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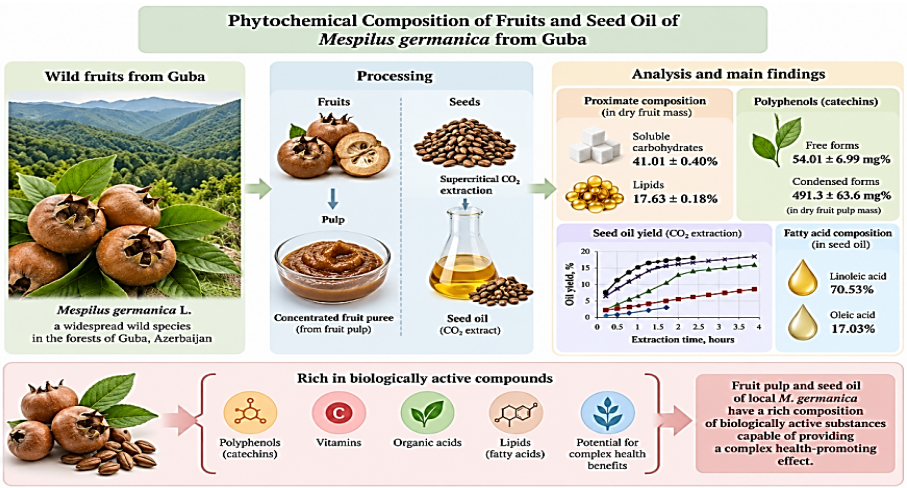
Phytochemical Composition of the Fruits and Seed Oil of Mespilus Germanica from Guba

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ABSTRACT: The effectiveness of using plant products for medicinal purposes should be confirmed by medical observations, or such use may be justified by experimental pharmacological data and chemical composition. This study aimed to provide a comprehensive phytochemical characterization of German medlar from a forest area near the district center of Guba. The analytical part of the work was carried out using standard chemical analysis methods. Integrated processing of medlar fruits was tested, yielding concentrated pulp purée and oil (CO_2 extract) from the seeds. The main results were the identification of specific features in the phytochemical profile of this local wild plant, a proposal to improve the rheological properties of concentrated purée, and optimization of the technological parameters of CO_2 extraction from the seeds. In the dry fruit mass, soluble carbohydrates (41.01±0.40%) and lipids (17.63±0.18%) predominated. The largest group of P-active compounds was catechins, with contents of 54.01±6.99 mg% in free forms and 491.3±63.6 mg% in condensed forms, relative to the dry mass of the fruit pulp. The maximum yield of the lipophilic fraction from dry, ground seeds (>18% of their dry mass) and the best quality were obtained by CO_2 extraction for 4 hours at 40 °C and a pressure of 200 atm. Linoleic (70.53%) and oleic (17.03%) acids predominated among the fatty acids. Overall, the pulp of local medlar and the lipophilic fraction of its seeds had a rich composition of biologically active substances that may produce a combined health-promoting effect.

GRAPHICAL ABSTRACT



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1. INTRODUCTION

In Azerbaijan, German medlar (Mespilus germanica L.), which local residents more often simply call medlar or "cones" (əzgil in Azerbaijani), is widespread in the wild. The areas occupied by its natural thickets in the country's forests cover thousands of hectares.

German medlar is a typical component of the understory and edges of Azerbaijan's broad-leaved forests. The largest natural stands are concentrated in the following areas:

• Greater Caucasus: forests of the Guba-Khachmaz, Sheki-Zagatala, and Ismayilli regions;

• Lesser Caucasus: mountain-forest areas of the Ganja-Gazakh zone and Karabakh;

• Talysh Mountains (Lankaran zone): in the humid subtropical climate, medlar grows particularly well and often forms dense thickets together with hawthorn and rosehip.

Wild fruits are actively collected by the local population. Since they are rather astringent when fresh, they are eaten after the first frosts or after prolonged storage, when they become soft and sweet. They are also used to make preserves, jams, fruit leather, and traditional sauces.

All parts of the plant (leaves, bark, and fruits) are rich in tannins, vitamin C, and organic acids, and have therefore long been used in Azerbaijani folk medicine.

As a component of natural forests, medlar plays an important role in preventing soil erosion on mountain slopes and provides a food source for forest birds and animals.

In folk medicine, medlar is widely used to treat gastrointestinal disorders (diarrhea), kidney diseases, bronchitis, and other illnesses. The literature reports high consumer appeal for food products made from wild medlar fruits [1–3].

Because freshly harvested wild medlar fruits have a very limited shelf life, they are often used in powder form, produced from whole fruits or their seeds (commonly called stones). The main advantage of powdered food supplements is their ability to reconstitute quickly when liquid is added. After reconstitution, these plant-based supplements are almost indistinguishable from supplements made from fresh fruits in their organoleptic properties (color, taste, and aroma). They can serve as physiologically active structuring components to balance the nutritional value of functional foods and can also be used as fillers or fortifiers [4].

As a multivitamin product [5], wild medlar fruits are usually eaten fresh. They can also be used to prepare preserved products, such as pickles [6].

Wild medlar fruits weigh 4–10 grams, of which 61–62% is pulp, up to 20% is seeds (up to five), and 18–19% is skin [7].

Genetic studies have shown that medlar is so evolutionarily close to hawthorns that, technically, it would be more appropriate to include it in the genus Crataegus [8].

Published data indicate a close relationship between the chemical composition of medlar fruits and the geographical area in which they grow [9–10]. It is therefore necessary to clarify the main indicators of the chemical composition and nutritional value of medlar fruits growing in Azerbaijan.

2. MATERIALS AND METHODS

2.1 Research Materials

The study used fruits of German medlar (Mespilus germanica L.) growing wild under the soil and climatic conditions of the lowland part of Guba District, Republic of Azerbaijan (the Gechresh forest area).

Wild medlar fruits were collected in late October and early November in 2021–2023.

At least 5 kg of freshly collected fruit was delivered to the laboratory, where it was stored at 2–4 °C before analysis and experimental processing until it softened slightly, but for no longer than one week.

For preparation of samples for chemical analysis, the fruits were passed through a stainless-steel sieve with 0.4 mm openings.

The skin and pulp accounted for an average of 60% of the total fruit mass.

The ground pulp was also used to make confiture. The following auxiliary materials, compliant with sanitary and technical standards, were used: granulated sugar (GOST 21–94), citric acid E330 (GOST 3652-69), and drinking water (GOST P 51232-98).

The individual seeds were dehydrated in a laboratory drying oven and ground into a fine powder in a disintegrator. Fixed oil was then obtained from this powder by supercritical-fluid extraction with carbon dioxide (CO_2) at pressures of 100–280 atm and a temperature of 40 °C.

2.2 Analytical Methods

The following chemical analysis methods were used in this work:

•Dry matter was determined by drying to constant mass according to GOST 33977-2016.

•Total soluble solids were determined refractometrically in accordance with GOST 28562-90.

•Reducing sugars were determined by the Bertrand method (GOST 8756.13-87).

•Titratable acidity was determined by titration with 0.1 N NaOH and expressed as citric acid equivalents in accordance with ISO 750:2013.

•Ascorbic acid was determined by iodometric titration according to GOST 24556-89.

•pH was measured potentiometrically according to GOST 26188-2016.

•Crude protein was determined by the Kjeldahl method using a nitrogen-to-protein conversion factor of 6,2

• Fixed oil (lipids) was determined by extraction according to GOST 8756.21-89.

• Physicochemical properties of the fixed oil (acid value, etc.) were determined by volumetric titration methods that are standard (GOST 31933-2012) or regulated by pharmacopoeias (e.g., the State Pharmacopoeia of the Russian Federation).

• Fatty acid composition was determined by gas–liquid chromatography according to GOST 30418-96.

•P-active substances were determined by absorption spectrophotometry (specifically, photocolorimetry), based on color reactions and measurement of the optical density of solutions containing the target substances. The higher the concentration of active phenolic compounds in a sample, the more intense the solution's color and the greater its optical density. The exact amount of a substance is calculated using the Beer–Lambert law and a previously prepared calibration curve (using standard solutions such as rutin, quercetin, or gallic acid).

• Macroelements were determined by flame photometry [11].

• Microelements were determined by emission spectral analysis [12].

• Structural and rheological properties of the products were determined using a rotational viscometer (Reotest-2) and a ST-2 structurometer [13].

• Total antioxidant activity (TAA) was determined by the amperometric method using a Tsvet-Yauza-01-AA instrument and quercetin as the reference substance [14].

2.3 Processing of primary data

Statistical processing of the primary data was carried out using the Statistica 10.0 package. For each test portion, the mean value from three parallel determinations and the standard deviation (SD) were calculated.

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3. RESULTS AND DISCUSSION

3.1 Nutritional and Biological Value of Medlar Fruits

3.1.1 Quantitative Composition of Nutrients

Table 1 shows the quantitative composition of the main nutrients of medlar fruits as a percentage of their raw and dry weight.

As can be seen, the main components of medlar fruits are soluble carbohydrates, lipids, nitrogen-containing substances, minerals, and organic acids, accounting for 41.01±0.40%, 17.63±0.18%, 13.31±0.13%, 7.19±0.07%, and 1.08±0.02% of the total dry mass, respectively.

Table 1 - Quantitative contents of the main nutrients in medlar fruits.

Nutrients	% of fresh mass	% of dry mass
Dry matter	27,80±1,00	-
Nitrogen-containing substances (N × 6.25)	3,70±0,04	13,31±0,13
Lipids	4,90±0,05	17,63±0,18
Soluble carbohydrates	11,40±0,10	41,01±0,40
Ash	2,00±0,02	7,19±0,07
pH	4,30±0,01	-
Organic acids (as malic acid)	0,30±0,03	1,08±0,02
p-value	≤0,05	≤0,05

In addition to these macronutrients, the dry residue of medlar fruits also contains dietary fiber- a complex consisting of protopectin, hemicellulose, cellulose, and lignin- as well as tannins, which give the fruits their characteristic astringent taste.

These data show that soluble carbohydrates (41.01±0.40% of dry mass) form the basis of the dry mass of medlar fruits and provide their energy and nutritional value. Lipids (17.63±0.18%) and nitrogen-containing substances (13.31±0.13%) make up a substantial proportion, indicating a well-balanced protein and fat composition. Mineral components and organic acids are present in smaller amounts (7.19±0.07% and 1.08±0.02%), complementing the product's micronutrient profile.

Overall, this nutrient ratio makes medlar a valuable source of energy and beneficial macronutrients.

3.1.2 Complex of Bioactive Compounds

As shown in Table 2, medlar fruit pulp is rich in P-active compounds, particularly free catechins (194.44±6.99 mg% of dry mass), condensed catechins (1768.7±63.6 mg% of dry mass), proanthocyanidins (1118.9±40.3 mg% of dry mass), and chlorogenic acids (66.94±0.60 mg% of dry mass).

Table 2 - Quantitative composition of P-active substances in medlar fruit pulp.

P-active compounds	mg% of fresh mass	mg% of dry mass
Chlorogenic acids	18,61±0,19	66,94±0,60
n-Coumaroylquinic acid	8,31±0,09	29,92±1,09
n-Coumaric acid derivatives	3,63±0,04	13,07±0,47
Caffeic acid derivatives	2,27±0,02	8,17±0,29
Flavonol glycosides	6,97±0,07	25,09±0,90

Free catechins	54,01±0,52	194,44±6,99
Condensed catechins	491,3±5,0	1768,7±63,6
Proanthocyanidins	310,8±3,1	1118,9±40,3
p-value	≤0,05	≤0,05

In wild fruit pulp, the concentrations of these biologically active substances are often even higher than in the pulp of cultivated varieties, since wild plants synthesize more protective compounds to survive in their natural environment.

Due to their high content of catechins, proanthocyanidins, and chlorogenic acids, these fruits may have a range of beneficial effects on the body:

• Vascular protection: Free and condensed catechins strengthen capillary walls, reduce their fragility, and improve the elasticity of the entire circulatory system.

• Antioxidant barrier: Proanthocyanidins bind free radicals, slowing cellular aging processes and reducing oxidative stress. They are called "hormones of youth."

• Metabolic support: Chlorogenic acids help regulate blood glucose levels, stimulate metabolism, and support liver health.

3.1.3 Mineral Elements in the Fruits and Their Individual Parts

Table 3 presents the macro- and microelement composition of whole medlar fruits and of their two separated parts: pulp and individual seeds.

Table 3 - Quantitative composition of mineral elements in different parts of medlar fruits.

Mineral elements	mg% of dry mass		
	Whole fruits	Pulp	Seeds
Potassium	1515,0±54,5	2347,0±84,4	603,0±21,6
Sodium	12,4±0,4	18,7±0,6	5,4±0,2
Calcium	121,0±4,4	176,0±6,3	60,6±2,17
Magnesium	63,8±2,3	90,4±3,3	34,7±1,2
Iron	3,70±0,13	5,20±0,19	2,10±0,07
Manganese	0,67±0,02	1,10±0,04	0,28±0,01
Phosphorus	72,4±2,6	106,0±3,8	35,9±1,3
Lead	0,32±0,01	0,35±0,01	0,15±0,01
Cadmium	0,11±0,01	0,13±0,01	0,07±0,01
P-value	≤0,05	≤0,05	≤0,05

The table shows that potassium predominates in the mineral composition of medlar pulp (2347.0±84.4 mg%), followed, at considerably lower levels, by calcium (176.0±6.3 mg%), magnesium (90.4±3.3 mg%), and phosphorus (106.0±3.8 mg% of dry matter). The pulp contains the trace elements iron at 5.20±0.19 mg% and manganese at 1.10±0.04 mg% of dry matter.

This rich mineral profile, especially the high potassium and iron content, makes medlar a particularly useful food for supporting the cardiovascular system, regulating water and electrolyte balance, and preventing anemia.

Analysis of the experimental data indicates that consuming 100 grams of wild medlar fruits per day provides 13.30% of the

daily calcium requirement, 20.80% of the potassium requirement, 15.6% of the magnesium requirement, and 19.20% of the iron requirement. The safety assessment indicates that the levels of the toxic elements lead and cadmium in the different parts of the fruit do not exceed the permissible limits.

3.2 Low-Waste Processing of Medlar Fruits

The proposed processing scheme comprises two stages: (1) separating the seeds from the fruit pulp; and (2) processing the pulp and seeds separately. Confiture is produced from the pulp, and oil from the individual seeds.

3.2.1 Medlar Fruit Pulp Purée

Before processing, the fruits are sorted by quality on a conveyor belt. Fruits affected by pests or diseases, cracked or partially deformed fruits, and organic and mineral impurities are removed. The fruits are then thoroughly washed, inspected, and blanched.

Wild medlar fruits have a rather coarse, tough skin, so blanching is carried out continuously in a screw blancher until the product softens, while ensuring that the fruits retain their shape. The fruits then undergo pulping in a pulping machine fitted with long beaters and rubber seals.

The seeds obtained during this process are sent to the oil-production facility, while the pulped mass is ground further to obtain a homogeneous consistency and completely remove coarse inclusions. Final treatment is carried out using a finisher sieve with 0.4 mm openings.

The homogeneous mass is loaded into the feed hopper of a rotor-cavitation extractor. Ionized water is simultaneously fed into the extractor at a 1:1 ratio, containing a hydrolyzing mixture of citric and phosphoric acids (3:0.05) to obtain activated pectin. Hydroacoustic treatment of the fruit mass is carried out at room temperature for 10-15 minutes with a cavitation index of Pk=0.9. After hydroacoustic treatment, the resulting ground mass can be sterilized and used as the final product, "Natural Purée."

The finely ground mass can be concentrated in a vacuum evaporator to a dry-matter content of 60–65%. The resulting concentrated purée can be used to produce confiture, marmalade, and other gelled products.

Technical documentation was developed for the finished product, and a national standard was prepared and approved by the Azerbaijan Institute of Standardization (AZS 957:2024). A trial batch of "Medlar Confiture with Activated Pectin" was produced using this "cold" technology.

3.2.2 Experiments on Obtaining Oil from Medlar Seeds

Oil from wild medlar seeds contains a broad spectrum of biologically active substances and is therefore used in the food, cosmetics, and pharmaceutical industries.

To crack the hard shells, the seeds are first crushed to obtain a fraction measuring 2–3 mm. This fraction is then dried at 60–65 °C to a residual moisture content of 10%. The dried product is ground further to obtain a fraction measuring 0.25–0.45 mm.

Oil was extracted from medlar stones using supercritical-fluid CO_2 extraction on equipment manufactured by the Swiss company SITEC.

Regulation of carbon dioxide temperature and pressure is considered a key factor in fully extracting oil from the ground seed material.

Experiments increasing the extraction temperature from 40 °C to 80 °C in the hope of increasing CO_2-extract yield were unsuccessful, while energy consumption increased significantly.

This effect is tentatively attributed to a decrease in the solubility of medlar seed oil components as the extraction temperature increases, which in turn negatively affects the total oil yield.

The figure shows how oil yield changed during relatively low-temperature (40 °C) seed extraction as extraction time increased from 1 to 4 hours and pressure from 100 to 280 atm.

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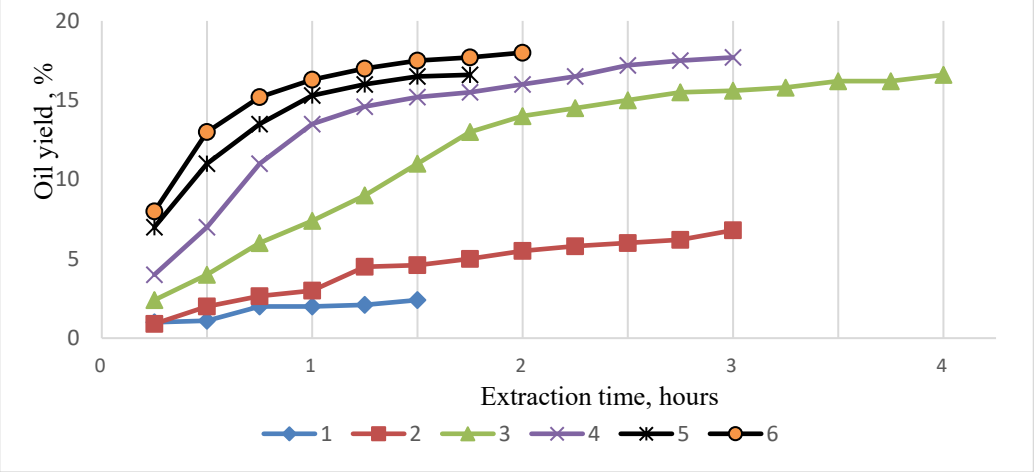


Figure - Oil yield during 4-hour CO_2 extraction of medlar seeds at 40 °C and different pressures (atm): 1 – 100; 2 – 120; 3 – 150; 4 – 200; 5 – 250; 6 – 280.

The figure shows that, to obtain the maximum amount of oil at 40 °C, the following technological parameters should be used: a pressure of 200 atm for 4 hours, or a pressure of 280 atm for 2 hours.

In such cases, speed and throughput generally prevail.

The second option (40 °C, 280 atm, 2 hours) is more economical for most industrial applications.

Halving the process time outweighs the costs of creating a higher pressure for the following reasons:

1. Increased productivity (CAPEX)

• Doubling production volume: Reducing the extraction cycle from 4 to 2 hours makes it possible to carry out twice as many operating cycles per shift or day using the same equipment.

• Lower production costs: This sharply reduces capital costs per unit (equipment depreciation), operating expenses, and labor costs per kilogram of finished extract.

2. Density and Solvent Power of CO_2

• Increased solvent density: At 40 °C, carbon dioxide is in a supercritical state. Increasing the pressure from 200 to 280 atm substantially increases fluid density (approximately from 840 to 890 kg/m³) and its solvent power.

• Faster kinetics: Higher density accelerates mass transfer, allowing the target components to be extracted from the raw material much faster and providing a high yield in a shorter time.

3. Energy Costs (OPEX)

• Although the compressor requires greater instantaneous power to generate 280 atm rather than 200 atm, total energy consumption involves more than gas compression alone.

• Operating the circulation pumps, heating and cooling systems, and ventilation for 4 hours is reduced to 2 hours in the second option. The total electricity consumption per cycle at 280 atm / 2 h is often lower than or equal to that at 200 atm / 4 h.

However, in this case only the first option can be considered, because at 280 atm undesirable heavy fractions, such as paraffins or waxes, begin to be extracted.

Table 4 gives the physicochemical properties of the oil extracted using the first option (40 °C, 200 atm, 4 hours).

Table 4 - Physicochemical properties of the oil.

Indicator	Value (description)
Transparency	Clear
Consistency	Viscous oily liquid
Color	Orange
Odor	Characteristic, strong
Taste	Pleasant, without bitterness
Density, g/dm³	919,0±3,0
Refractive index	1,475±0,06
Acid value, mg KOH/g	2,60±0,08
Saponification value, mg KOH/g	196,0±2,00
Ester value, mg KOH/g	193,0±2,00
Iodine value, g I_2/100 g	125,0±1,00
Peroxide value, ½ O mmol/kg	0,40± 0,03
Volatile substances, %	0,20±0,02
Total antioxidant activity, mg/mL (as quercetin)	4,25±0,10
p-value	≤0,05

These indicators are presented as evidence that the oil is a high-quality, fresh, biologically active, premium-grade vegetable oil:

• Peroxide value (0.40±0.03 mmol/kg): This is an exceptionally low value. Fresh oils are usually considered to have values up to 10, whereas this value is close to zero. This is interpreted as indicating that the oil has undergone virtually no oxidation (rancidity), retained its beneficial properties, and has substantial storage potential.

• Acid value (2.60±0.08 mg KOH/g): This indicates a low content of free fatty acids. The oil was obtained from healthy, high-quality seeds in compliance with the technological requirements.

• Volatile substances (0.20±0.02%): The product is described as being well cleared of residual moisture and impurities, which also protects it against hydrolysis and spoilage.

• Iodine value (125.0±1.00 g I_2/100 g): This classifies the oil as a semi -drying oil. The manuscript describes this as suitable for cosmetics because it can be absorbed quickly without leaving a heavy greasy film or clogging pores (does not cause acne).

• Triglyceride purity: The difference between the saponification value (196.0) and the acid value (2.60) gives a high ester value (193.0±2.00 mg KOH/g). The manuscript interprets this as evidence that the oil consists of pure, intact triglycerides—the form of lipids best absorbed by skin and hair.

• Antioxidant activity (TAA: 4.25±0.10 mg/mL): This is a high value expressed as quercetin equivalents (quercetin being a reference antioxidant). The manuscript states that medlar seed oil may combat free radicals, protect skin against photoaging caused by UV radiation, reduce inflammation, and stimulate cell regeneration.

Table 5- Fatty acid proportions in the oil (%).

Fatty acids	%
Myristic acid (14:0)	0,63
Palmitoleic acid (16:1, omega-7)	0,26
Palmitic acid (16:0)	7,26
Linoleic acid (18:2, omega-6)	70,53
Oleic acid (18:1, omega-9)	17,03
Linolenic acid (18:3, omega-3)	0,54
Stearic acid (18:0)	3,75

As shown in Table 5, the main components of the oil extracted from ground wild medlar stones are polyunsaturated linoleic acid (approximately 70.0%), monounsaturated oleic acid (approximately 17.0%), and the saturated fatty acids palmitic (approximately 7.0%) and stearic (approximately 4.0%) acids.

The oil contained 289.0±8.1 mg% tocopherols, 5.3±0.20 mg% chlorophyll, and 4.3±0.13 mg% carotenoids. Comparison with recognized leading oils (sea buckthorn and rosehip oils) indicates that medlar seed oil is inferior in the regenerative properties associated with carotenoids (provitamin A), but is described as dominant in antioxidant protection due to its exceptionally high vitamin E content.

The solid residue remaining after defatting wild medlar seed powder may serve as a source of polar biologically active antioxidants, particularly polyphenols, and may be used as a valuable food and feed additive.

4. CONCLUSION

Thus, local wild medlar fruits and their processed products showed a rich phytochemical composition characterized by high polyphenol and tocopherol contents. The presence of these biologically active substances and the low content of free fatty acids are stated to account for the high antioxidant stability of the finished products.

The results support the feasibility of incorporating local wild raw materials into small-scale production of confiture and oil for functional uses. This would not only expand the range of eco-products but also make effective use of the region's biological resources.

Further research may focus on optimizing extraction parameters and studying the synergistic effect of medlar with other local wild plants.

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AUTHOR CONTRIBUTIONS

Elsevar Farzaliyev: Conceptualization, Investigation.

Gharib Hafizov: Methodology, Laboratory Analysis.

Bahruz Jabrayilov: Data Curation, Formal Analysis.

Arzu Ashurova: Supervision, Writing—Review & Editing.

Nizami Huseynov: Supervision, Writing—Review & Editing.

CONFLICT OF INTEREST

The authors declare that there is no conflict of interest.

ETHICS STATEMENT

Lorem ipsum dolor sit amet, consectetur adipiscing elit. Ethical approval was not required for this study.

DATA AVAILABILITY STATEMENT

Lorem ipsum dolor sit amet, consectetur adipiscing elit. Data supporting the findings of this study are available from the corresponding author upon reasonable request.

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