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The study of some pharmacognostic aspects of *Ferula persica* Wild. (*Apiaceae*) roots in the flora of Azerbaijan

Aim. To determine the component composition of the monoterpene fraction of *F. persica* roots and identify the localization of biologically active substances in the raw material.

Materials and methods. The identification and quantitative analysis were performed using gas chromatography – mass spectrometry. The raw material was collected during the full seed ripening in the vicinity of the village Dzhangi in the Gobustan region of Azerbaijan.

Results and discussion. Using gas chromatography – mass spectrometry, 15 compounds of monoterpenes were identified. The predominant components were heneicosane – 5.10 %; tetradecane – 3.29 %; disulfide, bis(1-methylpropyl) – 2.86 %; heptadecane – 2.50 %; 9,12,15-octadecatrienoic acid 2,3-bis(acetyloxy)propyl ester – 2.39 %; thiophene, 2,3,4-trimethyl- – 2.05 % in the dry raw material. In addition, it was found that resin was localized in large schizogenous receptacles.

Conclusions. The content of monoterpenes in *F. persica* roots has been determined using gas chromatography – mass spectrometry. The roots contain monoterpenes of heneicosane; tetradecane; disulfide, bis(1-methylpropyl); heptadecane; 9,12,15-octadecatrienoic acid 2,3-bis(acetyloxy)propyl ester. Thiophene and 2,3,4-trimethyl predominate in the raw material.

***Keywords**: monoterpenes; sulfides; localization*

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Вивчення деяких фармакогностичних аспектів коренів *Ferula persica* Wild. (*Apiaceae*) флори Азербайджану

Метою дослідження було визначити компонентний склад монотерпенової фракції коренів *F. persica* та виявити локалізацію біологічно активних речовин у сировині**.**

Матеріали та методи. Ідентифікацію та кількісний аналіз проведено за допомогою газової хроматографії – мас-спектрометрії. Сировину зібрано під час повного дозрівання насіння в навколишній місцевості села Джангі Гобустанського регіону Азербайджану.

Результати та їх обговорення. За допомогою газової хроматографії – мас-спектрометрії ідентифіковано 15 сполук монотерпенів. Переважні компоненти: генкумарин – 5,10 %; тетрадекан – 3,29 %; дисульфід, біс(1-метилпропіл) – 2,86 %; гептадекан – 2,50 %; ефір 2,3-біс(ацетилокси) пропілового ефіру октадекатрієнової кислоти – 2,39 %; тіофен, 2,3,4-триметил – 2,05 % у сухій сировині. Крім того, виявлено локалізацію смоли у великих схізогенних судинах.

Висновки. Визначено вміст монотерпенів у коренях *F. persica* за допомогою газової хроматографії – мас-спектрометрії, зокрема монотерпенів генкумарину, тетрадекану, дисульфіду, біс(1-метилпропілу), гептадекану, ефіру 2,3-біс(ацетилокси)пропілового ефіру октадекатрієнової кислоти. У сухій сировині переважають тіофен, 2,3,4-триметил.

***Ключові слова**: монотерпени; сульфіди; локалізація*

Introduction. *Ferula persica* is widespread in Central Asia, the Mediterranean, and North Africa. In the Caucasus, there are 10 species, and in Azerbaijan, there are 8 of it [1]. Species of the *Ferula* L. genus growing in different regions of the world have been chemically, biologically, and pharmacologically studied.

F. persica contains essential oil, sesquiterpene coumarins of germacrane and eudesmanolide types [2-8]. The essential oil of *Ferula* L. species is predominantly composed of monoterpenes and sulfides [9]. Ostol, sitosterol, L-chimgin, and L-chimganin have been isolated and identified from *F. persica* [10]. The aboveground part of *F. persica* contains luteolin, apigenin, cinaroside, cosmosin, quercetin, and rutin [11, 12].

Sesquiterpene lactones – badkhysin and badkhysinin isolated from the roots of *Ferula oopoda* possess the anti-inflammatory action and are effective antimutagens and antioxidants [13]. Ferulen is the medicine with the estrogenic activity used for the treatment of prostate cancer [14]. The chloroform extract of *F. persica* var. inhibits the production of red pigment produced by *Serratia marcescens* [15]. Umbelliprenin exhibits anti-inflammatory and antipigment properties [16]. Sesquiterpene coumarins and polysulfides from *F. persica* have

cytotoxic, antibacterial, antifungal, anti-leishmanial, chemo-preventive actions against cancer, and inhibitory effects on lipoxygenase [17, 18]. Schematic diagrams of resin localization are available [19].

Thus, the **aim** of study was to determine the component composition of the monoterpene fraction of *F. persica* roots and identify the localization of biologically active substances in the raw material.

Materials and methods. ***Raw material.*** The raw material was collected in the phase of the full seed ripening in the vicinity of the village Dzhangi in the Gobustan region of Azerbaijan (40.50202801719172, 49.264562187572714). Accompanying plants are common sainfoin (*Onobrychis sativa*), cuneifolium (*Seseli cuneifolium*), gwathak (*Zosima absinthifolia*), wormwood (*Artemisia.sp.*), kozel (*Scorzonera sp.*), Persian camel thorn (*Alhagi persarum*), etc.

Chemicals. Ethanol (≥ 99 %, Merck KGaA, EMD Millipore Corporation).

Extraction procedure. The extraction of dried and crushed roots (500 g) was carried out three times with ethanol for 72 hours. During the procedure, 43 g of the total extractive substances were obtained (the yield – 8.6 % by weight of the raw material).

Gas chromatography – mass spectrometry method of analysis. The gas chromatography-mass spectrometric analysis was performed on a GC-MS-Agilent Technologies 7890B instrument coupled with a mass detector (Agilent 5975 C) equipped with an HP-5 MS fused silica column with an internal diameter of 30 m × 0.25 mm × film thickness 0.25 μm, fixed phase. The split ratio was 1:20. The inlet pressure was 88.5 kPa. The flow rate of the carrier gas helium (99.9 %, AGA Lithuania) was set at 1.58 mL/min. The oven temperature was maintained at 40°C for 2 min after injection, then programmed at 9°C/min to 200°C, 10°C/min to 250°C where the column was held for 17 min. The injector temperature was 275 °C. The volume of the injected sample was 0.2 μL (chloroform). The electron ionization mass detector was set at 70 eV in the range of m/z 29-450 a.e.m. The percentage composition of essential oils was calculated based on the gas chromatographic peak areas without correction factors. The qualitative analysis was based on the comparison of retention times, indices, and mass spectra with the corresponding data from literature and NIST/FFSNC mass spectral libraries.

10 g of the total extractive substances obtained were subjected to chromatographic separation on a column with neutral aluminum oxide, grade II. Elution was performed with hexane; the volume of the collected fractions was 100 mL. In fractions 1-20, after evaporation of the solvent, an oily residue was obtained, and it was subjected to the gas chromatography-mass spectrometric analysis.

Microscopic analysis. The identification of diagnostic features in the plant raw material was conducted using well-established methods [19]. For the microscopic analysis, a BİOLAM-C microscope, a MBC-1 binocular, and a L74WIDE Samsung camera were used.

The dried roots were softened in a mixture of ethyl alcohol 95 % and glycerin (1:1), and longitudinal and transverse sections were studied to detect and determine the type of containers.

Results and discussion. The chromatogram shows 35 peaks indicating the presence of numerous components in the fraction 1-20. (Fig. 1) As a result of the chromatographic-mass spectrometric analysis of the fraction residue 1-20, 15 compounds were identified. The main substances include heneicosane – 5.10 %; tetradecane – 3.29 %; disulfide, bis(1-methylpropyl) – 2.86 %; heptadecane 2.50 %; 9,12,15-octadecatrienoic acid 2,3-bis(acetyloxy)propyl ester – 2.39 %; thiophene, 2,3,4-trimethyl- – 2.05 %. The remaining substances were not identified due to their presence in insignificant quantities and lack of practical significance (Table 1).

Table 1

The component composition of monoterpene residues in *F. persica* roots

No.	Identified compound	Chemical formula	Retention time, min	Content in the raw material
1	Disulfide, bis(1-methylpropyl)	$C_8H_{18}S_2$	1.593	2.86
2	Thiophene, 2,3,4-trimethyl-	$C_7H_{10}S$	2.194	2.05
3	1,2-dithiolane	$C_3H_6S_2$	9.880	1.49
4	3,5-diethyl-1,2,4-trithiolane	$C_6H_{12}S_3$	9.953	2.10
5	Tetradecane	$C_{14}H_{30}$	10.545	3.29
6	Isoledene	$C_{15}H_{24}$	13.187	1.99
7	Heptadecane	$C_{17}H_{36}$	17.965	2.50
8	Sulfide, 1-ethyloctylmethyl	$C_9H_{20}S$	19.775	1.08
9	Heptacosane	$C_{27}H_{56}$	21.819	0.80
10	5-alpha-Cholest-8-en-3-one, 14-methyl-	$C_{28}H_{46}O$	25.005	0.29
11	Heneicosane	$C_{21}H_{44}$	25.882	5.10
12	9,12,15-Octadecatrienoic acid 2,3-bis(acetyloxy)propyl ester	$C_{25}H_{40}O_6$	26.954	2.39
13	Tetratriacontane	$C_{34}H_{70}$	27.274	0.86
14	Octacosane	$C_{28}H_{58}$	29.864	1.28
15	Naphthalene, decahydro-2-methyl-	$C_{31}H_{60}$	31.360	0.19

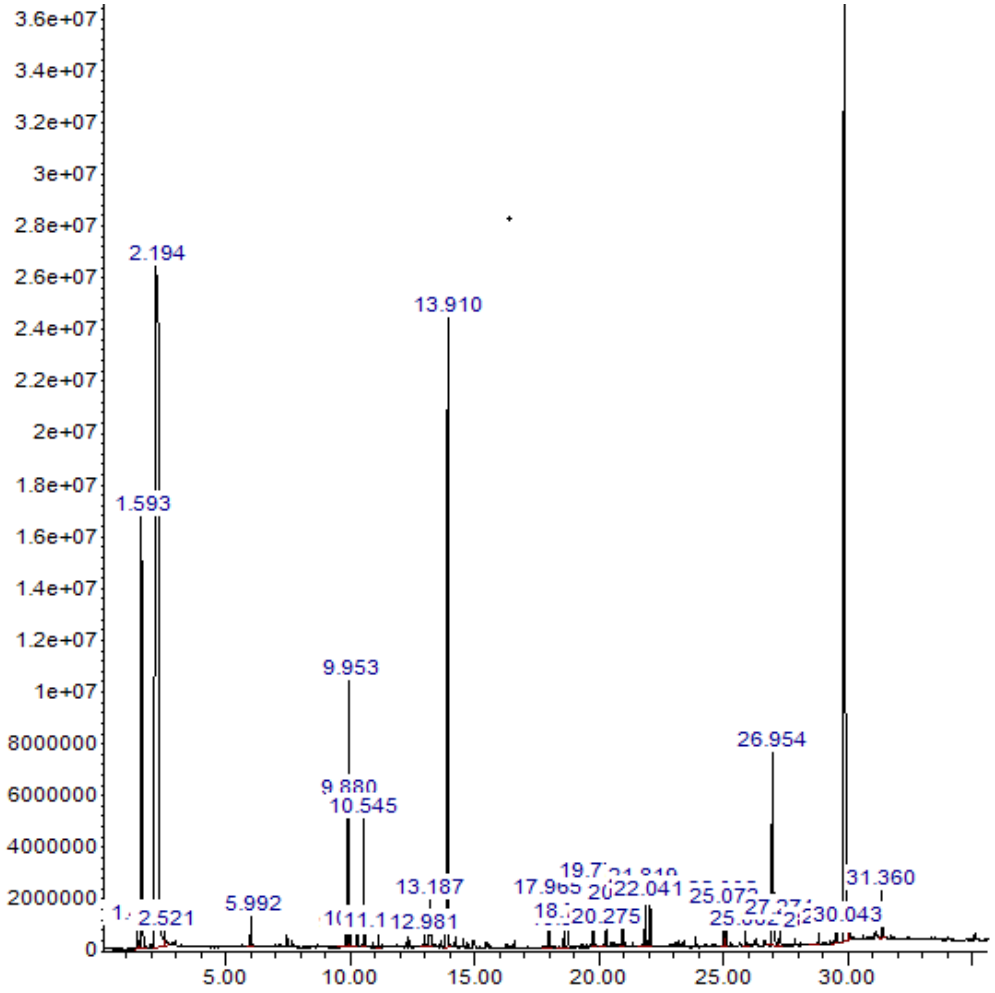
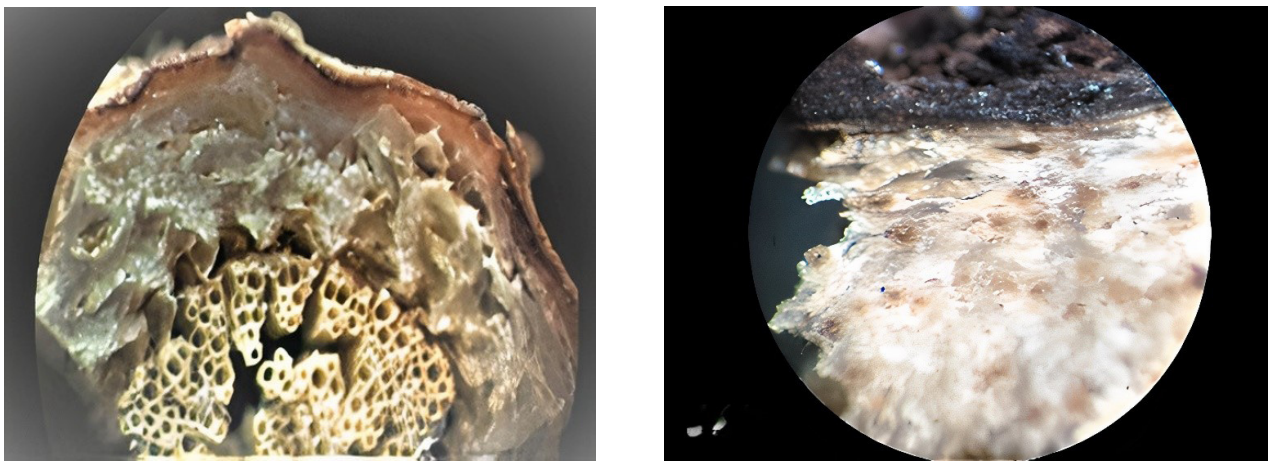


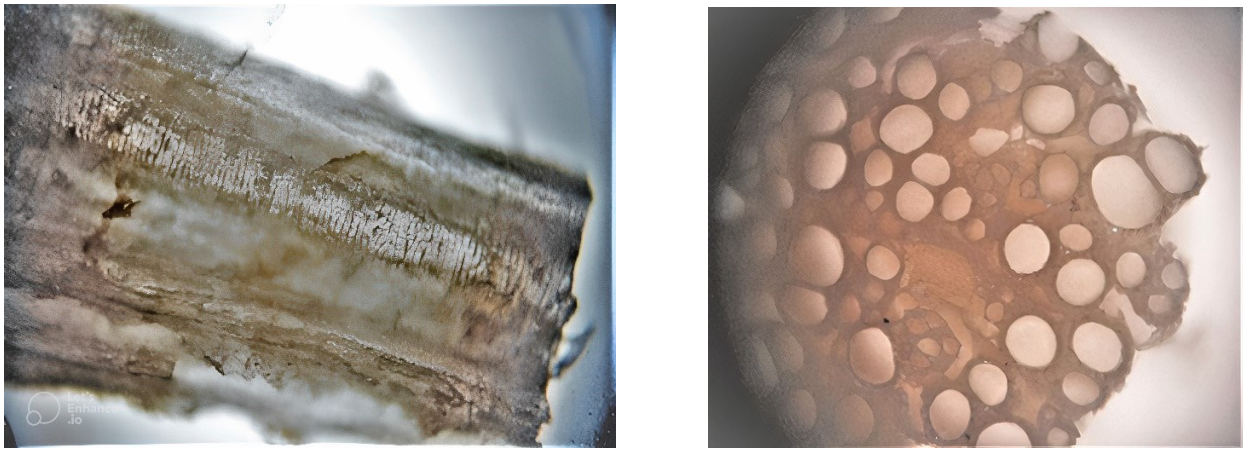
Figure 1. The HPLC fingerprint of monoterpene residues in *F. persica* roots

The roots of *F. persica* contain valuable biologically active substances and are a promising plant material for the development of herbal medicinal products. The main substance in the roots is resin. There is information about the presence of lysigenous, schizogenous, and schizolysigenous receptacles in the parenchyma of the bark of *F. foetida* depicted in a schematic diagram; however, they are not clearly visible in microscopic images. To determine the exact diagnostic features of the plant material, we examined the transverse section of the root and found the presence of schizogenous receptacles in the root parenchyma. Larger receptacles on the transverse section are located at the junction with the xylem. On the longitudinal section of the root, schizogenous receptacles are visible in the parenchyma of the bark (Fig. 2).



A B

Figure 2. Receptacles in the cortical parenchyma of F. persica root: A – transverse, (Zoom x 12); B – longitudinal (Zoom. x 12)



A B

Figure 3. Conductive vessels in the xylem of F. persica root:
A – longitudinal section of the root (Zoom x 12); B – cross section of the root (Zoom x 100)

Conductive vessels are visible on the longitudinal and transverse sections of the root in the xylem area (Fig. 3).

Conclusions. The content of monoterpenes in *F. persica* roots has been determined using gas chromatography – mass spectrometry. The roots contain monoterpenes of heneicosane – 5.10 %; tetradecane – 3.29 %; disulfide, bis(1-methylpropyl) – 2.86 %; heptadecane – 2.50 %; 9,12,15-octadecatrienoic acid 2,3-bis(acetyloxy) propyl ester – 2.39 %. Thiophene and 2,3,4-trimethyl – 2.05 % predominate in the raw material.

It has been identified that the resin is localized in large schizogenous-type reservoirs located in the parenchyma of the root bark.

Conflict of interests: authors have no conflict of interests to declare.

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