Nexo Revista Científica
Vol. 36, No. 02, pp. 139-147/Marzo 2023

ISSN-E 1995-9516
Universidad Nacional de Ingeniería

http://revistas.uni.edu.ni/index.php/Nexo
https://doi.org/10.5377/nexo.v36i02.16021

Designing the technology and composition of plant extracts using reduced atmospheric pressure

Diseño de la tecnología y composición de extractos vegetales a presión atmosférica reducida

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(recibido/received: 08-noviembre-2022; aceptado/accepted: 15-enero-2023)

ABSTRACT

The study aims to establish the regimes and parameters of extraction of bioactive substances from raw plant materials by means of extraction with reduced atmospheric pressure for the purpose of manufacturing non-alcoholic functional syrups. The work is performed by leading specialists of the Kemerovo State University using conventional methods. Preparation of the extracts involves aqueous alcoholic treatment of the crushed mass of raw plant materials selected according to the functional parameters. Extraction is performed in a Soxhlet extractor with an advanced liquid ring vacuum pump. The extraction process is monitored by changes in the content of terpenoids in the extract obtained and the mass fraction of dry matter. As a result, the study establishes the parameters and regimes for carrying out and intensifying the extraction of terpenoids and dry matter from fennel, anise, caraway, and dill: pressure – 0.2 MPa, extraction temperature – 50°C, duration of the process – 45 min, solvent – 40 % aqueous alcoholic solution, raw material particle size – 2-3 mm. These process conditions guarantee the highest degree of extraction of terpenoids and dry matter from raw materials, as well as reduce the time and economic costs.

Keywords: extraction, raw plant material, adjustable parameters, terpenoids, soxhlet extractor, extract.

RESUMEN

El estudio tiene como objetivo establecer los regímenes y parámetros de extracción de sustancias bioactivas a partir de materias primas vegetales mediante extracción con presión atmosférica reducida con el fin de fabricar jarabes funcionales no alcohólicos. El trabajo es realizado por destacados especialistas de la Universidad Estatal de Kemerovo utilizando métodos convencionales. La preparación de los extractos implica el tratamiento alcohólico acuoso de la masa triturada de materias primas vegetales seleccionadas de acuerdo con los parámetros funcionales. La extracción se realiza en un extractor Soxhlet con una bomba de vacío de anillo líquido avanzada. El proceso de extracción es monitoreado por cambios en el contenido de terpenoides en el extracto obtenido y la fracción de masa de materia seca. Como resultado, el estudio establece los parámetros y regímenes para realizar e intensificar la extracción de terpenoides y materia seca de hinojo, anís, alcaravea y eneldo: presión – 0,2 MPa, temperatura de extracción – 50°C, duración del proceso – 45 min, disolvente – solución alcohólica acuosa al 40 %, tamaño de partícula de la materia prima – 2-3 mm. Estas condiciones de proceso garantizan el más alto grado de extracción de terpenoides y materia seca de las materias primas, además de reducir el tiempo y los costes económicos.

Palabras claves: extracción, materia prima vegetal, parámetros ajustables, terpenoides, extractor Soxhlet, extracto.

1. INTRODUCTION

In recent years, the food and processing industry, in addition to the production of non-alcoholic drinks, has been showing great popularization of beverages based on natural raw plant materials. The matter of creating new types of drinks with extensive physiological effects on the human body takes on a special role at this point in the development of scientific and technological progress (Zolotin et al., 2020; Bakumenko et al., 2021; Sergeeva et al., 2019; Sosiura et al., 2019).

The quality of any food product is influenced by many factors, such as the balance of the recipe components and their successful combination, composition and quality parameters of primary raw materials, technologies and technical solutions used in the production, the level of material and technical base of production, equipment, etc. (Miller et al., 2021; Gurianov et al., 2012; Borodulin et al., 2021).

Furthermore, the design of modern food products gives attention to the foundation of its quality, which is accomplished by ensuring a balanced composition and formulation of the product. The functional components used in the production of syrups are often extracts derived from raw materials of plant origin. These extracts are concentrates of an extensive range of biologically active substances, which give the product a characteristic taste and flavor without the use of artificial flavorings, thereby fully meeting consumer demands and the criteria of healthy foods (Domaretskii, 2011; Soboleva and Kovaleva, 2020).

Without a doubt, the use of natural raw materials in the production of foods gives them high nutritional and biological value, which primarily owes to the original combination of physiologically and biologically active substances that is difficult to create artificially. The raw materials in question have great potential for application in the treatment and prevention of essential micronutrient deficiencies (Shatniuk et al., 2014; Kolberg et al., 2022). The health benefits of plant foods are due to the interaction of biologically active compounds and their synergism, as confirmed by various literary sources (Pozniakovskii et al., 2017; Shkolnikova and Averianova, 2021).

When obtaining biologically active substances from plant components, the complete extraction can be achieved by perfectly adjusted process parameters, including the type of solvent, the duration of the technological operation, and the hydromodule. This careful adjustment can also save energy resources and the amount of solvent, balancing the inevitable losses of the target component.

Research on the selection of technological parameters is becoming increasingly relevant. Employees of the Russian Research Center "Applied Chemistry" and the ITMO University have proposed several technological solutions, one of which is the application of rotary-pulsation and planetary apparatuses, the hydrodynamic modes of which allow initiating convective mass transfer in the pores of particles and thereby accelerate the process (Ivanov, Matveeva, 2015).

One method known to be used to increase the output of bioactive substances is two-phase extraction. Compared to single-phase extraction, this method is marked by a pronounced differentiation of lipophilic and hydrophilic substances in the extract (Vainshtein, Kaukhova, 2014).

The most popular method of optimizing the extraction process is diffusion, which provides a high output of bioactive substances (such as vitamins, flavonoids, mineral and pectin substances, organic acids, etc.) from raw plant materials. In particular, this method is utilized at the Department of Theory of Mechanisms and Machine Parts of the Tambov State Technical University in studying the effects of technological parameters

on the degree of extraction of biologically active substances from nettle, common hops, lemon monard, lemon balm, narrow-leaved cypress, honeysuckle, rose hips, and sea buckthorn (Guskov et al., 2018).

In light of the above, a priority direction in the development of the food and processing industry is the development of economically viable and rational technology for processing raw plant materials in order to maximize the extraction of BAS and their further preservation in the finished product. This supports the expediency of the experimental selection of technological modes and parameters of extraction of plant-origin raw materials. In the present study, the method used to raise the quantitative output of bioactive substances, particularly terpenoids as a target component for the production of functional syrups, is extraction under reduced atmospheric pressure with adjustable parameters.

The goal of the study is to determine the adjustable technological regimes and parameters for the extraction of raw plant materials using reduced atmospheric pressure in the preparation of extract compositions for further production of non-alcoholic functional syrups.

2. METHODS

The raw plant materials chosen for the application of the technology and the selection of optimal parameters were those with high terpenoid content – fennel, anise, caraway, and dill. The content of the target component determines the functional orientation of the syrup developed – normalization of the gastrointestinal tract. This owes to the fact that terpenoids are carbon chains that serve as an intermediate element in the biosynthesis of vitamins D, E, K, cholesterol, enzymes, steroid hormones, bile acids, etc.

Terpenoids derived from raw plant materials have a great range of biological effects on humans, which is why they are of interest in the search for new solutions in the creation of functional products. The terpenoid group includes the subgroups of bitters, oils, and fatty oils. Bitters are primarily nitrogen-free chemicals that have a positive effect on human digestion and stimulate appetite. Bitterness activates the salivary and biliary glands and pancreatic and gastric. These substances are chemically similar to essential oils. A distinguishing feature of essential oils is increased secretion of the listed glands at a slower rate, but with greater intensity and persistence. Fatty oils are represented by such bioactive substances as fatty acids, tocopherols, carotenoids, sterols, and other compounds that cause the respective pharmacological effect of the fatty oil (laxative, hepatoprotective, anti-sclerotic, wound healing, etc.).

The stage of extraction in the technological process is considered to be fundamental in the production of extracts. This stage can be divided into the substages of selecting the solvent, choosing the intensification method for the extraction process, including optimizing extraction parameters using the adopted intensification method.

Water is an extractant that is widely used in the extraction process, but its use in the extraction of terpenoids from raw plant materials is inexpedient. Aqueous extraction allows the extraction of hydrophilic substances, while for lipophilic substances it is advisable to use ethanol. For this reason, the solvent used in this study was an aqueous alcoholic mixture with an experimentally selected concentration.

Identification of the optimal extraction parameters was carried out using an extraction unit with a liquid ring vacuum pump designed to provide a reduced atmospheric pressure during the extraction of raw materials. This method of treatment intensifies the process of raw plant material processing and the extraction of terpenoids.

With this unit, the extractant is supplied under a vacuum, which allows the pores of the raw material to be filled with it. Reduced atmospheric pressure promotes not only external but internal diffusion, thus intensifying the process. The solvent is brought to the boiling point, which depends on the concentration of

the aqueous-alcoholic mixture. Boiling of the solvent helps to stimulate the retrieval of extractable substances in the extractant.

During the operation of the unit, the distillate is returned to the extractor and mixed with the extractant and then returned to the unit. This installation, as compared with similar ones, increases productivity and ensures high preservation of bioactive substances, including terpenoids.

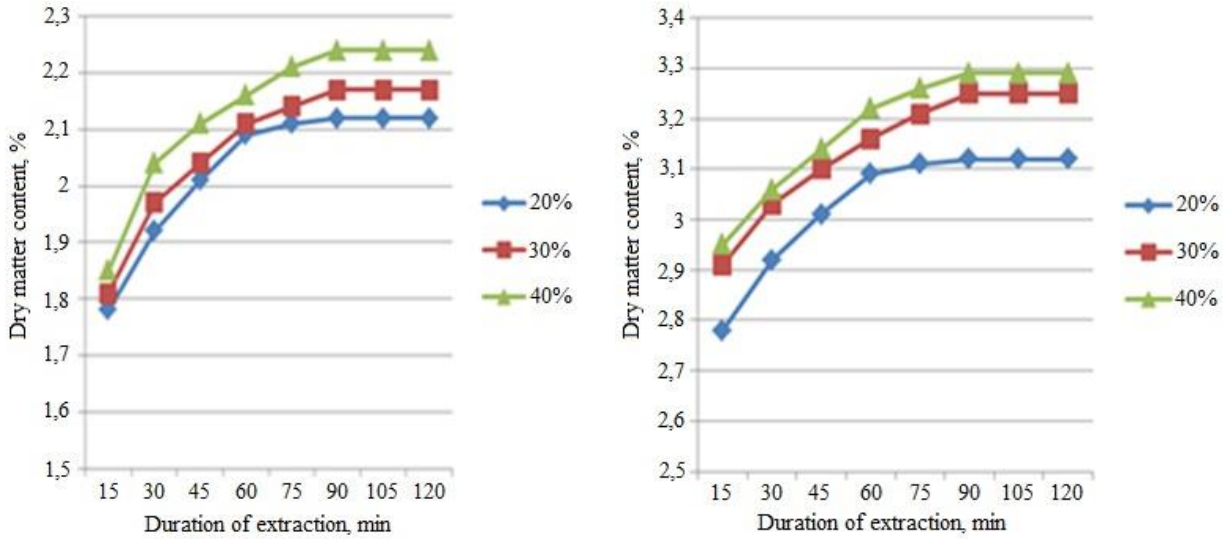
At the next stage, the dosage of extracts included in the composition for the preparation of syrup based on raw plant material was established. The formula of the composition included extracts of fennel, anise, caraway, and dill prepared by the previously described method.

The selection of a rational sample was carried out through organoleptic evaluation by a group of tasters using a 6-point scale for organoleptic evaluation of extract aroma. Evaluation of the prepared samples was conducted by the indicators of aroma, the harmony of scent, and the ability to evoke positive emotions in the taster (Shigina, 2005).

3. RESULTS AND DISCUSSION

The conducted study involved the selection of solvent for the extraction of raw plant materials and its concentration. As part of this selection, extract samples were prepared with different concentrations of the aqueous-alcoholic solvent (20, 30, and 40%). The extracts were obtained from raw materials ground to 2-3 mm under the temperature of 50°C in the course of 2 hours using the method of traditional maceration observing a hydromodule of 1:6 for fennel and dill and 1:5 for caraway and anise. According to literary sources, the most favorable value of the hydromodule for essential oil raw material ranges from 1:4 to 1:6, while a decrease in this value significantly reduces the yield of dry matter due to changes in hydrodynamic conditions inside the extractor (Egorova et al., 2011).

Figures 1a-d show the results obtained in the experiment on selecting the concentration of aqueous alcoholic solvent.



a **b**

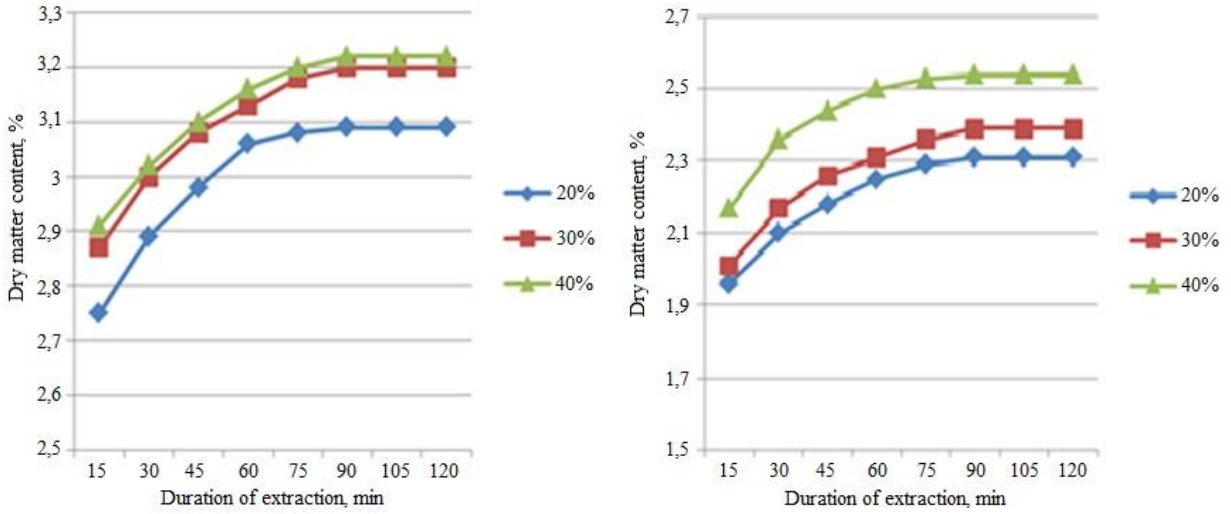


Figure 1. Results of the experiment on the selection of aqueous alcoholic solvent concentration

The provided figures demonstrate that the rational concentration of the aqueous-alcoholic extract is 40% because it achieved the maximum extraction of dry matter in all samples of raw plant materials.

Maceration with an aqueous alcoholic solution is a fairly effective method of extracting terpenoids from raw materials. Nevertheless, this type of extraction has one drawback – high time costs. There are several alternative methods able to reduce the prolonged time of processing, such as ultrasonic, fluid supercritical, vacuum, microwave, or infrared extraction. All of these prove to be more energy-efficient relative to the traditional Soxhlet extraction or maceration.

For the purposes of speeding up the extraction process and increasing the level of extraction of bioactive substances, particularly terpenoids, the conducted experiment employed the vacuum-assisted extraction method. This type of extraction assists in freeing the pores of the selected raw material from air, which impedes the occurrence of contact between the extractant and the solid particles of the raw material.

To determine the optimal technological parameters of the selected type of extraction of raw materials, a series of experiments were conducted varying the parameters of temperature, pressure, and time.

During the conducted experiment, fennel, anise, caraway, and dill were ground in a mill to a size of 2-3 mm. Then, the raw materials were soaked in an aqueous alcoholic solution (40%) with a hydromodule of 1:6 for fennel and dill and 1:5 for caraway and anise. Afterward, the entire prepared working mixture was loaded into the unit for extraction, the duration of which varied from 15 to 45 min.

Analysis of the extracts obtained in the experiment was conducted by the parameters of dry matter mass fraction and total terpenoid content (Tables 1 and 2).

Table 1. Effect of the use of reduced atmospheric pressure on the content of dry matter mass fraction

Extraction parameters	Dry matter mass fraction, %			
	Fennel	Anise	Caraway	Dill
p=0.1 MPa, T=40°C, t= 15 min	3.04	4.02	3.95	3.10
p=0.1 MPa, T=40°C, t= 30 min	3.25	4.15	4.07	3.32
p=0.1 MPa, T=40°C, t= 45 min	3.67	4.52	4.24	3.58
p=0.2 MPa, T=50°C, t= 15 min	4.13	5.14	4.38	4.16

p=0.2 MPa, T=50°C, t= 30 min	4.22	6.01	4.49	4.25
p=0.2 MPa, T=50°C, t= 45 min	4.43	6.17	5.14	4.52
p=0.3 MPa, T=60°C, t= 15 min	4.10	6.07	5.12	4.21
p=0.3 MPa, T=60°C, t= 30 min	4.12	6.14	5.19	4.24
p=0.3 MPa, T=60°C, t= 45 min	4.18	6.19	5.23	4.25

Table 2. Effect of the use of reduced atmospheric pressure on the content of terpenoids in the extract

Extraction parameters	Terpenoid mass fraction, mg/100 cm^3			
	Fennel	Anise	Caraway	Dill
p=0.1 MPa, T=40°C, t=15 min	1,326	652	1,105	821
p=0.1 MPa, T=40°C, t=30 min	1,431	702	1,137	847
p=0.1 MPa, T=40°C, t=45 min	1,495	728	1,201	906
p=0.2 MPa, T=50°C, t=15 min	1,528	751	1,257	849
p=0.2 MPa, T=50°C, t=30 min	1,674	792	1,305	914
p=0.2 MPa, T=50°C, t=45 min	1,729	812	1,380	925
p=0.3 MPa, T=60°C, t=15 min	1,695	797	1,307	915
p=0.3 MPa, T=60°C, t=30 min	1,728	806	1,325	927
p=0.3 MPa, T=60°C, t=45 min	1,745	811	1,376	938

Compared with the method of traditional maceration, the mass fraction of dry matter and terpenoids in the experimental samples is higher due to the use of vacuum, which results in a stronger diffusion of the extractant into the cell structure of the raw material. The rational parameters of extraction determined in the experiments are the temperature of 50°C, the pressure of 0.2 MPa, and 45 min duration of the process. Given that the composition of extracts is intended for the production of syrup, which assumes zero concentration of alcohol in the final product, another stage of preparation of the extracts was alcohol evaporation (T = 40-45°C, t = 25 min, p=0.2 MPa). Through this operation, the ethanol evaporates, while the target component, the high-temperature-resistant terpenoids, remains. These extracts can therefore be used in the formula of syrups that do not contain alcohol. In the next step, the extracts are filtered and immediately used in production.

The extracts of fennel, anise, caraway, and dill prepared by the proposed method with the selected parameters contain 1,729 mg/100 cm^3, 812 mg/100 cm^3, 1,380 mg/100 cm^3, and 925 mg/100 cm^3 terpenoids, respectively, with the following dry matter mass fractions: fennel extract – 4.43%, anise extract – 6.17%, caraway extract – 5.14%, and dill extract – 4.52%.

Next, we determined the content of the composition of non-alcoholic extracts for the preparation of the syrup. In these studies, seven samples of extract compositions were prepared, each of which had either the minimum or the maximum dose of each component in different combinations (the composition of these samples is given in Table 3).

Table 3. Analysis of extract content for the preparation of syrup

Extract/Sample	Composition variants (extract content, %)						
	№ 1	№ 2	№ 3	№ 4	№ 5	№ 6	№ 7
Common fennel extract	25	25	15	25	25	15	35
Common anise extract	45	45	45	35	35	45	35
Caraway extract	25	15	25	25	35	35	25
Dill extract	5	15	15	15	5	5	5

The number of plant extracts in the composition was chosen based on the organoleptic assessment of the extracts' aroma performed by a group of tasters using a 6-point scale [17]. Figure 2 presents the data obtained in the experiment.

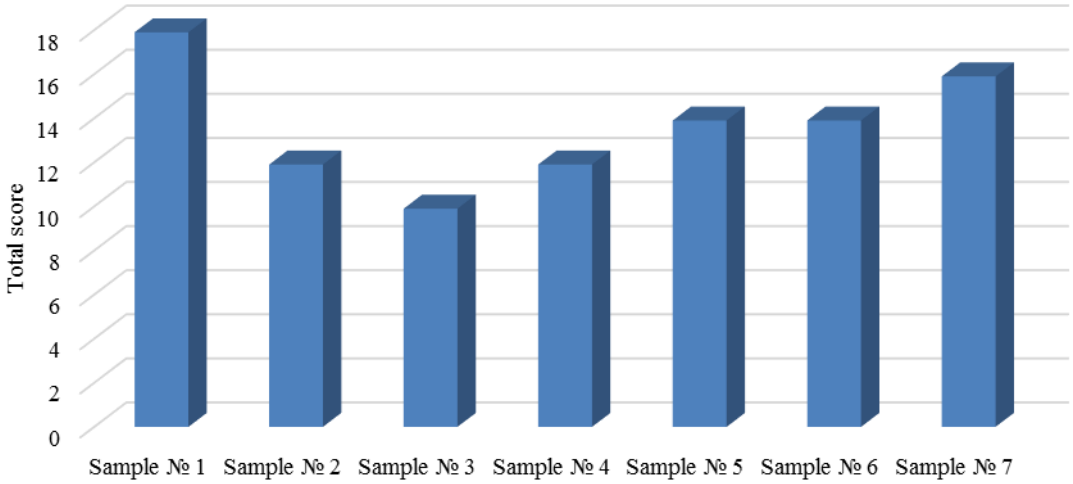


Figure 2. Assessment of the aroma of raw plant extracts

Analysis of experimental data reveals the sample with the most harmonious organoleptic parameters, which received the highest average score from tasters (sample 1). The composition of this sample includes the extracts of fennel, anise, caraway, and dill in the proportions of 25:45:25:5%, respectively. The tasters mark the pronounced, memorable aroma and smell of this composition.

4. CONCLUSION

The study experimentally establishes the adjustable technological parameters of plant raw material extraction using reduced vacuum (p=0.2 MPa, T=50°C, t=45 min, 40% aqueous alcoholic solution, particle size of raw plant materials 2-3 mm) when preparing a composition of extracts for functional syrups. The optimal ratio of extracts, according to organoleptic indicators, is 25:45:25:5,% of extracts of fennel, anise, caraway, and dill, respectively.

The obtained sample (composition) demonstrates pronounced organoleptic properties.

ACKNOWLEDGMENTS

The work was carried out within the framework of government research under the project part of the state assignment on the topic "Screening of plant-based bioactive compounds with geroprotective properties and the development of a technology for obtaining anti-aging nutraceuticals" (topic number FZSR-2020-0006), additional agreement to the Agreement on Subsidy Granting №075-03-2020-097/1 of March 2, 2020.

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