

Chemical Composition of Flower Volatiles and Seeds Fatty Acids of *Rosa iliensis* Chrshan, an Endemic Species from Kazakhstan

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Abstract: In the present work, the flower volatiles and seed fatty acids compositions of three populations (P-I, P-II, P-III) of *Rosa iliensis* were investigated for the first time. *R. iliensis* is a rare, endangered, narrow-endemic species growing in floodplains of the Ili and Sharyn rivers of Almaty region. The flower volatiles have been investigated with the tandem of MSD-SPME and GC-MS/FID techniques. The seed lipids were extracted from the ripe fruits with microextraction technique. The flower volatiles of *R. iliensis* three populations were characterized by the abundance of oxygenated monoterpenes with benzaldehyde (13.3-38.7 %) and citronellol (2.6-8.8 %) as the major constituents. There were detected significant differences in floral scents between the populations. The flowers of P-I (from Sharyn River) contain sesquiterpene γ -elemene (8.8%), the flowers of P-II (upper reaches of the Ili River) were rich in α -gurjunene (12.8%), while flowers of P-III (lower reaches of the Ili River) contained any sesquiterpenes. Seven fatty acids were determined in the seeds and unsaturated acids were found to be dominant for studied populations. Linoleic (43.0-51.0%), α -linolenic (26.5-28.1%), and oleic (12.0-16.1%) acids were detected as the major constituents. The present study shows that *R. iliensis* species is rich source of valuable volatiles and fatty acids.

Keywords: *Rosa iliensis*; population; volatiles; fatty acids; MSD-SPME; microextraction; GC-MS/FID.

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1. Introduction

The genus *Rosa* L. (Rosaceae family) grows naturally throughout the temperate and subtropical regions of the northern hemisphere and consists of approximately 120 species that are

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widely distributed in Europe, Asia, the Middle East and North America. The genus is divided taxonomically into four subgenera: *Hulthemia* (Dumort) Focke, *Platyrrhodon* (Hurst) Rehder, *Hesperhodos* Cockerell and *Rosa*. The subgenus *Rosa* comprises nine sections: *Cinnamomeae* (DC. Ser.) (incl. *Carolinae* Crép.), *Caninae* (DC.Ser.), *Synstylae* DC., *Pimpinellifoliae* (DC. Ser.), *Banksianae* Lindl., *Bracteatae* Thory, *Indicae* Thory, *Laevigatae* Thory, and *Rosa* [1]. In the CIS (Commonwealth of Independent States) there are 63 species of rose hips and 10 types of cultivated roses, including 21 species of rose hip and 3 types of cultivated roses in Kazakhstan [2].

Rosa L. species are an object of great scientific interest as a source of biologically active substances and are widely used as medicinal, vitamin, and food raw materials. For food purposes, rose hip is used in tea [3], syrups [4], jam, marmalade [5], wine [6], soups, and noodles [7, 8].

The main commercial value of *Rosa* species is known to be as essential oil (rose oil) [9, 10] and fruits (rose hip) that are good source of valuable bioactive metabolites such as vitamin C [11], carotenoids, polyphenols (anthocyanins, etc.) [12], organic acids (citric, malic etc.), fatty acids, and carbohydrates (glucose etc.). In the scent of roses, more than 400 volatiles have been reported [13].

The fruits of the rose hip have been used in folk medicine for a long time. Rose hip positive effects have been demonstrated in reducing the risk of heart disease [14], various forms of cancer [15-17], diarrhea [18], inflammation, and arthritis [19], pre obese and diabetes [20]. Rose hips have prophylactic and therapeutic actions against the common cold, infectious diseases, gastrointestinal disorders, urinary tract diseases, and inflammatory diseases [21, 22]. Rose hip as the richest natural source of vitamin C [23] was used for the treatment of osteoarthritis in several clinical studies [24].

The aim of this study was to characterize the floral scent and seed's fatty acids profile of *R. iliensis* three populations for subsequent characterization of the distribution and the raw material reserves that formed potential commercial element for national pharmaceutical industry. To the best of our knowledge, there is no previous report about flower volatiles and seed fatty acids of *R. iliensis*.

2. Materials and Methods

2.1. Chemicals

Lipid Extraction Kit, boron trifluoride reagent (BF₃), and *n*-hexane (Sigma-Aldrich, Germany) were purchased from Sigma-Aldrich (St. Louis, USA). A C₈-C₄₀ *n*-alkane standard solution was purchased from Fluka (Buchs, Switzerland). A manual SPME holder (57330-U, SUPELCO, Bellefonte, PA) and the PDMS-DVB 65 mm fiber (blue type) were used for SPME procedure of volatiles.

2.2. Plant Material

The samples of *Rosa iliensis* (FigureS 1) were collected in 2018 (September - October) during the flowering and fruiting stages along floodplain of the Ili and Sharyn rivers in Almaty Region of the Republic of Kazakhstan (Figure S2). The coordinates of the populations are given in Table 1. Botanical identification was performed by Dr. Ametov A.A. (Al-Farabi Kazakh National University). The plant material was presented as the flowers and the fruits with seeds. Voucher specimens are deposited at the Herbarium of the Institute of Botany and Phytointroduction of the Forestry and Wildlife Committee of the Ministry of Ecology, Geology and Natural Resources of the Republic of Kazakhstan (index AA, voucher specimen number U. Fisyun 3389).

Table 1. Collection data of *Rosa iliensis* populations

Population	Plant material	Location	GPS coordinate	Altitude
P-I	Flowers, seeds	Sharyn River	79°15'44.1" north latitude, 43°31'26.4" east longitude	629 m
P-II	Flowers, seeds	upper reaches of the Ili River	79°34'46.4" north latitude, 43°58'19.8" east longitude	494 m
P-III	Flowers, seeds	lower reaches of the Ili River	76°57'37" north latitude, 44°09'50.6" east longitude	417 m

2.3 Extraction of Volatiles with MSD-SPME Technique

Microsteam distillation - solid phase microextraction (MSD-SPME) of the volatiles was carried out using an assembly reported previously [25]. MSD-SPME technique involved concurrent solid-phase microextraction combined with continuous hydrodistillation of the volatiles. This method significantly reduces the time required for the isolation of volatiles. It should be noted that this technique allows the isolation of volatiles from a very small amount of plant material [26, 27]. Tandem of MSD-SPME with GC-MS/FID techniques is simple, sensitive, rapid, solvent-less, and non-toxic green technique for analysis of the volatile compounds at microscale level. In experiment, the ground flowers (0.3 g) were placed in 25 mL round bottom flask along with 3 mL of water. The flask was fitted with a Claisen distillation head with plug and a condenser set up for refluxing rather than distillation. The threaded plug was used for SPME fiber assembly. A SPME holder equipped with PDMS-DVB “blue type” fiber was used for the extraction of volatiles. Previously, the fiber was conditioned at 250 °C for 10 min before the experiment. After the SPME needle pierced the plug, the fiber was expressed through the needle and exposed to the headspace above the plant sample. The extraction time for the flower volatiles was 3 min. After trapping of the volatiles, the loaded SPME fiber was withdrawn into the needle, and then the needle was removed from the plug and subsequently used for GC-MS/FID analyses. Thermal desorption of analytes from the fiber coating was performed by injection of the fiber in the injection port (at 250°C) for 5 min.

2.4. Lipid Extraction and Fatty Acid Derivatization

The protocol for fatty acid analysis comprises the following steps: sample preparation, total lipid extraction, methylation of fatty acids, and analysis of the fatty acid methyl esters using GC-MS/FID techniques. The Lipid Extraction Kit was used for extraction of the total lipids from the seeds [28]. The lipids were typically extracted using a dual solvent partition system containing a lipophilic solvent and an aqueous solvent. The lipids were retained in the lower chloroform layer; whereas, aqueous-soluble compounds were retained in the upper methanol-water layer. According to the kit protocol, 0.15 g mill-ground plant material was homogenized with 3 mL extraction solvent consisting of chloroform/methanol (2:1, v/v). After homogenizing and vortexing, 0.5 mL of an aqueous buffer of the kit (composition is not disclosed by the company) was added and the sample was vortexed again. Subsequently, the extraction solution was poured into a syringe system containing a filter (trapping the water). The eluted solvent contained the chloroform phase with total lipids that comprised all extracted lipids from the seeds. The aliquot 200 µL of the total lipids was dried under a stream of nitrogen for subsequent transesterification. After drying, 1 mL of Boron trifluoride-methanol solution and 0.3 mL of *n*-hexane were added. The mixture was heated at 95°C for 1 hour under reflux. Then, 1 mL of *n*-hexane and 1 mL of distilled water were added to the reaction vessel, vortexed and centrifuged at 500 × g for 5 min. The top *n*-hexane layer was transferred into vial and then injected into GC-MS/FID system without solvent evaporation prior to injection.

2.5. Gas Chromatography-Mass Spectrometry (GC/MS) Analysis

GC-MS analysis was performed with an Agilent 5975 GC-MSD system (Agilent Technologies, Santa Clara, CA, USA), as reported previously [25]. An Agilent Innowax FSC column (60 m × 0.25 mm, 0.25 µm film thickness) was used with He as the carrier gas (0.8 mL/min). The GC oven temperature was kept at 60°C for 10 min, increased to 220°C at a rate of 4°C/min, kept constant at 220°C for 10 min, and then increased to 240°C at a rate of 1°C/min. The split ratio was adjusted to 40:1, and the injector temperature was 250°C. MS spectra were monitored at 70 eV with a mass range of 35 to 450 *m/z*.

2.6. Gas Chromatography (GC-FID) Analysis

GC analysis was carried out using an Agilent 6890N GC system. To obtain the same elution order as with GC-MS, the line was split for FID and MS detectors, and a single injection was performed

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using the same column and appropriate operational conditions. Flame ionization detector (FID) temperature was 300°C.

2.7. Identification of the Volatile Constituents

The essential oil constituents and fatty acid methyl esters were identified by co-injection with standards (whenever possible), which were purchased from commercial sources or isolated from natural sources. In addition, compound identities were confirmed by comparison of their mass spectra with those in the Wiley -NIST GC/MS Library (Wiley, NY, USA) [29], MassFinder software 4.0 (Dr. Hochmuth Scientific Consulting, Hamburg, Germany) [30], and Adams Library [31]. Confirmation was also achieved using the in-house “Başer Library of Essential Oil Constituents” database, obtained from chromatographic runs of pure compounds performed with the same equipment and conditions. A C₈–C₄₀ *n*-alkane standard solution (Fluka, Buchs, Switzerland) was used to spike the samples for the determination of relative retention indices (RRI). Relative percentage amounts of the separated compounds were calculated from FID chromatograms.

3. Results and Discussion

Volatile substances are important attributes used for the assessment of the quality of *Rosa* species fragrance. In the present work, the flower volatiles of *Rosa iliensis* three populations, endemic species of the Flora of Kazakhstan, have been investigated with tandem of MSD-SPME and GC-MS/FID techniques. In addition, the lipids were obtained from the seeds of *R. iliensis* ripe fruits with microextraction technique and methyl esters of the fatty acids were prepared using Boron trifluoride reagent for subsequent gas chromatographic analysis.

The chemical composition of the flower volatiles was investigated using GC-FID and GC/MS simultaneously, and Tables 2 and 3 summarize the identified constituents, their percentage composition, and their relative retention indices (RRI) (compounds are listed in order of their elution on Innowax column). Qualitative and quantitative distinctions in the flower's volatile profiles of the three *R. iliensis* populations were demonstrated. A total of 51 compounds were identified in the flowers of *R. iliensis* three populations. The flowers of P-I and P-II contained 25 and 23 constituents, representing 97.3% and 97.7% of the total volatiles, respectively. The volatile profile of the flowers of P-III was found to be the most complex with 37 constituents, representing 97.7% of the total volatile's composition.

Table 2. Chemical composition of the flower volatiles of *Rosa iliensis* collected from three populations

No	RRI ^a	RRI ^b	Compound	% ^c		
				P-I	P-II	P-III
1	1032	1008-1039 [32]	α-Pinene	-	2.5	-
2	1093	1056–1106 [32]	Hexanal	8.0	3.8	11.4
3	1194	1163–1208 [32]	Heptanal	-	-	4.6
4	1225	1118–1160 [32]	(<i>Z</i>)-3-Hexenal	17.3	9.8	8.0
5	1348	1317–1357 [32]	6-Methyl-5-hepten-2-one	-	1.3	-
6	1244	1232 [32]	Amyl furan (=2- <i>Pentyl furan</i>)	3.8	-	1.4
7	1300	1300 [33]	Tridecane	-	-	0.7
8	1335	1289–1339 [32]	(<i>E</i>)-2-Heptenal	-	-	1.3
9	1360	1316–1377 [32]	1-Hexanol	3.1	1.7	0.8
10	1362	1331–1369 [32]	<i>cis</i> -Rose oxide	-	-	1.2
11	1376	1341–1386 [32]	<i>trans</i> -Rose oxide	-	-	0.4
12	1400	1400 [34]	Tetradecane	-	-	1.3
13	1402	1351–1405 [32]	(<i>Z</i>)-3-Hexen-1-ol	3.1	2.3	-

No	RRI ^a	RRI ^b	Compound		% ^c	
14	1400	1370–1414 [32]	Nonanal	-	0.8	1.4
15	1416	1415 [35]	3-Octen-2-one	-	-	t
16	1441	1407–1463 [32]	(<i>E</i>)-2-Octenal	-	-	1.4
17	1452	1411–1465 [32]	1-Octen-3-ol	-	-	1.2
18	1479	1478 [36]	(<i>E,Z</i>)-2,4-Heptadienal	-	-	1.3
19	1507	1455–1514 [32]	(<i>E,E</i>)-2,4-Heptadienal	-	-	0.6
20	1520	1521 [37]	3,5-Octadien-2-one	-	-	0.5
21	1544	1511–1545 [32]	α -Gurjunene	-	12.8	-
22	1497	1462–1522 [32]	α -Copaene	0.9	-	-
23	1466	1438–1480 [32]	α -Cubebene	0.7	-	-
24	1541	1481–1555 [32]	Benzaldehyde	32.9	38.7	13.3
25	1612	1570–1685 [32]	β -Caryophyllene	1.4	5.4	-
26	1600	1600 [38]	Hexadecane	-	-	0.3
27	1664	1624–1674 [32]	1-Nonanol	-	0.4	-
28	1661	1624–1668 [32]	Alloaromadendrene	-	0.4	-
29	1617	1609 [39]	6,9-Guaiadiene	0.8	-	-
30	1650	1612–1654 [32]	γ -Elemene	8.8	-	-
31	1663	1640 [32]	Phenylacetaldehyde (=Benzene acetaldehyde)	1.3	0.6	2.4
32	1665	1657 [40]	4-Methyl-benzaldehyde*	-	-	3.5
33	1694	1641–1706 [32]	Neral	-	0.3	1.3
34	1704	1655–1714 [32]	γ -Muurolene	0.7	-	-
35	1740	1686–1753 [32]	α -Muurolene	0.8	-	-
36	1742	1680–1750 [32]	Geranial	0.7	0.7	2.2
37	1772	1734–1789 [32]	Citronellol	2.6	3.7	8.8
38	1779	1729–1779 [32]	(<i>E,Z</i>)-2,4-Decadienal	-	-	0.6
39	1808	1752–1832 [32]	Nerol	-	-	0.6
40	1773	1722–1774 [32]	δ -Cadinene	1.5	-	-
41	1796	1750–1800 [32]	Selina-3,7(11)-diene	1.1	-	-
42	1802	1747–1805 [32]	Cumin aldehyde	0.7	1.0	0.6
43	1811	1811 [41]	<i>p</i> -Mentha-1,3-dien-7-al	-	0.2	0.4
44	1812	1808 [42]	Liguloxide	0.6	-	-
45	1827	1770–1834 [32]	(<i>E,E</i>)-2,4-Decadienal	-	-	2.8
46	1857	1795–1865 [32]	Geraniol	1.0	0.9	4.4
47	1868	1820–1873 [32]	(<i>E</i>)-Geranyl acetone	0.3	0.2	0.6
48	1896	1821–1905 [32]	Benzyl alcohol (=Benzenemethanol)	4.7	9.1	2.3
49	1925	1859–1944 [32]	Phenyl ethyl alcohol (=Benzeneethanol)	0.5	1.1	1.5
50	1940	1933 [43]	2-Phenyl-undecane	-	-	0.4
51	2300	2300 [33]	Tricosane	-	-	1.6
Total				97.3	97.7	97.7

RRI^a, relative retention index experimentally calculated based on retention of *n*-alkanes, **RRI^b**, relative retention index reported in literature for constituents analyzed on polar column; ^c The data are presented as relative % by weight for each component detected in *R. iliensis* flowers.; %, calculated from flame ionization detector data. Trace amounts (**t**) were present at <0.1%. **P-I, P-II, P-III**: populations I, II and III.

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Distribution of the main compound groups presented in the flower volatiles of *R. iliensis* from three populations is presented on Table 3. In general, the flower volatiles of *R. iliensis* were characterized with abundance of oxygenated monoterpenes constituting 38.2%, 45.7%, and 33.8% in populations P-I, P-II, and P-III, respectively. The main components of the volatiles were benzaldehyde (32.9%, 38.7% and 13.3%) and citronellol (2.6%, 3.7%, and 8.8%). The populations P-I (near to Sharyn River) and P-II (upper reaches of Ili River) were distinguished by noteworthy content of sesquiterpene hydrocarbons (17.3% and 18.6%, respectively). γ -Elemene (8.8%) was detected in P-I, while α -gurjunene (12.8%) and β -caryophyllene (5.4%) were found in P-II. Overall, there were significant differences in the flower volatiles compositions between *R. iliensis* populations from lower and upper reaches of the Ili River. However, the flowers of *R. iliensis* of P-III (lower reaches of the Ili River) contained any sesquiterpenes.

Aliphatic aldehydes were found to be the next noteworthy compound group constituting the flower volatiles of *R. iliensis*. The highest content (33.4%) of the aliphatic aldehydes was detected in P-III, then in P-II (25.3%) and P-I (14.4%). Hexanal (8.0%, 3.8%, and 11.4%) and (Z)-3-hexenal (17.3%, 9.8% and 8.0%) were found to be the major aldehydes in P-I, P-II, and P-III, respectively. Ten other compounds were present in P-I and P-II at concentrations $\leq 1.0\%$. In P-III twelve compounds were present at concentrations $\leq 1.0\%$.

Table 3. Distribution of the main compound groups presented in the flower volatiles of *Rosa iliensis* from three populations

Compound group	%		
	P-I	P-II	P-III
Monoterpene hydrocarbons	3.8	2.5	1.4
Oxygenated monoterpenes	38.2	45.7	33.8
Sesquiterpene hydrocarbons	17.3	18.6	0
Oxygenated sesquiterpenes	0.6	0	0
Aliphatic alcohols	6.2	4.4	2
Aliphatic aldehydes	25.3	14.4	33.4

%, calculated from flame ionization detector data. **P-I, P-II, P-III:** populations I, II, and III.

Comparison of the chromatographic profiles of *R. iliensis* floral volatiles presented on Figure S3, showed the obvious differences and similarities between three populations.

The chemical compositions of the floral scent from two distinct genotypes of Iranian *R. damascena* flowers have earlier been reported by Karami et al. (2013) [44]. The main floral headspace components were phenyl ethyl alcohol (2.2-40.6%), β -citronellol (0.1-45.1%), α -pinene (67.2-1.2%), benzyl alcohol (2.6-64%) and geranyl acetate (0.7-22.7%). The percentage of the constituents varied according to development of the flowers. Phenyl ethyl alcohol was one of the dominant scent compounds emitted from European roses such as *R. damascena* and *R. hybrida* 'Hoh-Jun' which were used for the commercial production of rose oils [13]. In our work, the flowers of *R. iliensis* from Kazakhstan contained this constituent in scarce amount (between 0.5 and 1.5%).

One of important healthy functions of *Rosa* species is their essential fatty acids that humans cannot synthesize and have to get through diet [45]. Among these acids the polyunsaturated ones have important role in preventing of neurodegenerative and different metabolic disorders [46, 47]. Earlier, linoleic and α -linolenic acids were reported as major fatty acids in lipid profile of *R. canina* seeds [48].

In the present work, the fatty acids of *R. iliensis* were microextracted from the seeds gathered from ripe fruits. Subsequent transesterification and GC-MS/FID analysis resulted with seven fatty acids, three of them were designated as major ones (Table 4). There was no noticed a great variation of fatty acids between the three populations of *R. iliensis*. The major unsaturated fatty acid in the seed lipids was linoleic acid (18:2 ω 6, 45.1%, 51.0%, and 44.0%) in populations P-I, P-II and P-III, respectively. It was followed by α -linolenic (18:3 ω 3, 31.1%, 29.0%, and 28.5%) and oleic (18:1 ω 9,

14.0%, 13.0%, and 17.1%) acids. Among saturated fatty acids, nonanedioic (9:0 dioic), palmitic (16:0), stearic (18:0), and nonadecanoic (19:0) acids have been detected.

It is well known that α -linolenic and linoleic acids are two essential fatty acids that humans require [49, 50]. Results of the seed oil investigation allowed us to think that the rose hip seed and seed oil may be proposed as ingredients in functional food formulations and dietary supplements.

Table 4. Fatty acid composition of the seed lipids of *Rosa iliensis* collected from three populations

No	RRI ^a	RRI ^b	Compound	% ^c		
				P-I	P-II	P-III
1	2105	2101 [51]	9:0 dioic (Dimethyl azelate)	1.1	t	t
2	2223	2223 [52]	16:0 (Methyl palmitate)	6.4	4.8	6.6
3	2436	2445 [53]	18:0 (Methyl stearate)	t	2.2	2.7
4	2468	2472 [53]	18:1 ω 9 (Methyl oleate)	14.0	13.0	17.1
5	2509	2502 [54]	18:2 ω 6 (Methyl linoleate)	45.1	51.0	44.0
6	2512	2513 [53]	19:0 (Methyl nonadecanoate)	t	t	t
7	2572	2590 [53]	18:3 ω 3 (Methyl α -linolenate)	31.1	29.0	28.5
Total:				97.7	100.0	99.9

RRI^a, relative retention index experimentally calculated based on retention of *n*-alkanes, **RRI^b**, relative retention index reported in literature for constituents analyzed on polar column; ^c The data are presented as relative % by weight for each component detected in *R. iliensis* flowers.; %, calculated from flame ionization detector data. Trace amounts (t) were present at <0.1%. **P-I, P-II, P-III:** populations I, II and III.

The residue products from rose hips have usually been used as animal fodder, but nowadays these residue products are gaining in importance as they can be used in cosmetics, pharmacology and in food applications due to containing the oil with a high degree of unsaturated fatty acids. The quantity of oil contained in the seeds depends on the species and ranges from 5 to 18% [55]. The fatty acid profile of seed lipids and the ratio of saturated / unsaturated fatty acids have contribution into medicinal importance of *Rosa* fruits due to their beneficial in cardiovascular issues (thrombosis, arteriosclerosis), lowering blood pressure, antimicrobial effect, etc. The efficacy of rose hip seed oil together with an oral fat-soluble vitamins on different inflammatory dermatitis such as eczema, neurodermatitis, and cheilitis, with promising findings of the topical use of rose hip seed oil on these inflammatory dermatoses has been reported by Lin *et al.* [56]. The rose hip oil has shown to reduce skin pigmentation, discoloration, acne lesions, scars and stretch marks, as well as retaining the moisture of the skin and delaying the appearance of wrinkles [57].

In literature there are a number of reports about fatty acids of *Rosa* species. Review on *Rosa* species has recently been reported by Ahmad *et al.* [58]. Szentmihályi *et al.* (2002) applied different techniques for extraction of oil from *R. canina* seeds [48]. Ilyasoğlu (2014) has recently characterized the seeds and seed oil of *R. canina* [59]. The rose hip seed oil was rich in polyunsaturated fatty acids, linoleic acid (54.05%), linolenic acid (19.37%), and phytosterols, mainly β -sitosterol (82.1%). α -Linolenic acid was found to be dominant for the fruits of *R. canina* L., *R. dumalis* subsp. *boissieri* O. Nilsson, *R. dumalis* subsp. *antalyensis* (Manden.) Ö. Nilsson, *R. villosa* L., *R. pulverulenta* M. Bieb and *R. pisiformis* (H. Christ) Sosn. [60, 61]. Oleic, linoleic, and α -linolenic have been reported as major fatty acids in the fruits of *R. rubiginosa* L., *R. subcanina* (H. Christ) Vuk., *R. dumalis* (*besseriana*), *R. inodora* Fr., *R. villosa*, *R. rugosa* Thunb., while palmitic, linoleic, and α -linolenic were determined in noteworthy amounts in *R. dumalis* Bechst., *R. pisiformis*, *R. villosa* L. and *R. pulverulenta* [62-64].

4. Conclusion

We report here about qualitative and quantitative distinctions in the flower's volatile profiles and seed fatty acids of the three *R. iliensis* populations. It was found that flower volatiles of *R. iliensis* contain a high amount of the oxygenated monoterpenes with benzaldehyde and citronellol as major

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constituents. Moreover, we determined the distinguish between populations distributed on upper and lower reaches of Ili River. The findings of the study indicated that *R. iliensis* volatiles rich in benzaldehyde and citronellol may be interesting for the further studies as components of different cosmetic products. It has earlier been well illustrated in the case of otto of roses that aromatic constituents like the alcohols (citronellol, phenyl ethyl alcohol etc.) as well as the aldehydes (benzaldehyde), although present in very small quantities, undoubtedly play an important role in the aromatic profile and commercial value of *Rosa* species [44, 65, 66]. In this respect, the commercial value of these volatiles should be evaluated as a production of concrete (extraction of the petals with hydrocarbon solvents) instead of pure essential oil.

On the other hand, it worth to notice that unsaturated part of the fixed oil of *R. iliensis* fruit seeds has quite high amount (90%) together ω 3, ω 6 and ω 9 fatty acids which play important role and required for human's health. So, the results of the seed oil investigation allowed us to think that the rose hip seeds and seed oil may be proposed as ingredients in functional food formulations and dietary supplements. The nutritional composition and the presence of bioactive compounds make the rosehip seed a valuable source of phytonutrients.

Thus, our data provide a previous basis to explain at least part of the beneficial properties of the flower volatiles and seed lipids from *R. iliensis*.

Conflicts of Interest: The authors declare no competing financial interest.

Author Contributions:

G.Ö. and A.Ch. conceived and designed the project.

A.Ch. and A.A. collected and identified plant material.

G.Ö., A.Ch., and T.Ö. performed the experiments.

G.Ö. and A.Ch. analyzed and interpreted the data, drafted and revised the manuscript.

All authors have read and agreed to the published version of the manuscript.

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Supporting Information

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References:

- [1] V. Wissemann and C.M. Ritz (2005). The genus *Rosa* (rosoideae, rosaceae) revisited; molecular analysis of nrITS-1 and atp B-rbc L intergenic spacer (IGS) versus conventional taxonomy, *Bot. J. Linnean Soc.* **147**(3), 275-290.
- [2] N.V. Pavlov, ed. *Flora of Kazakhstan. Vol. 4.* 1961, Academy of Sciences of Kazakh SSR: Alma-Ata. 288.
- [3] S.-H. Choi (2009). Essential oil components in herb teas (rose and rosehip), *Kor. J. Life Sci.* **19**, 1333-1336.
- [4] A. Leahu, C. Damian, M. Oroian, S. Ropciuc and R. Rotaru (2014). Influence of processing on vitamin C content of rosehip fruits, *Scient. Papers Animal Sci. Biotechnol.* **47**, 116-120.
- [5] S. Ercisli and M. Güleriyüz (2004). Rose hip utilization in Turkey, in *1 Int. Rose Hip Conference 690*.
- [6] R. Stoličná (2016). Possibilities of using wild plants in the traditional culinary culture of Slovakia, *Sloven. Národopis.* **64**(2), 241-250.
- [7] O. Sagdic, O.S. Toker, B. Polat, M. Arici and M.T. Yilmaz (2015). Bioactive and rheological properties of rose hip marmalade, *J. Food Sci. Technol.* **52**(10), 6465-6474.
- [8] I. Koca, B. Tekguler, V.A. Yilmaz, I. Hasbay and A.F. Koca (2018). The use of grape, pomegranate and rosehip seed flours in Turkish noodle (erişte) production, *J. Food Proces. Preserv.* **42**(1), e13343.
- [9] Z. Xiao, J. Li, Y. Niu, Q. Liu and J. Liu (2017). Verification of key odorants in rose oil by gas chromatography– olfactometry/aroma extract dilution analysis, odour activity value and aroma recombination, *Nat. Prod. Res.* **31**(19), 2294-2302.
- [10] H. Baydar (2006). Oil-bearing rose (*Rosa damascena* Mill.) cultivation and rose oil industry in Turkey, *Euro Cosmetic.* **14**(6), 13-17.
- [11] A. Sevket, S. Ercisli, T. Jurikova, O. Cakir and S. Gozlekci (2016). Bioactive content of rose hips of different wildy grown *Rosa dumalis* genotypes, *Not. Bot. Horti. Agrobi.* **44**(2), 472-476.
- [12] N. Koczka, É. Stefanovits-Bányai and A. Ombódi (2018). Total polyphenol content and antioxidant capacity of rosehips of some rosa species, *Medicines* **5**(3), 84 (10 pages).
- [13] S. Baldermann, Z. Yang, M. Sakai, P. Fleischmann and N. Watanabe (2009). Volatile constituents in the scent of roses, *Floriculture Ornament. Biotechnol.* **3**(1), 89-97.
- [14] P. Hagan (2012). Daily dose of rosehip extract could help cut heart disease by lowering blood pressure and cholesterol levels', *Daily Mail Online* [7] *Naruszewicz, Marek, et al.* "Effect of *Lactobacillus plantarum* 299v on cardiovascular disease risk factors in smokers." *Am. J. Clinic. Nutr.* **76**(2002), 1249-1255.
- [15] V.T. Tumbas, J.M. Čanadanović-Brunet, D.D. Četojević-Simin, G.S. Četković, S.M. Đilas and L. Gille (2012). Effect of rosehip (*Rosa canina* L.) phytochemicals on stable free radicals and human cancer cells, *J. Sci. Food Agric.* **92**(6), 1273-1281.
- [16] M. Sameeullah, M. Gündoğdu, İ. Canan, T. Karadeniz, M. Aasim and K.M. Khawar (2018). Fruits of Rosaceae family as a source of anticancer compounds and molecular innovations, In: *Anticancer plants: mechanisms and molecular interactions*, Springer, pp. 319-336.
- [17] P. Cagle, O. Idassi, J. Carpenter, R. Minor, I. Goktepe and P. Martin (2012). Effect of Rosehip (*Rosa canina*) extracts on human brain tumor cell proliferation and apoptosis, *J. Cancer Therapy.* **3**, 534-545.
- [18] G. Ivady (1952). Treatment of diarrhoea in infants and young children with rosehip flour and extract, *Ann. Paediat.* **178**, 107-115.
- [19] M. Cohen (2012). Rosehip: an evidence based herbal medicine for inflammation and arthritis, *Austr. Family Physic.* **41**(7), 495-498.
- [20] A. Nagatomo, N. Nishida, I. Fukuhara, A. Noro, Y. Kozai, H. Sato and Y. Matsuura (2015). Daily intake of rosehip extract decreases abdominal visceral fat in preobese subjects: a randomized, double-blind, placebo-controlled clinical trial, *Diabetes Metab. Syndr. Obes.* **8**, 147-156.
- [21] Z. Ayati, M.S. Amiri, M. Ramezani, E. Delshad, A. Sahebkar and S.A. Emami (2018). Phytochemistry, traditional uses and pharmacological profile of rose hip: a review, *Current Pharm. Design.* **24**(35), 4101-4124.
- [22] S. Patel (2013). Rose hips as complementary and alternative medicine: overview of the present status and prospects, *Mediterr. J. Nutr. Metab.* **6**(2), 89-97.
- [23] S. Georgieva, G. Angelov and S. Boyadzhieva (2014). Concentration of vitamin c and antioxidant activity of rosehip extracts, *J. Chem. Technol. Metal.* **49**(5), 451-4.
- [24] J. Gruenwald, R. Uebelhack, and M.I. Moré (2019). *Rosa canina*–Rose hip pharmacological ingredients and molecular mechanics counteracting osteoarthritis–A systematic review, *Phytomedicine* **60**, 152958.

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- [25] G. Özek, T. Özek, K. Başer, A. Duran, M. Sagioglu and H. Duman (2006). Comparison of the essential oils of *Prangos turcica* A. Duran, M. Sagioglu et H. Duman fruits obtained by different isolation techniques, *J. Essent. Oil Res.* **18(5)**, 511-514.
- [26] G. Özek, F. Demirci, T. Özek, N. Tabanca, D.E. Wedge, S.I. Khan, K.H.C. Başer, A. Duran and E. Hamzaoglu (2010). Gas chromatographic–mass spectrometric analysis of volatiles obtained by four different techniques from *Salvia rosifolia* Sm., and evaluation for biological activity, *J. Chrom. A.* **1217(5)**, 741-748.
- [27] É. Héthelyi, S. Szarka, É. Lemberkovics and É. Szöke (2010). SPME-GC/MS identification of aroma compounds in rose flowers, *Acta Agr. Hungarica* **58(3)**, 283-287.
- [28] R. Möller, G. Nürnberg, E. Albrecht, W. Ruth, G.A. Brockmann and D. Dannenberger (2019). A method for analyzing fatty acids in cattle hair, with special emphasis on lauric acid and myristic acid, *Eur. J. Lipid Sci. Technol.* **121(11)**, 1900143.
- [29] WILEY&NIST (2011). Wiley - NIST GC/MS Library Ver: W9N11.
- [30] K. Hochmuth, W.A. König, and D. Julain. MassFinder 4 software tool (http://massfinder.com/wiki/MassFinder_4); Ver: 4.
- [31] R.P. Adams (2007). Identification of Essential Oil Components by Gas Chromatography/Mass Spectroscopy. Allured Pub. Corp.
- [32] V.I. Babushok, P.J. Linstrom and I.G. Zenkevich (2011). Retention indices for frequently reported compounds of plant essential oils, *J. Physical Chem. Refer. Data.* **40(4)**, 043101.
- [33] J. Paolini, J. Costa and A.-F. Bernardini (2005). Analysis of the essential oil from aerial parts of *Eupatorium cannabinum* subsp. *corsicum* (L.) by gas chromatography with electron impact and chemical ionization mass spectrometry, *J. Chrom. A.* **1076(1-2)**, 170-178.
- [34] J.B. Boti, A. Muselli, F. Tomi, G. Koukoua, T.Y. N'Guessan, J. Costa and J. Casanova (2006). Combined analysis of *Cymbopogon giganteus* Chiov. leaf oil from Ivory Coast by GC/RI, GC/MS and ¹³C-NMR, *Comptes Rendus Chimie.* **9(1)**, 164-168.
- [35] J.S. Elmore, I. Nisyrios and D.S. Mottram (2005). Analysis of the headspace aroma compounds of walnuts (*Juglans regia* L.), *Flav. Fragr. J.* **20(5)**, 501-506.
- [36] S. Vichi, J.M. Guadayol, J. Caixach, E. López-Tamames and S. Buxaderas (2007). Comparative study of different extraction techniques for the analysis of virgin olive oil aroma, *Food Chem.* **105(3)**, 1171-1178.
- [37] F. Bianchi, M. Careri, A. Mangia and M. Musci (2007). Retention indices in the analysis of food aroma volatile compounds in temperature-programmed gas chromatography: Database creation and evaluation of precision and robustness, *J. Separ. Sci.* **30(4)**, 563-572.
- [38] A. Cork, S. Alam, A. Das, C. Das, G. Ghosh, D. Farman, D. Hall, N. Maslen, K. Vedham and S. Phythian (2001). Female sex pheromone of brinjal fruit and shoot borer, *Leucinodes orbonalis* blend optimization, *J. Chem. Ecol.* **27(9)**, 1867-1877.
- [39] A. Casilli, E. Decorzant, A. Jaquier and E. Delort (2014). Multidimensional gas chromatography hyphenated to mass spectrometry and olfactometry for the volatile analysis of citrus hybrid peel extract, *J. Chrom. A.* **1373**, 169-178.
- [40] P. Steullet and P.M. Guerin (1994). Identification of vertebrate volatiles stimulating olfactory receptors on tarsus I of the tick *Amblyomma variegatum* Fabricius (Ixodidae), *J. Compar. Physiol. A.* **174(1)**, 27-38.
- [41] G. Özek, T. Özek, G. Işcan, K.H.C. Başer, E. Hamzaoglu and A. Duran (2007). Composition and antimicrobial activity of the essential oil of *Tanacetum cadmeum* (Boiss.) Heywood subsp. *orientale* Grierson, *J. Essent. Oil Res.* **19(4)**, 392-395.
- [42] H. Merle, M. Verdeguer, M.A. Blázquez and H. Boira (2007). Chemical composition of the essential oils from *Eriocephalus africanus* L. var. *africanus* populations growing in Spain, *Flav. Fragr. J.* **22(6)**, 461-464.
- [43] C. Peng, R. Hua, and D. Maltby (1992). Prediction of retention indexes: IV. Chain branching in alkylbenzene isomers with C10–13 alkyl chains identified in a scintillator solvent, *J. Chrom. A.* **589(1-2)**, 231-239.
- [44] A. Karami, M. Khosh-Khui, H. Salehi, M.J. Saharkhiz and V. Rowshan (2013). Headspace analysis of floral scent from two distinct genotypes of Iranian Damask Rose (*Rosa damascena* Mill.), *J. Essent. Oil Bear. Plants.* **16(4)**, 489-498.
- [45] E. Murru, G. Carta, L. Cordeddu, M.P. Melis, E. Desogus, H. Ansar, Y. Chilliard, A. Ferlay, C. Stanton and M. Coakley (2018). Dietary conjugated linoleic acid-enriched cheeses influence the levels of circulating n-3 highly unsaturated fatty acids in humans, *Int. J. Molec. Sci.* **19(6)**, 1730 (16 pğages).
- [46] P. Acosta-Montaño and V. García-González (2018). Effects of dietary fatty acids in pancreatic beta cell metabolism, implications in homeostasis, *Nutrients* **10(4)**, 393 (14 pages).

- [47] R. Zárate, N. el Jaber-Vazdekis, N. Tejera, J.A. Pérez and C. Rodríguez (2017). Significance of long chain polyunsaturated fatty acids in human health, *Clinic. Translat. Medicine*. **6(1)**, 1-19.
- [48] K. Szentmihályi, P. Vinkler, B. Lakatos, V. Illés and M. Then (2002). Rose hip (*Rosa canina* L.) oil obtained from waste hip seeds by different extraction methods, *Biores. Technol.* **82(2)**, 195-201.
- [49] F. Marangoni, C. Agostoni, C. Borghi, A.L. Catapano, H. Cena, A. Ghiselli, C. La Vecchia, G. Lercker, E. Manzato and A. Pirillo (2020). Dietary linoleic acid and human health: Focus on cardiovascular and cardiometabolic effects, *Atherosclerosis* **292**, 90-98.
- [50] M.E. O'Reilly, Y.M. Lenighan, E. Dillon, S. Kajani, S. Curley, R. Bruen, R. Byrne, A.M. Heslin, A.P. Moloney and H.M. Roche (2020). Conjugated Linoleic acid and alpha linolenic acid improve cholesterol homeostasis in obesity by modulating distinct hepatic protein pathways, *Mol. Nutr. Food Res.* **64(7)**, 1900599.
- [51] H. Van den Dool and P.D. Kratz (1963). A generalization of the retention index system including linear temperature programmed gas-liquid partition chromatography, *J. Chromatogr. A*. **11**, 463-471.
- [52] S. Selli, C. Rannou, C. Prost, J. Robin and T. Serot (2006). Characterization of aroma-active compounds in rainbow trout (*Oncorhynchus mykiss*) eliciting an off-odor, *J. Agric. Food Chem.* **54(25)**, 9496-9502.
- [53] T. Hanai and C. Hong (1989). Structure-retention correlation in CGC, *J. High Resol. Chrom.* **12(5)**, 327-332.
- [54] M. Miyazawa and Y. Okuno (2003). Volatile components from the roots of *Scrophularia ningpoensis* Hemsl, *Flav. Fragr. J.* **18(5)**, 398-400.
- [55] I. Mármol, C. Sánchez-de-Diego, N. Jiménez-Moreno, C. Ancín-Azpilicueta, and M.J. Rodríguez-Yoldi (2017). Therapeutic applications of rose hips from different *Rosa* species, *Int. J. Molec. Sci.* **18(6)**, 1137.
- [56] T.-K. Lin, L. Zhong and J.L. Santiago (2018). Anti-inflammatory and skin barrier repair effects of topical application of some plant oils, *Int. J. Molec. Sci.* **19(1)**, 70.
- [57] M. Michalak and A. Kiełtyka-Dadasiewicz (2018). Oils from fruit seeds and their dietetic and cosmetic significance, *Herba Polonica*. **64(4)**, 63-70.
- [58] N. Ahmad and F. Anwar (2016). Rose hip (*Rosa canina* L.) oils, In: *Essential Oils in Food Preservation, Flavor and Safety*, Elsevier, pp. 667-675.
- [59] H. Ilyasoğlu (2014). Characterization of rosehip (*Rosa canina* L.) seed and seed oil, *Int. J. Food Proper.* **17(7)**, 1591-1598.
- [60] S. Ercisli (2007). Chemical composition of fruits in some rose (*Rosa* spp.) species, *Food Chem.* **104(4)**, 1379-1384.
- [61] S. Ercisli, E. Orhan, and A. Esitken (2007). Fatty acid composition of *Rosa* species seeds in Turkey, *Chem. Nat. Comp.* **43(5)**, 605-606.
- [62] S. Machmudah, Y. Kawahito, M. Sasaki and M. Goto (2007). Supercritical CO₂ extraction of rosehip seed oil: fatty acids composition and process optimization, *J. Supercrit. Fluids.* **41**, 421-428.
- [63] R. Nowak (2005). Fatty acids composition in fruits of wild rose species, *Acta Soc. Bot. Poloniae.* **74(3)**, 229-235.
- [64] S. Kazaz, H. Baydar and S. Erbas (2009). Variations in chemical compositions of *Rosa damascena* Mill. and *Rosa canina* L. fruits, *Czech J. Food Sci.* **27(3)**, 178-184.
- [65] M. Kiralan (2015). Use of headspace solid-phase microextraction in rose (*Rosa damascena* Mill) products for volatile compounds, *J. Essent. Oil Bear. Plants.* **18(5)**, 1266-1270.
- [66] A. Dobrova (2013). Dynamics of the headspace chemical components of *Rosa damascena* Mill. flowers, *J. Essent. Oil Bear. Plants.* **16(3)**, 404-411.