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DEVELOPMENT OF CONCENTRATED AROMATIC EXTRACTS FROM CURRANT SHOOTS FOR BEVERAGES

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Abstract. Herbal spicy-aromatic raw materials are widely used in the production of non-alcoholic and alcoholic beverages. They enrich beverages by aromatic compounds, create colour and impart a characteristic taste. Research on the chemical composition of young blackcurrant shoots, which are mainly agricultural waste but are a valuable source of aromatic and biologically active compounds, is perspective. The article is focused on the development of technology for concentrated aromatic extracts from young blackcurrant shoots for beverages. The research materials were young shoots of black currant (*Ribes Nigrum L.),* water-alcohol extracts and concentrated aromatic extracts from black currant shoots. The research showed that extracting crushed young blackcurrant shoots by a water-alcohol solution makes it possible to obtain an extract enriched by phenolic substances with a pleasant currant aroma and additional descriptors. Concentrating the water-alcohol extract by thermal vacuuming at a temperature of 50 ±2 °C and a pressure of 10 kPa to a dry matter content of 65% allows two concentrates to be obtained – extractive and aromatic substances. The concentrated extract and aromatic concentrate can be used as independent products of commercial value or in the form of a concentrated aromatic extract. It has been established that the aromatic profile of the concentrate is formed by a complex of monoterpenes, among 3-carene (23%) dominates, giving it fruity and floral notes with woody and coniferous shades. The significant contributions are made by caryophyllene (16.8%), terpinolene (9.64%), β-phellandrene (6.4%) and geraniol (6%), which provide a harmonious aroma with clove-woody, spicy, green, citrus-mint and rose notes. The combination of concentrated extract with aromatic concentrate allows to obtain a composite flavouring with a pronounced taste and aroma profile. In addition to its intense organoleptic characteristics, extract concentrate from the blackcurrant shoots is a source of biologically active compounds, in particular a phenolic complex, which determines its additional functional value in beverage technology.

Keywords: extract, black currant, shoots, concentrated flavouring, beverages, aromatic substances, phenolic substances, biological value, chromatography.

Introduction. Formulation of the problem

The modern beverage market has a stable tendency to expand its assortment due to products with improved functional properties and original taste and aroma profiles [1].

Plant spice and aromatic raw materials are widely used in the technology of non-alcoholic and alcoholic beverages. They enrich beverages by substances of the aromatic complex, create colour and give a characteristic taste [2, 3, 4].

In this context, it is important to search for and study new sources of aromatic and biologically active substances, in particular among the spice and aromatic raw materials of Ukrainian flora, that remain insufficiently studied [2].

Blackcurrant (*Ribes Nigrum L.)* is a widespread crop cultivated throughout the territory of Ukraine. Blackcurrant berries and buds are widely used in the production of alcoholic and non-alcoholic beverages – in the technology of juices, tinctures, fruit and berry wines and spirits, while blackcurrant shoots are mainly horticultural waste, although they contain essential oils enriched by phytoncides, polyphenols and are a source of vitamins, minerals that have a positive effect on the human body, increasing its resistance to negative external factors [5–7].

Therefore, development of extracts from blackcurrant shoots, which are mainly agricultural waste but are a valuable source of aromatic and biologically active compounds, is a perspective area of research [6, 7].

Analysis of recent research and publications

The use of infusions and decoctions from blackcurrant leaves, buds and shoots in folk medicine for various diseases of the blood vessels, bladder, liver, bile ducts, rheumatism and colds is widely known due to their high content of essential oils, phytoncides and tannins [3, 5, 8]. Currant leaves are also used in vitamin preparations. In cosmetology, currant berries, leaves, buds, roots, shoots and bark are used for therapeutic purposes.

They contain many vitamins, polyphenols, flavonoids, phytoncides and mineral salts [6, 7, 9]. Currant shoots, but mainly buds, are used to prepare an ancient Ukrainian alcoholic beverage called kontabas – a tincture with a pronounced specific aroma and taste [10].

There are references in literature to the use of currant shoots and leaves for flavoring alcoholic beverages, although this is rarely done compared to the application of berries [2].

In recent years, several articles have been published on the use of anatomical parts of black currants as sources of active phytochemicals [6–8].

The polyphenolic composition of blackcurrant buds is very wide and characterized by the presence of cinnamic acids – chlorogenic, caffeic and ferulic acids, benzoic acid – gallic acid; quercetin and catechin. The authors noted that blackcurrant buds and shoots have a higher content of phenols and antioxidants than fully ripe berries [6]. Similar results were obtained by other scientists, which noted that extracts from the leaves of some berry crops had higher antioxidant activity than extracts from berries due to their higher content of phenolic compounds [12].

Blackcurrant berries and buds have the same component composition of terpene alcohols and terpenoids. The shoots contain essential oils consisting of mono- and sesquiterpenes. It is these volatile compounds that are responsible for the characteristic aroma of blackcurrants [12]. This aroma is described as a harmonious combination of expressive sulfuric, green notes and a fruity shade. It also has minty and pink notes, similar to geraniol, which is found in roses [13, 14].

Some authors indicate that blackcurrant bud essential oil is extremely rich in aromatic components and characterized by a set of descriptors that have woody, terpenic, fruity, sweet, citrus, herbaceous, pine, green, oily and musty tones [15, 16]. Studies have demonstrated the presence of valuable macro- and microelements in various components of the blackcurrant bush, including potassium, manganese, iron and others [17].

The leaves of berry plants contain minerals such as potassium (K), calcium (Ca), magnesium (Mg), phosphorus (P), sodium (Na) and iron (Fe), as well as trace elements such as copper (Cu), zinc (Zn), manganese (Mn) and boron (B) [6].

Although berries are the main source of vitamins [18], some studies also indicate the presence of vitamins in the shoots of various berry crops, in particular vitamin C [19].

Taking into account the rich chemical composition of the vegetative parts of black currants, it is reasonable to develop a technology for extracting from shoots of this crop and investigate phenolic substances, components of the aromatic complex, based on which it is possible to determine the prospects for their application in beverage technology.

The purpose of the research is to develop a concentrated aromatic extract from blackcurrant shoots for beverage production.

To achieve this objective, the following objectives were formulated:

1. To study the qualitative characteristics of blackcurrant shoots.
2. To prepare water-alcohol extracts from blackcurrant shoots.
3. To investigate the dynamics of the content of extractive and phenolic substances in the extract of blackcurrant shoots during infusion and evaluate their organoleptic characteristics.
4. To concentrate the extract by vacuuming, followed by adsorption of aromatic vapours on a column with adsorbents that are selective to specific aromatic compounds.
5. To determine the qualitative composition of the aromatic complex of the concentrated extract using gas chromatography analysis.
6. To justify the ratio of the aromatic and extractive parts of the extract.

Research materials and methods

The materials used in the study were young shoots of black currant (*Ribes Nigrum L.)*, water-alcohol extracts, and concentrated aromatic extracts from black currant shoots.

The shoots were collected in the spring, before budding and during sap flow. The collection process was combined with routine care for young plants.

At this time, the amount of biologically active and aromatic substances is highest. A water-alcohol solution with a concentration of 70±2% vol. was used as an extractant.

The shoots were grinded into pieces of 1.2±0.2 cm long and covered by a water-alcohol solution in the ratio of 1:10 for preparation of the water-alcohol extract in the hydromodule and left to infuse for 10 days at a temperature of 20±2 °C with periodic mixing. During extraction, a portion of the extract on 2, 3, 5, 7 and 10 days was taken for analysis of extractive substances and phenolic compounds. The extracts were filtered before analysis.

For the preparation of aromatic substance concentrate (part B) and extract concentrate (part A), filtered water-alcohol extracts were thermo-vacuum extracted at a temperature of 50 ±2 °C and a pressure of 10 kPa to a dry matter content of 65%. Next, the two concentrates were mixed to obtain a concentrated aromatic extract, where the proportion of part B ranged from 5 to 20%.

The content of extractive substances and the volume fraction of alcohol were determined using the generally accepted method: extractive substances were determined refractometrically in the remaining residue after distillation, and the volume fraction of alcohol was determined using an alcohol meter in the distillate.

Phenolic compounds were determined by colorimetry using Folin-Ciocalteu reagent, that is capable of oxidizing phenolic compounds to form a blue solution. The optical density was measured on a photoelectric colorimeter at a wavelength of 680 nm in a 10 mm cuvette. The mass concentration of phenolic substances was converted to gallic acid using a calibration graph, taking into account the dilution factor.

Organoleptic characteristics were determined using a descriptive method.

The aromatic profile of the concentrated aromatic extract was determined using a profile descriptor method of sensory analysis. Five descriptors were selected: clove, currant, wood, greenery and pine needle. The intensity of the descriptors was rated on a scale from 0 to 5, where 0 – means the descriptor is absent, 1 – slightly noticeable tones, 2 –weakly expressed, 3 –medium intensity, 4 – expressed and 5 – intense.

The qualitative composition of the aromatic complex of the concentrated extract was determined by gas chromatography [20].

Sample preparation and analysis

The sample was prepared before analysis: 20–25 cm³ of extract was evaporated under vacuum for 10 minutes. During this process, the aromatic compounds were adsorbed on a special column.

Next, thermodesorption was performed. The aromatic compounds were desorbed from the adsorption column in a thermodesorber at a temperature of 200 °C during 4 minutes. They then entered an analytical chromatographic column with a PEG-6000 stationary phase.

Gas chromatographic analysis

Gas chromatographic analysis was performed under the following conditions:

• Column temperature: programmed from 90 to 175 °C with step of 5 °C.

• Detector temperature: 200 °C.

• Detector sensitivity: $2 \cdot 10^{-10}$.

• Nitrogen flow rate: 33 cm³/min.

The quantitative content of aromatic substances was determined by the internal standard method using menthol (1 mg/dm³). Compounds were identified by their relative retention times.

The regimes for concentrating the extract of aromatic raw materials with the capture of aromatic substances from water-alcohol vapours by adsorption using an adsorption column are given in Table 1 [21].

Table 1 – Concentration regimes of extract from young shoots of black currant *(n=3, p≤0.05)*

Regimes	Values
Volume of blackcurrant shoots extract, cm^3	200.0
Concentration by mass, %	65±2.0
Evaporation temperature + adsorption, °C	50.00±2.0
Residual pressure, kPa	10
Evaporation duration, min	10
Adsorption column	Polysorb-1, Tenax-GC, BAU-A
Volume of water-alcohol distillate, cm^3	105.0…145.0

The content of aromatic substances in the aroma concentrate was evaluated based on the results of chromatographic analysis obtained using a universal method with NF 20% Dinonyl Fhthalate [22].

In the Table 2 presented the material balance of the evaporation of water-alcohol extracts from young blackcurrant shoots.

Table 2 – Material balance of evaporation of extracts from young shoots *(n=3, p≤0.05)*

Shoots	Utilized		Obtained			Losses, %
	Extract		Concentrate		Steam + aromatic substances	
	sm^3	*DS,%	sm^3	*DS,%		
Blackcurrant	200.00	3.52±0.22	51.4±2.1	65±2.0	146.6±2.8	4.8
*DS – mass concentration of dry substances, %						

The capture of aromatic substances during the concentration of extract from young blackcurrant shoots proceeds in an adsorption column, in that three adsorbents are sequentially arranged along its entire length. The first layer is Polysorb-1, followed by Tenax-GC and the last layer is BAU-A. There are no boundaries between the adsorbents in the column, that ensured the free migration of aromatic compounds.

Statistical data processing. All results obtained were processed with using Excel from the Microsoft Office 2007 software package. The experiment was conducted in three repetitions. The data are presented as mean values ± standard deviation.

Results of the research and their discussion

The authors analysed the quality indicators of young blackcurrant shoots. They were completely greenish-brown in colour, flexible and smooth to the touch, without any damage from viruses or pests, without disease and had a characteristic currant aroma, that was more intense after rubbing. The buds along the shoots were round or egg-shaped form with slightly lighter in colour. Table 3 shows the dynamics of changes in the content of extractive and phenolic substances in the extract of black currant shoots during infusion.

The data in Table 3 demonstrated that intensive accumulation of extractive and phenolic compounds occurs during five days of extraction, after which the increase in the concentration of the tested substances was insignificant and amounted to about 1% for each subsequent day of infusion. In addition, the tasters noted that an increase in the duration of extraction resulted in the appearance of an unpleasant bitter and astringent taste.

Therefore, in further studies, a five-day infusion extract was used. The next stage of the work was focused on concentrating the obtained extract and developing a technological scheme for a concentrated aromatic extract. Three material flows are formed during the concentration of water-alcohol extracts from the shoots of berry bushes: concentrated extract, aroma concentrate and water-alcohol distillate.

Technological scheme for concentrated aromatic extract from young blackcurrant shoots presented in Fig. 1.

Table 3 – Dynamics of changes in the content of extractive and phenolic substances in the extract from young blackcurrant shoots during infusion *(n=3, p≤0.05)*

Name of substance class	Infusion duration, days					
	Initial	2	3	5	7	10
Extractives, %	0	1.78±0.24	2.84±0.56	3.52±0.84	3.55±0.12	3.58±0.12
Phenolic, mg/dm^3	0	239.5±2.4	571.8±4.8	774.0±6.3	782.95±6.6	790.08±6.2

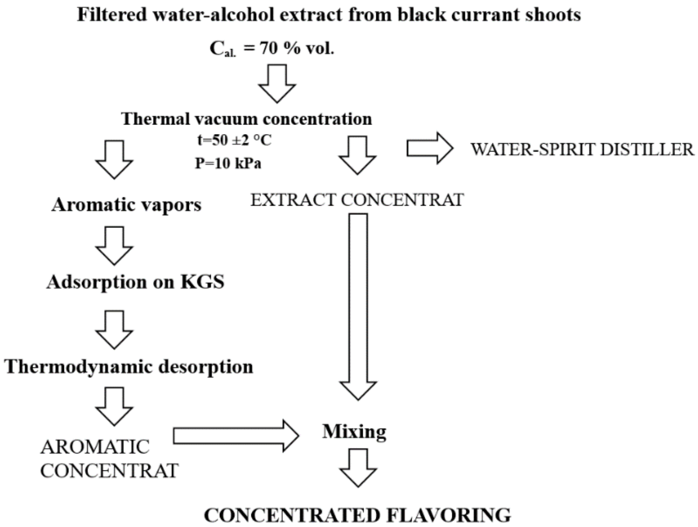

Fig. 1. Technological scheme of concentrated aromatic extract from blackcurrant shoots

Concentrated aromatic extracts consist of two parts: an extractive part and an aromatic part. The extractive part (concentrated extract, part A) is obtained by vacuum evaporation of blackcurrant shoots extract. The aromatic part (aroma concentrate, part B) is a concentrate of aromatic substances captured from the vapours during the concentration of blackcurrant shoots extract. The aroma concentrate is intended for repeated injection into the concentrated extract to enrich the concentrated extract by the original aroma of blackcurrants.

The flexibility of the proposed scheme consists in obtaining extractive and aromatic concentrates separately and their combination in different ratios that expands the range of choices for various technological solutions.

Table 4 shows the organoleptic characteristics of the extract concentrate from young blackcurrant shoots. In addition to the rich blackcurrant notes, pleasant tones of wood and spices were noted, that enriched the aroma of the extract.

Table 4 – Organoleptic characteristics of concentrate extracts from young blackcurrant shoots

Indicator	Characteristics
Appearance	homogeneous viscous liquid
Colour	brown
Taste	spicy, slightly tart, pleasant
Aroma	intense currant, with pleasant tones of wood and spices

The qualitative composition and quantitative content of substances in the aromatic complex in the aroma concentrate were evaluated using chromatograms (Fig. 2) obtained using a universal method with NF 20% Dinonyl Phthalate [22].

Experimentally, 18 aromatic components were identified in the aroma concentrate of black currant shoots. The aroma is mainly formed by a complex of monoterpenes, that is in agreement with the literature data [10]. The major component of the aroma concentrate of black currant shoots is the monoterpene 3-carene (23±3.5%), that gave it fruity and floral notes with a characteristic note of wood and pine needle [11, 12]. Next in terms of quantitative content is caryophyllene (16.8±2.6%), that gives the aroma of cloves and wood; terpinolene (9.64±1.7%), which is distinguished by its spicy character with a shade of greenery; β-phellandrene (6.4±2.5%), that has a citrus-spicy aroma with a slight minty freshness; and geraniol (6±1.67%), which gives a light rose aroma. We also identified and determined the amount of α-pinene (4.5 ± 1.0%), β-myrcene (0.6±0.5%), limonene (3.3±1.2%), linalool (3.6±1.22%) and esters represented by ethyl acetate and ethyl butyrate.

The efficiency of adsorption-desorption cycles on a gradient selectivity column has been confirmed by: an indicator $P^{ADC}_{aver.}$ (average value of the absolute reproducibility coefficient of the process) at the level of 99.68%; preservation of the initial ratio of aromatic substances, which is a guarantee of restoring the natural aroma; the desorption temperature range of aromatic substances T_{des} – 220...250 ºС without local overheating of successive layers of adsorbent; the rational duration of desorption of aromatic substances without chemical transformations under the action of excessive temperatures; the efficiency of the organization of adsorption-desorption processes.

The final stage of the work was to establish the optimal ratio of concentrated extract and aroma concentrate based on studies of the sensory profile of their mixture (Fig. 3).

Fig. 3 shows that the leading descriptor of the aroma of concentrated aromatic extract is currant tones. With the addition of 5% aroma concentrate, a weak intensity of woody notes was detected and shades of cloves, pine needles, and greenery were almost imperceptible..

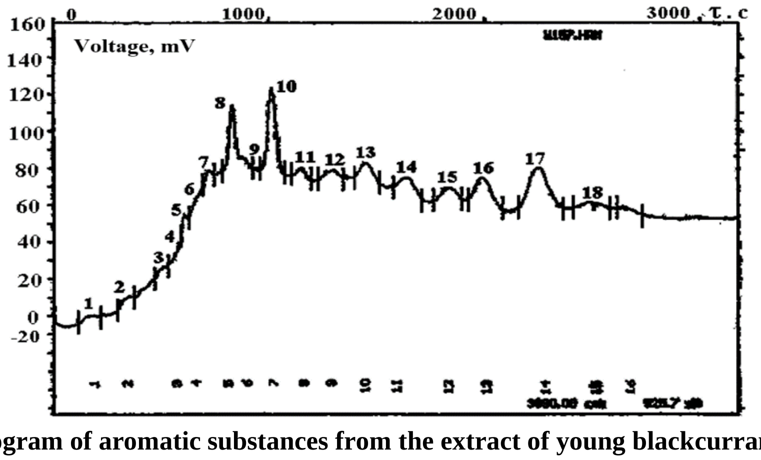

Fig. 2. Chromatogram of aromatic substances from the extract of young blackcurrant shoots

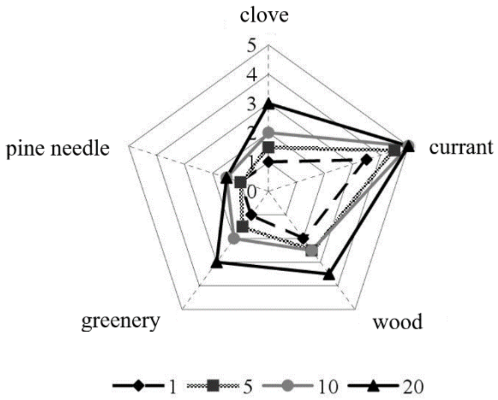

Fig. 3. Aroma profiles of concentrated aromatic extract depending on the proportion of aroma concentrate used, %

With an increase in the proportion of aromatic extract, the intensity of all descriptors increases. The addition of 20% aroma concentrate enriches the aromatic profile by the new original shades, that expands its application possibilities in beverage technology for assortment formation.

In the Table 5 presented the physicochemical parameters of the concentrated aromatic extract.

Table 5 – Physicochemical indicators of concentrated aromatic extract from young blackcurrant shoots

Indicators	Values
Density, g/cm³	0.87
Refractive index, at 20 °C	1.464
Angle of rotation of the surface of polarization, degrees (αD)	+10
Acid number, mg KOH per 100 cm^3	1.0

The determined values of density and refractive index of aromatic concentrates confirm the absence of structural modifications of aromatic compounds that may occur during the process of extract concentration or during thermodynamic desorption. The angle of rotation of the polarization surface indicates the presence of optical isomers in the aroma concentrates that are absent in synthetic analogues, confirming their natural origin.

Conclusion

Blackcurrant shoots were studied as a source of biologically active, in particular phenolic and aromatic compounds for the production of natural concentrated aromatic extract for beverages. It was found that young blackcurrant shoots (Ribes Nigrum L.) are enriched by phenolic and aromatic compounds and the extract obtained from them by maceration in a water-alcohol mixture with a concentration of 70% vol. for 5 days contains 774.0±6.3 mg/g of phenolic compounds, that indicates its biological value.

The technological scheme for obtaining a concentrated aromatic extract from young blackcurrant shoots, that is a source of a complex of extractive substances and can be used in beverage recipes, has been created.

The products obtained – concentrated extract (part A) and aromatic concentrate (part B) – can be used as independent products of commercial value or as a concentrated aromatic extract.

Investigation of the aromatic extract (part B) allowed to establish that currant shoots have a rich currant aroma with additional descriptors of wood and spices and research into its qualitative composition made it possible to establish that this aroma is due to a set of monoterpenes, mainly 3-carene (23%), that determines its fruity-floral currant note with woody-coniferous undertones. Caryophyllene (16.8%) with clove and woody tones, terpinolene (9.64%) with spicy and green tones, β-phellandrene (6.4%) with citrus-spicy and minty tones and geraniol (6%), which gives it a characteristic rose aroma. The combination of these components determines the original and harmonious aroma of the extract, suitable for use in beverage technology.

Combining the concentrated extract with the aromatic concentrate (A+B) into a composite concentrated flavouring extends the range of natural flavourings with an original taste and aroma profile. In addition to its intense original aroma, blackcurrant shoots extract concentrate is a source of a complex of biologically active compounds, in particular a phenolic complex, that will be useful in beverage recipes.

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РОЗРОБКА КОНЦЕНТРОВАНИХ АРОМАТИЧНИХ ЕКСТРАКТІВ ІЗ ГІЛОЧОК ЧОРНОЇ СМОРОДИНИ ДЛЯ НАПОЇВ

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Анотація. Рослинна пряно-ароматична сировина має широке використання в технології безалкогольних та алкогольних напоїв. Вона збагачує напої речовинами ароматичного комплексу, створює колір та надає характерного смаку. Перспективними є дослідження направлені на вивчення хімічного складу молодих гілочок чорної смородини, які переважно є відходом сільського господарства, але є цінним джерелом ароматичних та біологічно активних сполук. Статтю присвячено розробленню технології концентрованих ароматичних екстрактів із молодих гілочок чорної смородини для напоїв. Матеріалами досліджень були молоді пагони чорної смородини (*Ribes Nigrum L.*), водно-спиртові екстракти та концентровані ароматичні екстракти із гілочок чорної смородини. В результаті проведених досліджень було встановлено, що екстрагування водно-спиртовим розчином подрібнених молодих гілочок чорної смородини дозволяє отримати екстракт збагачений фенольними речовинами та з приємною смородиновою ароматичною з додатковими дескрипторами. Концентрування водно-спиртового екстракту термічним вакуумуванням за температури 50 ±2 ^{o}C і тиску 10 кПа до вмісту сухих речовин 65 % дозволяє отримати два концентрати – екстрактивних та ароматичних речовин. Концентрований екстракт та ароматичний концентрат можуть використовуватися як самостійні продукти комерційної цінності або у вигляді концентрованого ароматичного екстракту. Встановлено, що ароматичний профіль концентрату формується комплексом монотерпенів, серед яких домінує 3-карен (23 %), що зумовлює фруктово-квіткові відтінки з деревно-хвойними нюансами. Значний внесок роблять каріофіллен (16,8 %), терпінолен (9,64 %), β-фелландрен (6,4 %) та гераніол (6 %), які забезпечують формування гармонійного аромату з гвоздично-деревними, пряними, зеленими, цитрусово-м'ятними та трояндовими відтінками. Поєднання концентрованого екстракту з ароматичним концентратом дозволяє отримати композиційний ароматизатор із виразним смакоароматичним профілем. Окрім інтенсивних органолептичних характеристик, концентрат екстракту гілочок чорної смородини є джерелом біологічно активних сполук, зокрема фенольного комплексу, що визначає його додаткову функціональну цінність у технології напоїв.

Ключові слова: екстракт, чорна смородина, гілочки, концентрований ароматизатор, напої, ароматичні речовини, фенольні речовини, біологічна цінність, хроматографія