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LABORATORY TECHNOLOGY FOR EXTRACTION AND ANALYSIS OF VEGETABLE OILS

І. Ракіпов, О. Протункевич, Л. Пономарьова, І. Гайдаржи, Ю. Єпутатов. **Лабораторна технологія вилучення та аналізу рослинних олій.** Розроблено технологію холодного віджиму, екстракції та аналізу олії з кісточок льону, чорного кмину, винограду, шипшини, обліпихи та гранату. Визначено кислотне і йодне числа, досліджено молекулярну структуру та жирнокислотний склад екстрагованих зразків із застосуванням інфрачервоної спектроскопії та газорідинної хроматографії. Гексан і хлористий метилен залишаються найефективнішими екстрагентами для лабораторної екстракції в апараті Сокслета. Попередня мацерація подрібненої сировини сприяє збільшенню виходу ліпідів, що чітко простежується при екстрагуванні олії з насіння шипшини та обліпихи. Характеристики кислотного та йодного чисел свідчать про високий вміст ненасичених жирних кислот і низький ступінь гідролізу досліджуваних зразків. У складі виноградної, шипшинової, обліпихової та кминної олій переважає лінолева кислота, вміст якої становить від 41,2 ± 0,17% до 72,7 ± 0,06%. У лляній олії домінує ліноленова кислота (49,3 ± 0,04%), а у гранатовій – пунікова кислота (86,1 ± 0,28%). Водночас у складі лляної олії кількість лінолевої кислоти сягає лише 15,2 ± 0,04 %. Значна кількість ліноленової кислоти також виявлена у зразках олії з насіння шипшини (25,9 ± 0,11%) та кісточок обліпихи (27,8 ± 0,16%). Вміст олеїнової кислоти коливається від 4,3 ± 0,16% в олії з кісточок гранату до 25,1 ± 0,12% в олії з насіння чорного кмину. У досліджених зразках також виявлено пальмітинову (2,7 ± 0,07%...11,8 ± 0,00%), стеаринову (1,7 ± 0,03%...5,9 ± 0,01 %), арахінову (0,1 ± 0,00%...1,0 ± 0,03%) та *цис*-11-ейкозенову кислоти (0,1 ± 0,00%...0,7 ± 0,04 %). В інфрачервоних спектрах зразків олій спостерігаються піки, характерні для валентних і деформаційних коливань С–Н зв'язків метильних, метиленових і метинових груп; валентних і деформаційних коливань подвійних зв'язків С=С; валентних коливань карбонільних зв'язків С=О; і ефірних зв'язків С–О. Наявність характеристичних піків у діапазонах 938 см$^{-1}$ і 989 см$^{-1}$ в інфрачервоному спектрі олії з кісточок гранату свідчить про присутність кон'югованих подвійних зв'язків пунікової кислоти, яка є основною складовою цієї олії.

Ключові слова: рослинна олія, екстракція, хроматографія, жирнокислотний склад, інфрачервона спектроскопія

I. Rakipov, O. Protunkevych, L. Ponomarova, I. Gaidarzhy, Yu. Yeputatov. **Laboratory technology for extraction and analysis of vegetable oils.** A technology for cold pressing, extraction, and analysis of oil from flax seeds, black cumin, grapes, rose hips, sea buckthorn, and pomegranate has been developed. The acid and iodine values were determined, and the molecular structure and fatty acid composition of the extracted oil samples were investigated using infrared spectroscopy and gas-liquid chromatography. Hexane and methylene chloride were identified as the most effective solvents for laboratory extraction in a Soxhlet apparatus. Preliminary maceration of the comminuted raw material was found to increase the lipid yield, an effect that was particularly pronounced in the extraction of oil from rosehip and sea buckthorn seeds. The determined acid and iodine values indicate a high content of unsaturated fatty acids and a low degree of hydrolysis in the investigated oil samples. The fatty acid profile of grape seed, rosehip, sea buckthorn, and black cumin oils is dominated by linoleic acid, with its content ranging from 41.2 ± 0.17% to 72.7 ± 0.06%. In flaxseed oil, linolenic acid is the predominant fatty acid (49.3 ± 0.04%), whereas in pomegranate seed oil, punicic acid prevails (86.1 ± 0.28%). Conversely, the concentration of linoleic acid in flaxseed oil is only 15.2 ± 0.04%. Significant amounts of linolenic acid were also detected in the oil samples from rosehip seeds (25.9 ± 0.11%) and sea buckthorn seeds (27.8 ± 0.16%). The oleic acid content varied from 4.3 ± 0.16% in pomegranate seed oil to 25.1 ± 0.12% in black cumin seed oil. Furthermore, palmitic (2.7 ± 0.07%...11.8 ± 0.00%), stearic (1.7 ± 0.03%...5.9 ± 0.01%), arachidic (0.1 ± 0.00%...1.0 ± 0.03%), and *cis*-11-eicosenoic (0.1 ± 0.00%...0.7 ± 0.04%) acids were identified in the studied samples. The infrared spectra of the oil samples exhibit absorption bands characteristic of C–H stretching and bending vibrations of methyl, methylene, and methine groups; C=C double bond stretching and bending vibrations; C=O carbonyl stretching vibrations; and C–O ester bond vibrations. The presence of characteristic peaks in the regions of 938 cm^{-1} and 989 cm^{-1} in the infrared spectrum of pomegranate seed oil confirms the presence of conjugated double bonds of punicic acid, which is the principal component of this oil.

Keywords: vegetable oil, extraction, chromatography, fatty acid composition, infrared spectroscopy

Introduction

In recent years, there has been a substantial increase in interest regarding vegetable oils derived from the seeds of various crops. This heightened interest is attributed to their multifaceted value in both human health and diverse industrial applications. Global annual consumption of edible oils surpasses 170 million tons, unequivocally underscoring their dietary importance. Beyond their nutritional

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content, seed oils are rich in natural components such as lipids, proteins, fatty acids, chlorophyll, tocopherols, squalene, and carotenoids. These compounds exhibit numerous beneficial properties, including anti-inflammatory, anticancer, and antioxidant effects [1].

The escalating global demand for vegetable oils has amplified focus on their production methodologies. Product pricing is directly influenced by the efficiency and environmental sustainability of extraction technologies. In this context, traditional and mechanical pressing methods are gaining increasing popularity due to their ability to yield a high-purity final product. In addition to their primary application in the food industry, seed oils are extensively utilized as feedstock for biofuel production and other industrial commodities [2]. Furthermore, these resources are of particular interest in the pharmaceutical sector, where oils can function as emollient or antioxidant components within various cream formulations.

Of particular note is the prospective valorization of wine industry by-products, such as grape pomace. Grape seeds contain a multitude of valuable bioactive compounds, rendering their oil a potent natural antioxidant with antimicrobial properties for applications in the food industry [3]. Particular attention is paid to agro-industrial by-products, in particular to the seeds of soursop (Annona muricata L.), which contain a rich composition of phytochemical compounds and have therapeutic properties [4]. Similarly, rosehip oil (Rosa canina L.) holds significant scientific importance, recognized for its antioxidant, anti-inflammatory, hepatoprotective, and anticancer characteristics [5]. This renders plant oils highly sought after in the nutritional, cosmetic, and pharmaceutical domains.

The selection of the extraction method is pivotal, as it significantly influences oil yield, chemical characteristics, and volatile compound profiles, as evidenced by studies on musk melon seed oil [6]. Diverse extraction technologies can substantially alter the quality and composition of oils, affecting parameters such as acid and peroxide values, fatty acid ratios, bioactive compound content, and antioxidant activity levels.

One important area of research is waste recycling. By-products of the agro-industrial complex, which could previously be utilized, are now considered valuable sources for obtaining oils from seeds. Thus, the growing emphasis on waste recycling is becoming a key factor in finding and using new sources of seed oils [7].

Analysis of research publications

The most common extraction methods include traditional approaches such as Soxhlet extraction and cold pressing, as well as modern technologies including supercritical CO_2 extraction, ultrasound and microwave exposure. According to a study [6], the Soxhlet method provides the highest yield of oil from melon seeds, while simultaneously preserving their high antioxidant activity. The effectiveness of this method has also been confirmed by the example of oil extraction from red pepper seeds [8]. Another study [4] showed that agro-industrial waste can be a promising raw material for obtaining fixed oils with potential pharmaceutical value. A study [5] devoted to rose hips demonstrated that the use of supercritical CO_2 extraction allows for maximum extraction of biologically active components. It is also noted that the choice of extraction method affects the stability of oils during storage [9], and the fixation of biochemical characteristics directly depends on the method of obtaining lipids [10, 11].

Pre-treatment of raw materials further enhances the extraction process, increasing the quality and yield of oil. Pre-treatment alters the matrix, making the target compounds more accessible to the solvent or energetic action of subsequent advanced extraction and the nature of polar solvents affects the efficiency of oil extraction from plant materials [2].

Seed oils are complex matrices rich in both major and minor components that determine their nutritional and therapeutic value. Fig seeds (Ficus carica L.) are recognized as a valuable source of atypical vegetable oil, characterized by a high content of linolenic acid (from $18.11 \pm 0.255\%$ to $42.276 \pm 0.173\%$) and linoleic acid (from $27.75 \pm 0.019\%$ to $36.68 \pm 0.046\%$). The total oil content in fig seeds varies significantly depending on the genotype, from 6.69% to 39.97%. [12]. Pomegranate seed oil has been identified as a rich source of conjugated linolenic acid, particularly punicic acid (PA), an omega-5 fatty acid that accounts for a significant portion (approximately 73 wt %) of the total fatty acids [13]. The high content of polyunsaturated fatty acids (PUFA) in grape seed oil makes it particularly susceptible to oxidation, requiring careful storage to maintain its quality [7]. The high sensitivity of hemp seed oil to oxidative and photo-oxidative processes, even taking into account the presence of natural antioxidants, necessitates careful selection of materials, temperature conditions and lighting conditions to ensure the preservation of its quality throughout the entire shelf life [9]. Differ-

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ent plant varieties and geographic locations influence the chemical characteristics and composition of seed oils, as evidenced by studies of red pepper [8] and rosehip seed oils [14].

Modern analytical technologies play a key role in assessing the quality and safety of oils. The work [15] describes the effectiveness of infrared spectroscopy (FTIR), near infrared (NIR), fluorescence analysis and Raman spectroscopy. These methods allow non-destructive determination of such parameters as acid and peroxide values, oxidation state and presence of contaminants. Differential Scanning Calorimetry (DSC) used in combination with FTIR for rapid authentication of berry seed oils such as raspberry [16]. Gas chromatography-mass spectrometry (GC-MS) remains the gold standard for the identification and quantification of fatty acids and other volatile compounds in oils [17, 18].

The purpose and objectives of the research

Thus, the purpose of this work is to develop a laboratory technology for extracting vegetable oil using cold pressing and extraction in a Soxhlet apparatus methods. Seeds of flax, black cumin, grapes, rose hips, sea buckthorn and pomegranate were chosen as objects of research. To achieve this goal, it is necessary to solve the following tasks: prepare raw materials for oil production; extract oil; compare the extraction properties of solvents such as hexane, petroleum ether and methylene chloride; determine the acid and iodine number of the obtained samples; investigate the fatty acid composition using gas-liquid chromatography and determine features of the molecular structure obtained oils using infrared spectroscopy.

Materials and methods

Raw material preparation. For the cold pressing method, flax (Ukraine) and black cumin (India) oilseeds were used. For the Soxhlet extraction method, rose hips (Ukraine), sea buckthorn seeds (Ukraine), Odesskiy Chorny and Lydia grape seeds (Ukraine), pomegranate seeds (Azerbaijan), flaxseed and black cumin press cake were used. All raw materials used are commercially available in the market.

Flax, black cumin and rose hips seeds were used without additional processing, since the manufacturer recommended them for use without preliminary preparation. Odesskiy Chorny grape seeds were obtained as waste from winemaking. Sea buckthorn, Lydia grape and pomegranate fruits were thoroughly washed. The seeds were separated from the fruits, washed and dried. To obtain pure sea buckthorn seeds, juice was squeezed out of fresh-frozen sea buckthorn berries, and the remains of the mass were dried in a drying cabinet at a temperature of no more than 80 °C to achieve a minimum moisture content in the pulp. The seeds were taken out of the dried mass. Rose hips were dried at room temperature on gauze for 24 hours. The washed and dry seeds of the studied plants were ground in an electric mill. The ground seeds were sifted into three fractions: large 2.5 mm, medium 1.5 mm and small less than 0.5 mm.

Cold pressing. The pressing process was carried out using a screw oil press. The design of the device provides for the possibility of squeezing oil from flax and caraway seeds only after preliminary heating of the screw chamber. For this purpose, the required temperature, optimal for ensuring the process, is set on the control panel. Manufacturers recommend a temperature range of 180…230 °C for hard oilseeds. In all experiments, the heating temperature of the chamber was set at 200 °C. The countdown timer was set for 10 minutes of warming up to prevent the auger from turning on until the set temperature was reached. When the optimum temperature was reached, the switch was set to the “obverse” position and the auger shaft was turned on. Fresh flax and black cumin seeds were gradually loaded into the press in small portions through a funnel. After the pressing was completed, the oil and pulp were collected in appropriate containers. The oil temperature at the outlet was 42 °C. Then the oil was placed in a refrigerator for settling for three days at a temperature of 7 °C, after which the sediment was separated.

Extraction in a Soxhlet apparatus. Ground and pre-weighed seeds of grape, sea buckthorn, and pomegranate, as well as the press cake obtained from the mechanical pressing of flax and black cumin seeds, were placed into cellulose thimbles and loaded into a Soxhlet extractor. For the extraction experiments, methylene chloride, hexane, and petroleum ether were utilized as solvents. A volume of 250 mL of the solvent was added to a 500 mL round-bottom flask, which was heated using a water bath.

The solvent vapor was condensed by a reflux condenser, allowing the condensed solvent to percolate through the thimbles containing the raw material. Depending on the specific raw material and

solvent, the heating process was regulated to achieve 5-8 siphoning cycles per hour, with an observed increase in the duration of the final cycles.

To ensure a more thorough extraction of oil from rosehip and sea buckthorn seeds, the thimbles containing the ground material were subjected to an 8-hour maceration period in the corresponding solvent within the extractor's siphon prior to the start of the extraction process. Upon completion, the solvent was removed by distillation, and both the flask with the resulting oil and the thimbles with the spent meal were dried in an oven at 50 °C to a constant mass. The process was considered complete when the color of the siphoning solvent ceased to change, indicating the full depletion of extractable components from the raw material.

Determination of Acid Value. The acid value was determined using a standard laboratory method. A 100 mL mixture was prepared from two parts ethyl ether and one part ethyl alcohol, to which five drops of phenolphthalein were added. The resulting solution was neutralized with a 0.1 mol/L NaOH solution until a faint pink color appeared. A 3…5 g sample of the oil for analysis, weighed to an accuracy of 0.01 g, was placed into a 250 mL conical flask. Subsequently, 50 mL of the prepared alcohol-ether mixture was added. The flask's contents were stirred until the oil completely dissolved. Then, 3…5 drops of phenolphthalein were added to the solution. The resulting oil solution was shaken vigorously and rapidly titrated with a 0.1 mol/L potassium hydroxide solution until a stable, faint pink color, persisting for 30 seconds after stirring, was achieved.

Determination of Iodine Value. The iodine value was determined using the Kaufman method. For this, a solution of sodium bromide and bromine in methyl alcohol was prepared. A 1 g sample of vegetable oil was dissolved in 10 mL of chloroform. Then, 20 mL of the Kaufman solution was added from a burette. The flask was sealed with a stopper, and its contents were thoroughly mixed and left in a dark place for one hour. Following this, 10 mL of KI solution and 50 mL of distilled water were added to the flask. The liberated iodine was titrated with a 0.1 mol/L sodium thiosulfate solution until a straw-yellow color appeared. Then, 1 mL of freshly prepared starch solution was added, and titration continued until the blue color completely disappeared.

Gas-Liquid Chromatography of Samples. The fatty acid composition was analyzed using a Shimadzu 2010 chromatograph equipped with a flame ionization detector. A Thermo TR-FAME column was used, with helium as the carrier gas. A 1 µL injection volume was applied, and the thermostat temperature ranged from 140…220 °C, with a temperature gradient of 4 °C/min. Fatty acid methyl esters were obtained via a standard procedure. The oil sample under investigation was thoroughly mixed. Two to three drops of oil were pipetted into a glass test tube and dissolved in 2 mL of hexane. To this solution, 0.1 mL of a 2 mol/L sodium methylate solution in methanol was added. After intensive mixing for 2 minutes, the reaction mixture was allowed to settle for 5 minutes and then filtered through a paper filter. The chromatographic analysis time was 30 minutes.

Fourier transform infrared spectroscopy (FTIR). The molecular structure of the obtained oils was investigated using infrared spectroscopy. Infrared spectra of the oil samples were recorded on a PerkinElmer Frontier FTIR instrument in the wavenumber range of 4000…400 cm^{-1}.

Research results

To analyze the material balance of the cold pressing process, the container and raw materials were weighed at the initial stage, and the obtained products were weighed after the work was completed. The relative average oil yield for three experiments was $35 \pm 4.6\%$ for flax and $23 \pm 4.5\%$ for black cumin seeds. The main losses are associated with the remains of raw materials and oil in the auger chamber of the press.

A comparative analysis of the oil extraction yields from the ground seeds and press cake using methylene chloride, hexane, and petroleum ether indicates that hexane and methylene chloride are the most effective solvents in terms of product yield (Table 1).

The criteria for solvent selection in this extraction process were boiling point, which dictates the optimal extraction temperature, the solvent's ability to effectively dissolve the oil with subsequent regeneration, and its cost. "Green chemistry" principles [19] were not considered. Methylene chloride was chosen as a more convenient and accessible lipophilic extractant for oil recovery due to its lower boiling point compared to hexane and petroleum ether, which reduces the cost of its distillation.

To assess the quality of the obtained extraction oils, acid value and iodine value were determined. These parameters are crucial for both process control and for verifying the oils' compliance with standards and their suitability for specific applications.

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Table 1

Dependence of oil yield on the extractant used (average value for three extractions in % of the mass of loaded raw material and standard deviation from the average)

	Methylene chloride	Hexane	Petroleum ether
Rose hip	3.1 ± 0.1	2.5 ± 0.15	1.75 ± 0.15
Pomegranate	11.0 ± 0.2	11.5 ± 0.15	10.5 ± 0,2
Sea buckthorn	5.6 ± 0.15	5.7 ± 0.1	5.1 ± 0.1
Odessky black grapes	9.3 ± 0.2	10.1 ± 0.2	9.1 ± 0.15
Lidiya grapes	8.1 ± 0.2	8.4 ± 0.15	8.0 ± 0.2
Flaxseed press cake	3.2 ± 0.2	3.3 ± 0.2	2.9 ± 0.25
Black cumin press cake	2.4 ± 0.2	2.5 ± 0.2	2.3 ± 0.2

The acid and iodine values of oils obtained by extraction with methylene chloride and hexane are presented in Table 2.

Table 2

Acid and iodine values for extraction oils (average values based on the results of three titrations and standard deviation from the mean)

CH_2Cl_2 /C_6H_{14}	Acid value, mg KOH/g	Iodine value, g/100 g
Linseed oil	5.6 ± 0.3	165.1 ± 3.2
	5.7 ± 0.3	170.1 ± 2.9
Black cumin oil	1.7 ± 0.1	85.2 ± 2.4
	1.6 ± 0.1	84.5 ± 2.6
Grapeseed oil (Lydiya)	0.3 ± 0.1	145.4 ± 3.1
	0.4 ± 0.1	147.2 ± 3.2
Grapeseed oil (Odessa black)	0.4 ± 0.1	148.3 ± 3.1
	0.5 ± 0.2	146.2 ± 3.0
Pomegranate oil	7.4 ± 0.4	180.2 ± 3.7
	6.9 ± 0.4	181.3 ± 4.1
Sea buckthorn oil	11.2 ± 0.6	97.3 ± 2.8
	11.5 ± 0.5	96.1 ± 3.1
Rosehip oil	0.3 ± 0.1	180.1 ± 6.2
	0.4 ± 0.1	179.3 ± 4.2

Low acid values in the investigated oil samples are a positive indicator, signifying high oil quality and a low content of free fatty acids. Simultaneously, high iodine values for oil samples from flaxseed, grape, pomegranate, and rosehip indicate a high content of polyunsaturated fatty acids, such as linoleic and linolenic acids, and consequently, low oxidative stability for these oils.

As a result of chromatographic separation, 11 fatty acids methyl esters were identified in flaxseed, black cumin, and grape oils, 8 fatty acids in pomegranate oil, 10 fatty acids methyl esters in sea buckthorn seed oil and 13 fatty acids methyl estersin rosehip seed oil. The composition and content of fatty acids methyl esters for each respective oil are presented in Table 3.

As Table 3 shows, linoleic acid is the predominant fatty acid in rosehip, black cumin, grape, and sea buckthorn seed oils. In contrast, flaxseed oil and pomegranate seed oil contain significantly lower mass fractions of linoleic acid, at only 15.2 ± 0.04% and 4.1 ± 0.05% respectively.

Linolenic acid is most abundant in flaxseed oil (49.3 ± 0.04%). It is also present in substantial amounts in rosehip seed oil and sea buckthorn seed oil (25.9 ± 0.11% and 27.8 ± 0.16% respectively). However, its content in pomegranate, grape, and black cumin seed oils does not exceed 1.2 ± 0.01%.

Oleic acid is found in high quantities in almost all the investigated oils, with the exception of pomegranate seed oil, where its content is only 4.1 ± 0.16%. It is worth noting that palmitic (2.7 ± 0.07%...11.8 ± 0.00%), stearic (1.7 ± 0.03%...5.9 ± 0.01%), arachidonic (0.1 ± 0.01%...0.5 ± 0.01%), and *cis*-11-eicosenoic (0.1 ± 0.00%...0.7 ± 0.04%) acids are present in all the oils.

Notably, punicic acid is exclusively found in pomegranate seed oil, making up a significant 86.1 ± 0.28% of its fatty acid content. The content of other fatty acids in pomegranate seed oil ranges from 0.2 ± 0.04%...4.3 ± 0.15% (Figure 1).

Table 3

Average fatty acid content in oil samples (mass fractions in % of total content) and reproducibility of results for two injections

Component composition	Flax	Rosehip	Pomegranate	Sea buckthorn	Odessa black grapes	Lydia grapes	Black cumin
Myristic ($C_{14:0}$)	–	0.1 (0.01)	–	0.2 (0.12)	0.1 (0.02)	0.1 (0.01)	0.3 (0.02)
Pentadecanoic ($C_{15:0}$)	–	–	–	0.2 (0.02)	–	–	–
Palmitic ($C_{16:0}$)	5.7 (0.00)	3.8 (0.11)	2.7 (0.07)	8.2 (0.04)	6.9 (0.06)	8.7 (0.01)	11.8 (0.00)
Palmitinoleic ($C_{16:1}$)	0.1 (0.01)	0.1 (0.00)	–	0.6 (0.00)	0.7 (0.02)	0.2 (0.01)	0.2 (0.02)
Heptadecanoic ($C_{17:0}$)	–	0.1 (0.01)	–	–	0.1 (0.01)	0.1 (0.01)	0.1 (0.01)
Stearic ($C_{18:0}$)	5.9 (0.01)	2.4 (0.2)	1.7 (0,03)	3.1 (0.25)	2.9 (0.03)	2.7 (0.02)	3.3 (0.05)
Oleic ($C_{18:1n9c}$)	22.9 (0.02)	14.8 (0.11)	4.3 (0.16)	18.1 (0.08)	16.0 (0.00)	19.1 (0.01)	25.1 (0.12)
Linoleic ($C_{18:2n6c}$)	15.2 (0.04)	51.3 (0.19)	4.1 (0.05)	41.2 (0.17)	72.7 (0.06)	68.3 (0.00)	55.3 (0.03)
Punicic ($C_{18:3}$)	–	–	86.1 (0.28)	–	–	–	–
Linolenic ($C_{18:3n3}$)	49.3 (0.04)	25.9 (0.11)	0.2 (0,04)	27.8 (0.16)	0.3 (0.06)	0.4 (0.01)	1.2 (0.01)
γ-Linolenic ($C_{18:3n6}$)	0.2 (0.03)	–	–	–	–	–	–
Arachidonic ($C_{20:0}$)	0.2 (0.01)	1.0 (0.03)	0.4 (0,04)	0.5 (0.01)	0.1 (0.00)	0.1 (0.01)	0.2 (0.00)
Cis-11-eicosenoic ($C_{20:1}$)	0.1 (0.00)	0.3 (0.02)	0.7 (0.04)	0.2 (0,1)	0.2 (0.03)	0.2 (0.00)	0.3 (0.00)
Cis, cis-11.14- eicosadienoic ($C_{20:2}$)	–	–	–	–	–	–	2.3 (0.05)
Behenic ($C_{22:0}$)	0.2 (0.01)	0.2 (0.01)	–	–	–	–	–
Lignoceric ($C_{24:0}$)	0.1 (0.01)	0.1 (0.01)	–	–	–	–	–
Nervonic ($C_{24:1}$)	–	0.1 (0.02)	–	–	0.6 (0.02)	0.1 (0.01)	–

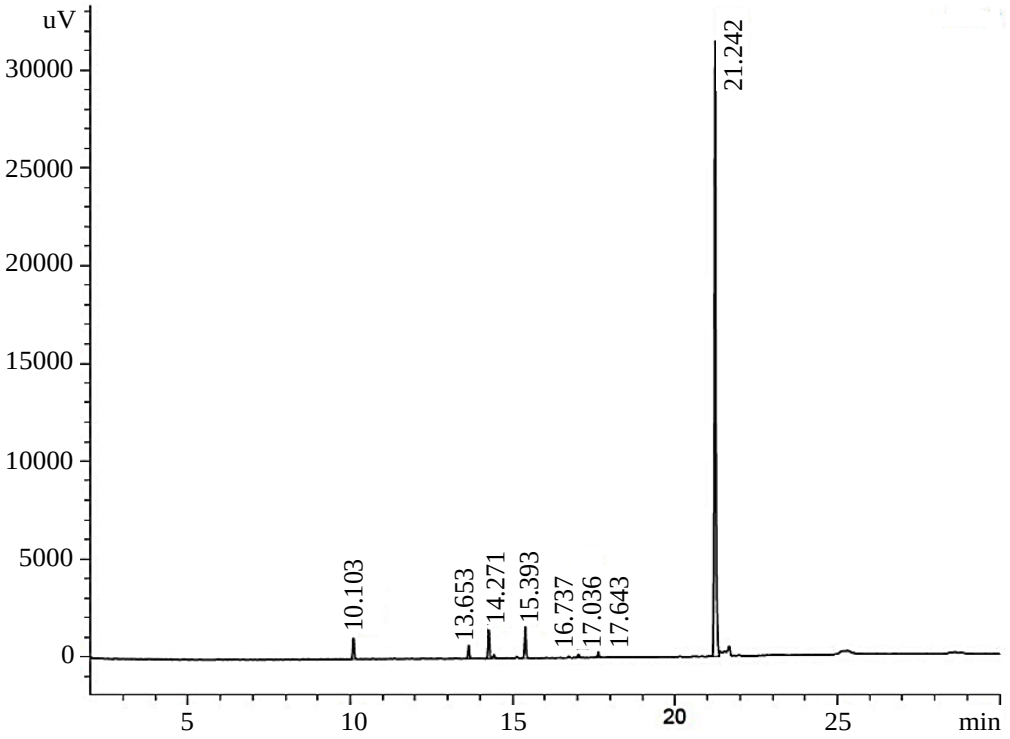

Fig. 1. Chromatogram of a sample of oil extracted from pomegranate seeds

Figure 2 presents the FTIR spectra of liquid films of the investigated oil samples dissolved in tetrachloromethane (764 cm^{-1} and 788 cm^{-1}). In the infrared spectra of all investigated oils, intense bands with maxima at 2956, 2928, and 2856 cm^{-1} are observed.

These bands can be attributed to the stretching vibrations of the C–H bond in the CH_3 (2962 ±10 cm^{-1}) and CH_2 (2926 and 2853 ±10 cm^{-1}) groups. The deformation (δ) vibrations of the C–H bonds in these groups correspond to bands with maxima at 1467 ± 10 cm^{-1} (δ_{asym} CH_2) and also 1378 cm^{-1} (δ_{sym} CH_3 and CH_2).

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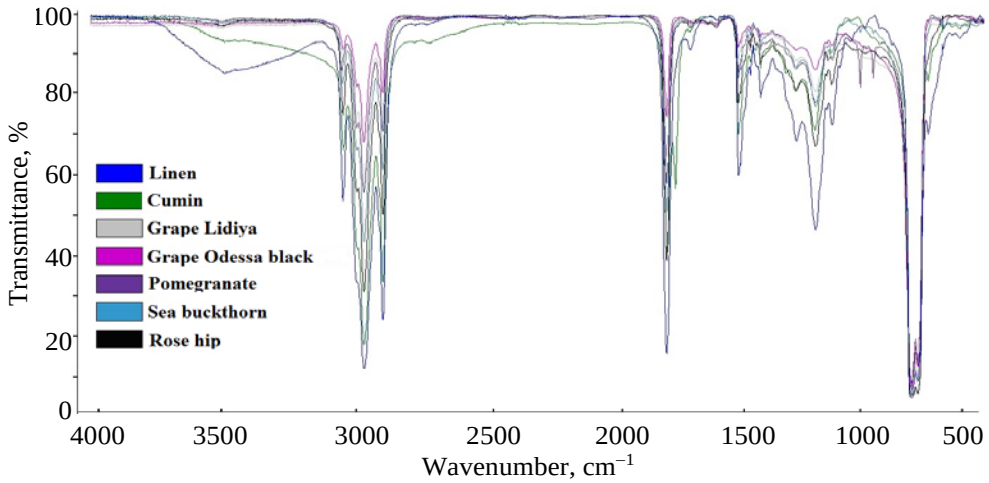

Fig. 2. FTIR-spectra (4000…400 cm^{-1}) of the investigated oil samples

Unsaturated hydrocarbon chains can be detected by the stretching vibrations of the C–H bond around 3011 cm^{-1} and deformation vibrations around 723 cm^{-1} of the –CH=CH– group. These bands do not overlap with the corresponding vibration bands of the saturated C–H bond in $–CH_2$ and $–CH_3$ groups, and their presence indicates the existence of a double bond (*cis*-form). It should also be noted that low-intensity bands of the in-plane deformation vibrations of the unsaturated C–H bond are observed in the 1419 cm^{-1} region.

Stretching vibrations of C=C bonds in the investigated samples are observed at characteristic absorption frequencies in the 1653 cm^{-1} region. In the 1746 cm^{-1} region, an intense peak corresponding to the stretching vibrations of the C=O bond is observed. The stretching vibrations of the C–O bond manifest as three characteristic peaks for triglycerides, with maxima around 1239, 1164, and 1100 cm^{-1}.

Characteristic absorption bands around 938 and 989 cm^{-1} in the spectrum of the pomegranate seed oil sample can be attributed to the deformation vibrations of two conjugated –CH=CH– bonds of punicic acid, which is the main component of pomegranate seed oil [13].

Conclusions

We successfully obtained oils from flaxseed, black cumin, rosehip, grape seeds (Lydia and Odesky Chorny varieties), sea buckthorn, and pomegranate using both cold pressing and extraction methods. The most effective approach proved to be extraction with methylene chloride and hexane in a Soxhlet apparatus. Notably, preliminary maceration significantly increased lipid yield in the cases of rosehip and sea buckthorn seeds.

The acid and iodine values indicate a low degree of lipid hydrolysis and a high content of unsaturated fatty acids in the obtained samples. Chromatographic analysis revealed that linoleic acid is the primary fatty acid in oils from rosehip, black cumin, grape, and sea buckthorn. In contrast, linolenic acid predominates in flaxseed oil, while punicic acid is dominant in pomegranate oil. A significant amount of linolenic acid was also observed in rosehip seed oil and sea buckthorn kernel oil.

Infrared spectra of the investigated samples show absorption bands corresponding to stretching and deformation vibrations of C–H bonds in methyl, methylene, and methine groups. Additionally, characteristic absorption bands for stretching and deformation vibrations of –CH=CH– double bonds were observed, along with bands corresponding to C=O and C–O group stretching vibrations. A unique feature in the infrared spectra of punicic acid is the presence of two peaks with characteristic absorption frequencies corresponding to the deformation vibrations of this acid's conjugated double bonds, which distinguishes it from its isomeric linolenic acid.

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