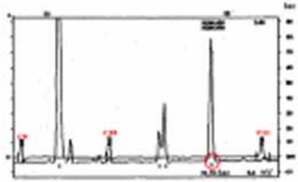
[32] Plemenkov V.V. Chemistry of isoprenoids. Chapter 5. Monoterpenes. *Chemistry of vegetable raw materials*. 2006. № 2. P. 63–87.

Mohamed S.A., Beshir D.M., Rabah Mohamed A.A. Simulation and characterization in the refining industry: A review. *Journal of Petroleum Technology and Alternative Fuels*. 2014. Vol. 5. № 3. P. 26–30.

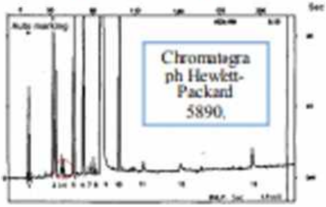
[33] Frolova N.E., Silka I. M.Computer program of calculation of gaschromatic parameters of containment of essential oils components. *Bulletin of the Khmelnitsk National University: Technical Sciences*. 2016. № 6. P. 133–137.



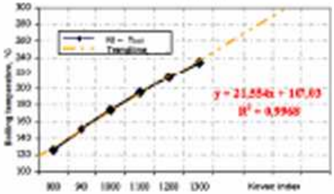
Picture 1a. The chromatograph of n-paraffins base matrix



Picture 1c. The imposition of the base matrix on the coriander EO chromatograph



Picture 1b. The coriander EO chromatograph



Picture 1d. The calibration schedule in coordinates KI – Tboil

By KI values and correlation dependence of KI to T_{boil}, the actual T_{boil} of EO components is determined (Picture 1d)[34] Values of the coriander EO components correlation constants are listed in Table 1.

Table 1

The correlation constants of coriander EO components

№ of apex	Calculated Kovac index (KI)	Identified component	T_{boil}, °C
1	942	α-pinene	150
2	951	camphene	160
3	979	β-myrcene	163
4	998	d-limonene	172
5	1017	cineol	173

[34] Development of a methodology of determining the boiling point of volatile aromatic components of essential oils / N.E. Frolova, S.I. Usatyuk, V.O. Usenko, I.M. Matko. *Food and processing industry*. 2006. № 12. P. 42–47.

№ of apex	Calculated Kovac index (KI)	Identified component	T_{boil}, °C
6	1032	β-phellandrene	175
7	1060	n-cymene	183
8	1089	l-linalool	185
9	1096	d-camphor	198
10	1118	linalyl acetate	204
11	1165	l-borneol	211
12	1180	α-terpineol	220
13	1243	geraniol	229

The reference points of the equilibrium pressures of EO distillation are situated in dependence of key components T_{boil} and relevant vapour pressure. In the information field, such data is limited to processing of spruce extracts, and also terebenthere terpenes[35]. We suggest carrying out calculations of equilibrium pressures values from key components T_{boil} according to the Duhring rule[40], which is represented by the equation:

$$\frac{t_1 - t_2}{t_1^{ст} - t_2^{ст}} = C, \quad (1)$$

where: t_1 – EO components T_{boil} under atmospheric pressure, °C; t_2 – EO components T_{boil} under the investigated pressure, °C; $t_1^{ст} - t_2^{ст}$ – difference in T_{boil} of standard liquid under atmospheric and investigated pressure, °C; C – Duhring constant. As a standard liquid for terpenoids water was used, for terpenes – α-pinene.

In order to prevent systematic errors that are possible within low pressures, it is additionally proposed to use Kalingert-Davis nomogram in the system of reference A-B[36]. In the Table 2 the set values of equilibrium pressures at the EO key components T_{boil} are listed.

[35] Rudakov G.A. Chemistry and technology of camphor: 2nd, revised and enlarged edition. Moskow: Forestry industry, 1976. 348 p.

[36] Wiles S. Phase equilibrium in chemical technology: in 2 parts / trans. from English A.B. Bespalovaya, ed. C. Beskov. Moscow: Mir, 1989. 304 p.

Table 2

The values of equilibrium pressures of the EO key components

Temprature, °C	Pressure, kPa					
	α-pinene	β-myrcene	cineol	l-linalool	citral	nerol
20	0.43	0.28	0.22	-	-	-
40	0.79	0.53	0.44	-	0.34	-
60	1.38	0.96	0.77	-	0.609	0.21
80	2.33	1.63	1.23	-	1.05	0.407
100	3.73	2.93	2.15	3.47	1.76	0.77
120	5.93	4.26	3.30	5.41	2.71	1.24
140	19.1	14.26	11.34	17.33	9.46	-
160	87.05	67.19	58.1	79.7	49.4	-
170	-	-	100.25	-	85.7	-

Calculated and refined data is used to determine the values of equilibrium pressures for distillation of EO. So, when distillation of EO from catnip to a key component linalool, it is sufficient to keep pressure within (2,66…1,33) kPa at distillation temperatures (57…62) °C.

In secundal subsystem linalool-citral operating pressure should be kept within (1.33…0.96) kPa at temperature (62…80) °C. For subsystems ciral-citronellol (80…92) °C – (0.96…0.66) kPa and citronellol-geranyl acetate (96…100) °C – (0.66…0.33) kPa. Relative fugitiveness of α characterize ability of EO components transfer to a steam phase. According to A. Gorak's data[37], pressure drop from 101.32 kPa to 6.67 kPa allows increase the α value from 1.11 to 1.23 and at a further decrease to 1.33 kPa, value of α increases to 2.68. Thus, when modeling the EO distillation regimes it is necessary to include the pressures behind which, within T_{boil} of EO key components, α has the biggest values (adjust with the temperatures of the selected refrigerants). The temperature intervals of EO distillation are selected within valid T_{boil} of components at the set equilibrium pressure value.

Construction of equilibrium curves for liquid's composition and steam У-X for the key EO components allows determine the minimum number of theoretically perfect distillation plates. Composition of a steam phase can be

[37] Gorak A.E. Sorensen Distillation: fundamentals and principles. *Academic Press*. 2014. 415 p.

found graphically, based on the composition of a liquid phase. However, this method is much more complicated than the calculation method; therefore we should restrict to only mentioning it. More detailed description is given by Thormann and Rozengart[38].

Determination of minimum number of theoretically perfect EO distillation plates n_{min}, is conducted by equation Fenske-Underwood[39]:

$$n_{min} = \frac{\lg(X_{1E}/X_{2E})\cdot(X_{2B}/X_{1B})}{\lg \alpha_{1,2}} \tag{2}$$

where: X_{1E} – diethylin of low-boiling components LBC from the prvious subsystem in a steam phase, % of mass; X_{2E} – diethylin of high-boiling component HBC of current subsystem in a steam phase, % of mass; X_{1B} – diethylin of LBC from the previous subsystem in a steam phase, % of mass; X_{2B} – HBC of current subsystem in a liquid phase, % of mass; α – relative fugitiveness of components critical steam.

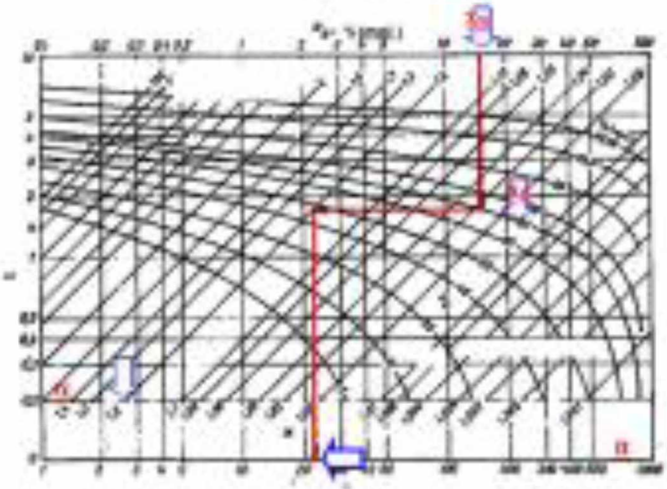
Also determination of n_{min} may be conducted using the nomogram of Melpolder and Hedington, developed using Fenske-Underwood equation (Picture 2).

Calculations prove that through real distillation of EO from catmint, enriching the distillate with low boiling components by 90% is possible even with their content in the initial mixture is about 5%. For subsystems citral-citronellol-geranyl acetate such a result may be achieved only when content of the components in the initial mixture starts from 40% of mass and demands a number of theoretically perfect plates =18…19.

To influential governing regimes of EO rectification belongs the reflux ratio V, as a ratio of hot reflux (of phlegm) to an amount of distillate. As V = min the subsystem of set composition may not be obtained under any conditions, and a number of theoretically perfect plates becomes infinitely large (n = ∞).

[38] Crele E. Manual on laboratory distillation / trans. from German, ed. V. M. Olevskiy. Moskow. Chemistry, 1980. 520 p.

[39] Frolova N.E., Ukrainets A.I. Theoretical substantiaton of parameters of essential oils fractional distillation. *Food Science and Technology*. 2009. № 2 (7). P. 54–58.



Picture. 2. The nomogram of Melpolder and Hedington for determination of minimum number of theoretical degrees of EO division as V = ∞

As V = ∞, rectification takes place without catch of distillate and the system works on its own. Increase of a reflux ratio of V_{min} value allows conduct rectification in a column with a determined number of theoretically perfect plates. Namely V_{min} i V_{∞} form limit conditions of rectification.

Operating reflux ratio is determined by practical tests. To start the rectification V_{min} should be determined out of equation:

$$V_{min} = \frac{X_{д}}{y} - 1; \qquad (3)$$

where: $X_{д}$ – weight percent of LBC in the distillate, У – estimated coefficient calculated from equilibrium curves.

Values of reflux ratios V and numbers of valid theoretically perfect distillation plates are determined from equilibrium curves for each pair of key EO components/

Range of operating reflux ratios $V_{operating}$ at the beginning and at the end of rectification are determined up to a number of theoretically perfect plates N, which is determined by McKeb-Tile graph method[40].

The calculated values of a minimum number of theoretically perfect plates of catnip EO distillation indicated that for pairs of key components

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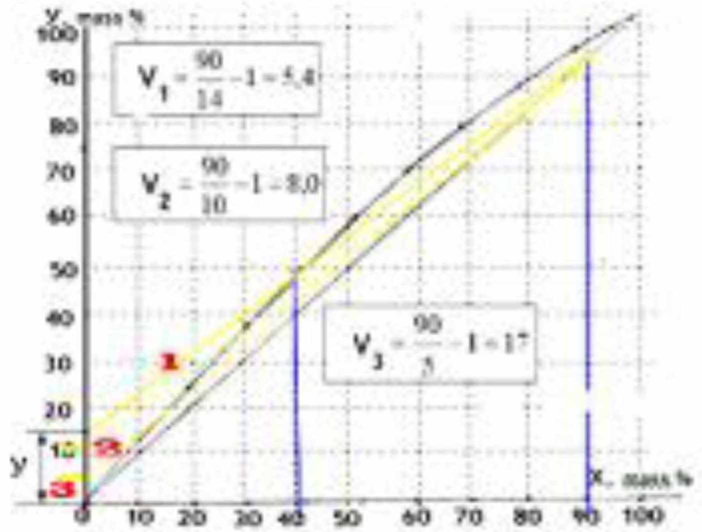
myrcene–cineol $n_{min} \geq 4.0$ is sufficient; cineol–linalool – $n_{min} \geq 5.6$; linalool–citral – $n_{min} \geq 6.1$. For a pair with high-boiling components, in particular citral–citronellol, n_{min} value rises to 18.0, and for citronellol–geranyl acetate $n_{min} \geq 19.5$. It was also found out that for pairs of low-boiling components (myrcene, cineol, linalool) enrichment of distillate with components up to 90% of mass may be achieved even if the content of the components in the initial mixture is from 5% of mass. For the pairs citral–citronellol, citronellol–geranyl acetate such a result may be achieved if content of the components in the initial mixture is from 40% of mass.

On Picture 3 the equilibrium curve for the pair citronellol- geranyl acetate of catnip EO is shown.



Picture 3. The equilibrium curve for the pair citronellol- geranyl acetate of catnip EO

The results of theoretical modeling of catnip EO distillation regimes re given in Table. 3

A similar theoretical modeling of fractional distillation parameters can be made for any EO if there is corresponding output data. Flavors obtained using theoretically modeled and realized in real processes are relevant in formulations of perfumery, cosmetics, pharmaceutical and nutrition products.

Nowadays it is complicated to imagine at least one branch of the food industry, which would not use food flavors in manufacturing of its products.

Table 3

Model regimes of catnip EO distillation

Pairs of key components	Temperature ranges, °C	Pressure, kPa	α	nmin	V
Myrcene – cineol	57…62	2.66	3.21	2.6	4.0 ± 0.5
Cineol – linalool	62…80	1.33	2.58	2.8	6.0 ± 0.5
Linalool – citral	80…92	1.33	2.24	3.1	7.0 ± 0.5
Citral – citronellol	90…100	0.66	1.56	10	12.0 ± 1.0
Citronellol – geranyl acetate	100…120	0.33	1.44	10	14.0 ± 1.0

A promising area of scientific research is application of flavors in the technology of milk and milk-containing products. Aromatisation of dairy products can be carried out in different ways, in particular:

– direct application of flavors of natural or chemical origin;

– usage of aromatic components to enhance the smell of fruit and berry fillers used in manufacturing of products;

– application of various compositions of spices.

Widespread is the usage of flavors in production of butter with reduced fat content and spreads (mainly of "creamy flavor"), and also rennet cheese (creamy, evaporated milk aroma, a specific cheesy in the production of cheeses with accelerated ripening modes, etc.) aroma of mushrooms, ham, shrimp in production of processed cheeses, and also in composition of drinkable types of milk, yoghurts, caseous products, etc.

Widespread is the usage of flavors in production of drinkable types of milk. Instability of a protein phase of milk during a reduction of environment acidity makes it impossible to use natural fruits and berries in the technology of drinkable milk and cream. Therefore, to provide attractive and diverse organoleptic characteristics, flavors with identical to natural fillers aromas – strawberry, kiwi, raspberries, melon, banana, vanilla, etc., as well as food colors and sweeteners are introduced into these products. The technology of the technology of milk cocktails for preschool- and school-age children with balanced composition, enriched with a complex of vitamins and minerals was developed[41]. Providing bright attractive organoleptic characteristics of milk cocktails is also achieved by the introduction of food colors and the addition of flavors.

At the National University of Food Technologies sour cream products were developed. As flavoring fillers salt, dill, dry mushrooms, taste and aromatic additives[42] (with the taste and aroma of cheeses Cheddar, Gauda, Parmesan etc.) were used. Among one of the most popular products are yoghurts that are deservedly in demand due to their nutritional value and dietary properties. Yogurts are produced using cow, goat, sheep milk without or with the addition of fillers or supplements. Yoghurts are made according to the basic recipes, which include dry skimmed milk or a structure stabilizer, a starter and sugar (if necessary). All varieties of organoleptic characteristics are achieved using taste and flavoring fillers and additives, in the quality of which pieces of fruits, nuts, cereals, dried fruits, etc., can be used as well as flavors, often synthetic ones[43].

In production of dairy products, milk fat or (and) protein are partially replaced by appropriate constituents of vegetable or animal origin (vegetable fat, milk fat substitutes, soy protein isolate, etc.). In addition, dairy products based on soy milk, such as yogurt and drinkable milk are becoming popular. This is primarily due to individual intolerance to lactose of a part of adult and child population, and also spread of vegetarianism. Naturally, such products have a specific taste and smell of beans, which necessitates use of taste and aroma fillers and additives.

In the technology of dairy products outcomes of processing of fruits and berries – jams, basins etc. are widely used. The technology of their production involves heat treatment of raw material, which naturally leads to a decrease in the intensity and change in aroma and color. Colouring agents and flavors are used to enhance and restore aroma of such products.

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A promising source of natural taste and aroma compounds in dairy products technology is spices. What is more, the spices contain biologically active substances (essential oils, terpenoids, phenolic and polyphenol substances, vitamins, micro-and macro elements, etc.), that are involved in a metabolism of the human body, have an ability to regulate metabolism, exhibit bacteriostatic and antioxidant activity. Thus, addition of oregano essential oils to sour milk cheese allows reduce the speed of processes that cause spoilage of the product, thereby extending its shelf life[44].

At the National University of Food Technologies recipes of sour milk paste with compositions of spices on the basis of sour milk cheese and sour cream are developed[45]. Compositions for dairy pastes included spices such as anise, anise, cinnamon, cardamom, ginger, turmeric, cloves, black currants, sweet pepper, trigonella, nutmeg and sumac. Application of compositions of spices allowed not only to get products with a diverse flavor and aroma range, but also to provide more stable quality indicators of final products during storage.

As an alternative to the usage of spices in their natural state, conditioned by the need for their preparation and somewhat unstable quality indicators, depending on the conditions of obtaining, transporting and storage of raw materials, is removal of an active fraction with obtaining of extracts. Extracts of spices depending on the concentration of useful substances are - liquid, thick and dry, from the type of solvent – water, alcohol, essential, oil and obtained using liquefied gases (CO_2-extracts).

Liquid extracts are widespread due to predicted correlation between active substances, convenience in measurement, ease of obtaining. However, there is also a number of disadvantages: increased content of concomitant substances received from plant raw material, the appearance of sediment, need for sealed packaging[46].

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[44] Asensio Claudia M., Grosso Nelson R., Juliani H. Rodolfo Quality preservation of organic cottage cheese using oregano essential oils. *Food Science and Technology*. 2015. Vol. 60. Issue 2. P. 664–671.

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[46] Denisova E.A. Caseous and cereal products. *Food and processing industry*. 2005. Issue 2. P. 12–13.

Spices oleoresins usually contain a complex mixture of volatile oils (10–25%), triglycerides, sterols, waxes and resinous substances. Oleoresins usually have low mutual solubility, but completely preserve the taste and aroma of natural spices[47].

Today, the cleanest and most natural product is CO_2-extracts obtained using purified liquefied gases, while retaining their naturalness and correspondence of taste and flavor of initial raw materials but are of high cost.

Since aroma of spices is determined by the content of essential oils, their extraction in pure form and usage for dairy products is promising.

But the spectrum of aromatic characteristics of such essential oils is rather narrow, not always combined with the milk base, especially the dessert ones.

To avoid these disadvantages and create a variety of flavors that will satisfy the most demanding consumers, usage of fractional distillation of essential oils should be used. In accordance with the proposed method, rectification of essential oils of plants from the climatic zone of Ukraine (buds of garden trees – apple, pear, cherry and bushes – currants, raspberries) was conducted[48].

On the basis of obtained fractions concentrated flavors were obtained with usage of which drinkable yogurt "Harmonious" and spread "Caraway aromas" and "Citrus" were developed. Through the production of yogurt flavors were added into fermented mixture immediately before packaging. After adding flavors, the fermented mixture was stirred for 5 to 10 minutes and proceeded to packaging. Spreads are made by transforming a high fat mixture. Flavors "Caraway aromas" and "Citrus" in the amount of 0.02% and 0.025%, respectively, to the mass of dry matter was pre-diluted in a small portion of the emulsion and added to a normalized, high-fat mixture at constant stirring, then the process was carried out according to the general technological scheme. The resulting products had a characteristic taste and smell with an after-taste of spices and a spicy aroma.

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The usage of natural flavors obtained by fractional distillation of essential oils will contribute to the expansion of the spectrum of food flavors and the range of nutrition products with the usage of natural ingredients, which in its turn will increase consumer confidence and allow them to be offered for use in special-purpose product technologies, in particular for children and diet nutrition.

CONCLUSIONS

1. Fractional distillation of natural sources of aromatic substances is an effective process in processing of natural sources of aromatic substances in order to obtain a wide range of natural flavors.

2. Theoretical modeling of fractional distillation regimes allows, in real life conditions, form a set to aroma fraction by collecting into a separate fraction of key aromatic components and a number of other, close to the key ones in fugitiveness and boiling temperature, and also significantly reduce expenses of energy and material resources.

3. The modeling regimes of fractional distillation of essential oils include the actual T_{boil} of EO components at atmospheric pressure, the equilibrium pressures of EO distillation, the relative fugitiveness of α, the minimum number of theoretically perfect plates and operating reflux ratio.

4. The target products of the development are fractions of different aroma, which can be used as independent natural flavors, and as components of fractions combinations of one essential oil or various essential oils.

5. Fractional distillation does not destroy the natural structural bonds of essential oils components, preserves their biological capacity. Combination of essential oils fractions according to the determined mass ratios provides numerous variants of natural flavors for usage in food, in particular dairy industry.

SUMMARY

In this paper the results of theoretical justification of coherence of setting model regimes of distillation of essential oils into separate aroma fractions, which are highly concentrated flavors of long-term storage, are represented.

High-performance essential oils distillation requires adherence to strict process conditions, crucial for regulating fraction's aroma. Complexity in determining of such regimes consist in duration of experimental distillations with mentioned essential oils expenses.

Theoretical modelling of distillation parameters provides, through real distillations, development of set fraction's aroma on account of collecting key aromatic components and a number of others, similar to the key ones in fugitiveness and boiling point, into a separate fraction.

To model regimes of essential oil fraction distillation valid Tboil of essential oils components at ambient pressure, equilibrium pressures of essential oils distillation, value of relative fugitiveness of α, as ratio of pressures at a constant temperature, are attributed. A minimum number of theoretically perfect plates and operating reflux ratio were taken into account.

Fraction distillation doesn't ruin natural structural bond of essential oils compounds, preserves their biologic ability. Combining fractions of one essential oil or various ones by the determined mass ratios provides numerous variants of natural flavors for usage in food, in particular dairy industry.

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Information about authors:
Frolova N. E.,
Dr. in Technical Sciences, Associate Professor,
Professor of Department of catering and Ayurvedic produce
National University of Food Technologies
68, Volodymyrska Str., Kyiv, 01601, Ukraine
Yushchenko N. M.,
PhD in Technical Sciences, Associate Professor,
Associate Professor of Department of catering and Ayurvedic produce
National University of Food Technologies
68, Volodymyrska Str., Kyiv, 01601, Ukraine