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SUBSTANTIATION OF ULTRASOUND-ASSISTED EXTRACTION PARAMETERS FOR OBTAINING AN EXTRACT FROM ROSE HIPS *(ROSA CANINA* L.)

Abstract: *The article presents the results of a research of the influence of extraction agents with different ethanol concentrations (30%, 40%, 50%, 60%, 70%, 80% and 96%) on the yield of extractives and found that the highest yield of extractives have been obtained when using 40% ethyl alcohol as an extraction agent. Therefore, this concentration has been chosen as the main extractive agent for further experiments. Ethanol, in particular, is a widely used extractive agent for plant materials due to its ability to extract a wide range of compounds, including phenolic compounds, flavonoids and organic acids. In recent years, ultrasonic-assisted extraction has gained attention due to its ability to improve extraction efficiency, reduce extraction time, and minimize the use of solvents, making it an environmentally friendly and cost-effective method. The optimal parameters of ultrasonic-assisted extraction have been established for obtaining an alcoholic extract from hips of Rosa canina (RosaCanina L.) – the highest yield of extractives was obtained with extraction duration of 30 minutes, extraction temperature was 50^{0}C, ultrasound power was 35 kHz. It is possible to obtain the maximum amount of biologically active compounds from raw materials by optimizing these parameters, which leads to a higher quality of the final product.*

Key worlds: *rose hips, extractive agent, yield of extractives, ultrasonic-assisted extraction, extraction parameters.*

Introduction

The fruits of various wild fruit and berry plants are used as non-traditional sources of plant raw materials in food production. The shift in priorities towards the use of wild plants is associated with their ability to synthesize and accumulate simultaneously hundreds, or even thousands of biologically active substances (BAS), which determines the effect of multiple effects on the human body and the formation of multi-vector technological properties. Also, being fundamentally closer in nature to the human body than synthetic drugs, they are characterized by higher safety indicators [1-3]. Plants serve not only as a direct source of food, but also as technological raw materials for processing traditional food products – obtaining original taste and maximum benefits [4].

Our country is rich in many medicinal plants, which are found both in wild and cultivated forms. About 18 000 species of wild plants grow within the territory of Kazakhstan. Wild plants are the richest sources of vitamins (vitamin C, provitamin A-carotene, etc.), carbohydrates, fats (fatty oil), proteins, organic acids (citric, malic, etc.), aromatic substances, antioxidants, which the human body urgently needs. Plants and their parts, containing more than 15% of a person's daily physiological need for biologically active substances, can be used as functional ingredients in the production of functional food products, which today form the basis of therapeutic and preventive nutrition for the population. Medicinal plants in the human diet, along with their nutritional function, are also important, performing a comprehensive defense of the body, represented by an antioxidant system of vitamins, polyphenols and microelements, and also play a primary role in cleansing the body of various toxic substances, due to the presence of dietary fiber and they contain pectin [5-8].

Thus, the use of wild plant materials makes it possible to improve the taste range of products and enrich them with biologically active substances. Among wild fruit plants, rose hips are of particular interest; they have not only high taste properties, but also a wide range of physiological effects, due to the rich set of biologically active substances they contain, including those with antioxidant properties.

Rosehip (*Rosa canina* L.) is a fruit of the *Rosaceae* family, which contains a large amount of phenolic compounds, carotenoids, tocopherols, flavonoids and vitamin C. In addition, rosehip contains other vitamins (A, B1, B2, K), minerals (calcium, phosphorus, potassium), carbohydrates (pectin), tannins and essential oils. Due to its rich bioactive compounds, rose hips are commonly used to prevent and treat several diseases such as colds, gastrointestinal disorders, infections and diabetes. Rose hips are widely used in cosmetics and pharmacology, and in the food industry, rose hips are used as marmalade, nectar, jam, syrups and dyes [9]. The effectiveness of consuming rose hips to reduce the risk of cardiovascular diseases, as well as to prevent vitamin C deficiency, is shown in the work [10]. The antioxidant activity of *Rosa canina* L fruits extract has been documented in several studies. The antioxidant and anti-inflammatory effects of *Rosa canina* L. are consistent with its clinical effects - especially given new data on the pharmacological picture of osteoarthritis [11-13].

In terms of vitamin C content (in seeds – 4,8%, in pulp – up to 8,5%), it has no equal among fruit and berry crops. In addition, rose hips contain P-active compounds (up to 9%), vitamin E (6-10 mg / 100 g), B1, B2, B9, carotene, tannins, pectin, nitrogen compounds, flavonoids, sugar, organic acids, fats and many microelements of the hematopoietic complex: Fe, Mg, Ca, K, Cu, Zn. The seeds contain up to 12% fatty acids, rubixanthin, gazaniaxanthin, β-cryptoxanthin and zeaxanthin and phenolic compounds such as quercetin, ellagic acid, quercetin glycosides, hydroxycinnamic acids, proanthocyanidin, aglycones [14]. The work [15] established a high content of biologically active compounds, primarily ascorbic acid (from 6.0 to 8.2 mg g^{-1} of live weight (FW)), flavonols (427.9 ± 0.4 $\mu g g^{-1}$ FW) and antioxidant activity.

Rose hips (Rosacanina L.) are becoming increasingly popular due to their bioactive components. Rosehip is distinguished by its antioxidant, immunomodulatory and anticancer properties. However, the abundance of these bioactive substances determines its tart taste, as a result of which it is consumed mainly in processed form [16].

The introduction of minor components of fruits and berries in the form of extracts into biologically active dietary supplements accelerates their absorption in the gastrointestinal tract while maintaining functional properties [17]. Recently, there has been increasing interest in bioactive compounds with properties beneficial to human health, which includes polyphenolic compounds and flavonoids. Therefore, the search for the most effective and environmentally friendly methods for extraction from natural products remains an urgent task. The efficiency of extraction of biologically active components from plant materials depends on various factors, such as extraction technology, nature of the solvent, time, temperature, module, i.e., the ratio of plant material and solvent, and many others [18]. However, optimal extraction technology is critical to ensure efficient extraction of target components from plant material. Over the past decades, several new extraction technologies have been actively introduced, including ultrasonic and microwave, which are energy-saving and environmentally friendly, producing high-quality extracts. Theoretically, the optimal extraction technology should be simple, safe, reproducible, inexpensive and suitable for industrial application [19].

Ultrasonic-assisted extraction is used to extract plant components. The ultrasonic-assisted extraction technique is especially attractive due to its simplicity and low cost of equipment. It is based on the use of energy obtained from ultrasound at frequencies above the human audible range (sound waves with a frequency above 20 kHz), facilitating the extraction of active substances from plant materials with a solvent. It has been established that the use of ultrasonic activation makes it possible to increase the yield of extractives in the resulting extract [20-22].

The purpose of this work is to determine the yield of extractives and substantiate the optimal parameters of ultrasonic-assisted extraction for obtaining an alcoholic extract from the fruits of the Rosa Canina (*RosaCanina* L.).

Research methods and conditions

Determination of the yield of extractives. About 3 g (exactly weighed) of crushed raw material passing through a sieve with a hole size of 1 mm is placed in a flask with a ground section, 50 ml of extractive agent is added, the flask is capped, weighed to the nearest 0.01 g and left for 1 hour. Then the flask is attached to reflux, it is heated, maintaining a low boil for 2 hours. The flask is cooled, stoppered, weighed and the loss in mass is replenished with extractive agent. The contents of the flask are thoroughly shaken and filtered through a paper filter into a dry flask. 25 ml of the filtrate is evaporated to dryness in a water bath in a dried and accurately weighed porcelain dish. The dry residue is dried in an oven at a temperature of (102.5 ± 2.5) ^{0}C to constant weight, then cooled in a desiccator for 30 minutes and it is weighed [23].

The content of extractives (X) as a percentage in terms of absolutely dry raw materials is calculated using Formula (1):

$$X = \frac{m \times 200 \times 100}{m_1 \times (100 - W)} \quad (1)$$

where: m – is mass of dry residue, g;
m_1 – is mass of raw materials, g;
W – weight loss when drying raw materials, %.
Weight loss on drying (W) was calculated using the following formula (1):

$$W = \frac{(m - m_1) \times 100}{m} \quad (2)$$

where: m – mass before drying, g;
m_1 – mass after drying, g.

Research results

Different concentrations of ethanol (30%, 40%, 50%, 60%, 70%, 80% and 96%) have been used as extractive agents to study their effect on the yield of extractives. The percentage yield of extractives was calculated based on the weight of the extracted material, and the results were rounded to the nearest 0.01. The data obtained during the determination are presented in Table 1, which provides an overview of the yields of extractives obtained using different concentrations of extractive agents.

Table 1 – Yield of rosehip extractives

Extractive agent	**Yield of extractives, %**
Ethanol 30%	34,85
Ethanol 40%	65,41
Ethanol 50%	40,94
Ethanol 60%	36,83
Ethanol 70%	54,11
Ethanol 80%	29,35
Ethanol 96%	23,02

Based on the data presented in Table 1, we can conclude that the highest yield of extractives has been obtained when using 40% ethyl alcohol as an extractive agent. Therefore, this concentration has been chosen as the main extractive agent for further experiments.

The process of extraction of plant materials is influenced by several factors that must be taken into account when choosing extraction conditions: anatomical structure, nature or degree of grinding of plant materials, concentration differences, temperature and duration of extraction, viscosity and nature of the extractive agent, surfactants and hydrodynamic layer of plant material.

The preparation of an alcoholic extract of Rosa Canina hips (*RosaCanina* L.) has been performed by solvent extraction in an ultrasonic bath. Extraction of samples has been performed under the following conditions (Table 2):

– Ultrasonic wave frequency was 35 kHz
– Extraction time was from 15 min to 75 min with an interval of 15 min
– Temperature was 50ºC.

From the data in Table 2 it can be seen that the highest yield of extractives have been obtained with extraction duration of 30 minutes.

Based on the research results obtained, we have established the optimal parameters of ultrasonic-assisted extraction for obtaining an alcoholic extract from the Rosa Canina hips (*RosaCanina* L.). The obtained data are shown in Table 3.

Table 2 – Effect of extraction duration on the yield of extractives

Wild growing raw materials (rose hips), gr	Extractive agent 40%, ml	Extraction duration, min	Yield, %
10	100	15	22,044
10	100	30	36,26
10	100	45	21,64
10	100	60	29,66
10	100	75	32,12

Table 3 – Optimal ultrasonic-assisted extraction parameters for obtaining alcoholic extract of the Rosa Canina hips (*Rosa Canina* L.)

Parameters	Optimal extraction value
Extraction time, min	30
Extraction temperature, ^{0}C	50
Solvent type	Ethanol 40%
Raw material/extractive agent ratio	1:10
Ultrasonic power, W	150
Ultrasonic frequency, kHz	35
Mains voltage, V	220
Total input frequency, W	280

Discussion of scientific results

The use of different ethanol concentrations made it possible to study the influence of these parameters on the yield of extractives from rose hips. Ethanol, in particular, is a widely used extractive agent for plant materials due to its ability to extract a wide range of compounds, including phenolic compounds, flavonoids and organic acids. The highest yield of extractives has been obtained when using 40% ethyl alcohol as an extractive agent.

The results obtained in this study provide valuable information on the yield of extractives from rose hips and can be used to optimize the extraction process to isolate bioactive compounds from this plant. By selecting 40% ethyl alcohol as the primary extractive agent based on the results of this study, researchers can optimize the extraction process and focus on identifying and characterizing the bioactive compounds contained in rose hips.

In recent years, ultrasonic-assisted extraction has gained attention due to its ability to improve extraction efficiency, reduce extraction time, and minimize the use of solvents, making it an environmentally friendly and cost-effective method. When studying the influence of ultrasonic-assisted extraction parameters on yield and composition, it was found that higher extraction temperatures above 50 °C destroyed the polyphenols in the extracts; The most effective are low frequencies in the ultrasonic power range below 40 kHz; the yield of polyphenols usually increases with increasing power, but with a threshold beyond which no significant increase is observed; Higher ultrasound power leads to the formation of free hydroxyl radicals, which destroy polyphenols, especially in the presence of high water content [24]. Thus, parameters affecting ultrasonic-assisted extraction include extraction time, extraction temperature, ultrasonic power, and liquid-to-solid extraction ratio. By optimizing these parameters, it is possible to obtain the maximum amount of biologically active compounds from raw materials, which leads to a higher quality of the final product.

Conclusion

Based on a research of the influence of extractive agents with different ethanol concentrations (30%, 40%, 50%, 60%, 70%, 80% and 96%) on the yield of extractives, it was found that the highest yield of extractives has been obtained when using 40% ethyl alcohol as an extractive agent. Therefore, this concentration has been chosen as the main extractive agent for further experiments.

The optimal parameters of ultrasonic-assisted extraction have been established for obtaining an alcoholic extract from Rose Canina hips (*Rosa Canina* L.) – the highest yield of extractives has been obtained with extraction duration of 30 minutes, extraction temperature was 50^{0}C, ultrasound power was 35 kHz. By optimizing these parameters, it is possible to obtain the maximum amount of biologically active compounds from raw materials, which leads to a higher quality of the final product.

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ИТМҰРЫН ЖЕМІСІНЕН ЭКСТРАКТ АЛУ ҮШІН УЛЬТРАДЫБЫСТЫҚ ЭКСТРАКЦИЯ ПАРАМЕТРЛЕРІН НЕГІЗДЕУ (ROSA CANINA L.)

Мақалада экстрактивті заттардың шығуына этанолдың әртүрлі концентрациясы бар (30%, 40%, 50%, 60%, 70%, 80% және 96%) экстрагенттердің әсерін зерттеу нәтижелері келтірілген және экстрактивті заттардың ең көп шығымы экстрагент ретінде 40% этил спиртін пайдалану кезінде алынғаны анықталған. Сондықтан бұл шоғырлану одан арғы эксперименттер үшін негізгі экстрагент ретінде таңдалды. Этанол, атап айтқанда, фенол қосылыстарын, флавоноидтар мен органикалық қышқылдарды қоса алғанда, қосылыстардың кең спектрін алу қабілетінің арқасында өсімдік шикізаты үшін кеңінен пайдаланылатын экстрагент болып табылады. Соңғы жылдары ультрадыбыстық экстракция экстракция тиімділігін арттыру, экстракция уақытын қысқарту және еріткіштерді пайдалануды барынша азайту қабілетінің арқасында назарды өзіне аударды, бұл әдіс экологиялық таза және экономикалық тиімді етеді. Ит итінің жемістерінен спирттік сығындыны алу үшін ультрадыбыстық экстракцияның оңтайлы параметрлері белгіленді (RosaCaninaL.) - экстрактивті заттардың ең көп шығымы экстракция ұзақтығы 30 минут, экстракция температурасы 50 0С, ультрадыбыстың қуаты 35 кГц болғанда алынды. Осы параметрлерді оңтайландыра отырып, шикізаттан биологиялық белсенді қосылыстардың ең көп санын алуға болады, бұл түпкілікті өнімнің неғұрлым жоғары сапасына әкеледі.

Түйін сөздер: *итмұрын, экстрагент, экстрактивті заттардың шығуы, ультрадыбыстық экстракция, экстракция параметрлері.*

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ОБОСНОВАНИЕ ПАРАМЕТРОВ УЛЬТРАЗВУКОВОЙ ЭКСТРАКЦИИ ДЛЯ ПОЛУЧЕНИЯ ЭКСТРАКТА ИЗ ПЛОДОВ ШИПОВНИКА (ROSA CANINA L.)

В статье приведены результаты исследования влияния экстрагентов с различной концентрацией этанола (30%, 40%, 50%, 60%, 70%, 80% и 96%) на выход экстрактивных веществ и установлено, что наибольший выход экстрактивных веществ был получен при использовании 40%-ного этилового спирта в качестве экстрагента. Поэтому эта концентрация была выбрана в качестве основного экстрагента для дальнейших экспериментов. Этанол, в частности, является широко используемым экстрагентом для растительного сырья благодаря его способности извлекать широкий спектр соединений, включая фенольные соединения, флавоноиды и органические кислоты. В последние годы ультразвуковая экстракция привлекло к себе внимание благодаря своей способности повышать эффективность экстракции, сокращать время экстракции и сводить к минимуму использование растворителей, что делает этот метод экологически чистым и экономически эффективным. Установлены оптимальные параметры ультразвуковой экстракции для получения спиртового экстракта из плодов шиповника собачьего (RosaCaninaL.) – наибольший выход экстрактивных веществ был получен при продолжительности экстракции 30 минут, температура экстракции 50^{0}С, мощность ультразвука 35 кГц. Оптимизируя эти параметры, можно получить максимальное количество биологически активных соединений из сырья, что приводит к более высокому качеству конечного продукта.

Ключевые слова: *шиповник, экстрагент, выход экстрактивных веществ, ультразвуковая экстракция, параметры экстракции.*

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