MEDDOCS Open Access Publisher

ISSN: 2637-7721

Journal of Plant Biology and Crop Research

Open Access | Research Article

Seasonal Changes in Chemical Composition of *Valeriana Officinalis L.* Roots in Natural Conditions and Organic Production System in Latvia

Chem Ilva Nakurte[1]*; Ieva Mežaka[1]; Inese Taškova[1]; Andrea Primavera[1]; Alekss Vecvanags[1]; Arta Kronberga[2]

[1]Institute for Environmental Solutions, "Lidlauks", Priekuļi parish, Priekuļi county, Latvia.

[2]Field and Forest, SIA, 2 Izstades Str, Priekuļi parish, Priekuļi county, Latvia.

***Corresponding Author(s): Chem Ilva Nakurte**

Institute for Environmental Solutions, "Lidlauks", Priekuļi parish, Priekuļi county, Latvia.

Tel: 00371-26330172;

Email: ilva.nakurte@videsinstituts.lv

Received: Nov 12, 2020

Accepted: Jan 24, 2021

Published Online: Jan 29, 2021

Journal: Journal of Plant Biology and Crop Research

Publisher: MedDocs Publishers LLC

Online edition: http://meddocsonline.org/

Keywords: Valeriana officinalis L; Genetic resources; Roots; Essential oil; Sesquiterpenic acids.

Abstract

Common valerian *(Valeriana officinalis L.*) is among top selling medicinal plants in Europe and America due to its calming and sleep-promoting properties. High essential oil and sesquiterpenic acid content is required for high quality produce. This study aims to access quality of valerian roots collected in wild and harvested in organic production system in Latvia in terms of the essential oil composition and content and sesquiterpenic acid. For the first time, the quality assessment of wild Valerian grown in Latvia territory has been studied and described.Essential oil was extracted from dried roots by hydro-distillation method. Concentration of essential oil from roots harvested in natural conditions in September and October in 2017 varied from 8.4till 10.7 mL/kg, from samples harvested in April 2018 varied from 11.5 till 14.2 mL/kg. Essential oil in two-year-old roots of wild populations harvested organic production system in October 2019 varied from 8.46 till 11.3mL/kg. Separation of the biologically active substances was performed by gas chromatography-mass spectrometry, thirty-seven components were identified. The major compounds identified in the oil were bornyl acetate, valerianol, valeranone, intermedeol, camphene, myrtenyl acetate, agarospirol, and γ-eudesmol. Separation of valerenic acids was performed by liquid chromatography.The content of sesquiterpenic acids in Latvian accessions did not exceed 0.02% both in wild and in organic cultivationthus did not reach the threshold of European Pharmacopoeia (0.17 %) thus wild populations of Latvian origin cannot be directly incorporated in commercial growing.

Cite this article: Nakurte CI, Mežaka I, Taškova I, Primavera A, Vecvanags A, et al. Seasonal Changes in Chemical Composition of *Valeriana Officinalis L.* Rootsin Natural Conditions and Organic Production System in Latvia. Plant Biol Crop Res. 2021; 4(1): 1031.

Introduction

Common valerian (*Valeriana officinalis L.*) belongs to Valerianaceae family and plant root has been used worldwide over centuries for medicinal purposes. The specie is native to Europe and Asia, and has spread in eastern North America [15]. Globally there are about 300 species belonging to Valerianacea family, five of them have been observed in the wild flora of Latvia but only common valerian is widespread [16]. This perennial plant prefers moist woodlands, wet, swampy meadows, shrubs, and the water bodies [13]. Valerian has mild sedative and sleep-promoting properties, and it is often used as a milder substitute synthetic additive, such as the benzodiazepines, in the treatment of nervous states and anxiety-induced sleep disturbances [6,10]. Valerian is also a component of many herbal mixtures widely used to treat sleeping disorders. Nowadays, valerian extracts are commercially available as dietary supplements, which primarily involve dried root or extracts from the root, formulated into tablets or soft gelatin capsules [15]. Well over one hundred products which are marketed in Europe contain various extracts based on valerian, [5] making it one of the most selling natural medicine in the United States of America (USA) and Europe [4,22]. According to the State Agency of Medicines of the Republic of Latvia Statistics on Medicines Consumption 2018, valerian (as tincture) is also included in the Latvian manufacturing medical sector TOP 15[19]. *Valeriana* spp. is listed in the European (EP) and USA pharmacopeias. According to European Pharmacopeia (monograph 04/2017: 2526) the minimum content of essential oil must be 4 mL·kg^{-1}expressed on a dry root weight. Since 1980 there has been rising interest in valerenic acids and analytical procedures for these compounds have been developed for valerian [11]. As a result, analytical methods for determination of valerenic acids have been included in European Pharmacopeia which define that the content of sesquiterpenic acids (%) in valerian samples should be more than 0.17% m/m (Ph Eur 9.0 2017). The market demands high-quality valerian raw material, preferably organically grown with desirable chemical profile [3,9]. Essential oil content and composition is influenced by several factors such as genotype, geographical location, and climate [3]. There are limited studies on chemical composition of wild valerian [14,20] and no studies have assessed quality of the wild and cultivated valerian in Latvia. The current research is the very first report to essential oil composition and content, and sesquiterpenic acid content in valerian roots collected in wild and harvested in organic production system in Latvia. This preliminary study has been carried out to provide an initial screening for commercial growing of wild valerian populations in organic farming system.

Methods and materials

Sample collection

Totally five wild populations of common valerians elected for the study. Root samples were collected during the period from August to September in 2017 and in April 2018 at the beginning of vegetation (Table 1). In each site roots of 10 randomly selected plants were collected. Roots were washed, chopped, and dried at temperature 40°C [24] until the residual moisture was below 12%. Loss of drying using gravimetry method was detected for each sample. The amount of essential oil and ingredient contents were calculated based on the valerian root dry mass. Seeds from wild populations were collected and used for propagation of plantlets for field trials of wild populations under organic farming system. 'Lubelski', 'BLBP', and 'Stamm Phasa' were used as check varieties. Field trials were set in 2018 and 2019 on organically certified field of SIA 'Field and Forest' (57°19'22.7"N 25°19'11.8"E, 115 m altitude). The field assays were set up as randomized complete block designs, three replications for each accession. The plot size was 60 m^2 (8 rows). Plants were spaced at 40 cm in rows and 75 cm between rows with density 3.3 plants per m^2. The seeds were sown in May 2018 in trays in a greenhouse. Sixty days old plantlets were planted in agricultural fields on the 10 July 2018. The experiment was conducted under organic farming system, without any use of fertilizers or pesticides. Granular lime 600 kg/ha was applied before planting. The soil properties at the experimental field were as follows: Soil type: Stagnicluvisol, sandy loam, PH_{KCL}5.7%, organic matter 2.4%. Soil is characterized by low P_2O_5 (93 mg/kg) and medium K_2O (131 mg/kg).To control weeds, interrows were regularly cultivated and rows were weeded. The roots were harvested within five days starting on the 25 October, 2019. The roots of randomly selected 20 plants from each replication were harvested manually. Plants from outer rows were not harvested.

Essential oil - sample preparation, sample analyses and content measurements

Amount of essential oil was determined based on the European Pharmacopeia 9.0 as follows. Ten grams of freshly powdered mixed valerian root sample was subjected to hydro-distillation for 4h using a Clevenger type apparatus. Grinded material was transferred into 500 mL flask, water as the distillation liquid was added and 0.50 mL of 1,2,4-trimethylbenzene as oil capturer in graduated tube. Distillation were performed at a rate of 3-4 mL/min. The oil dissolved in the organic layer was separated and dried over anhydrous sodium sulphate to eliminate moisture. Samples were preserved in a sealed vial at 4°C until GC-MS analysis.

Organic acids and terpenoids - sample preparation, sample analyses and content measurements

The volatile constituents from collected roots of valerian were investigated using GC/MS (Gas chromatography- mass pectrometry) method. 100 µl of test solution of essential oil was diluted with 900 µl of cyclohexane. Analyses were performed on an Agilent Technologies 7820A gas chromatograph coupled to Agilent 5977B Mass Selective Detector (MSD) equipment. An apolar HP-5 capillary column (30 m × 0.25 mm, 0.25 µm film thicknesses) coated with 5%phenyl, 95% methyl polysiloxane (Agilent Technologies, U.S.A.). The carrier gas was helium (6.0) with the split ratio of 1:150, and the flow rate of 1.8 mL/min was applied. The oven temperature was programmed from 60°C for 3 min, then from 60°C to 280°C at 6°C/min. The injector temperature was 280°C. Mass spectra were recorded at 70 eV. Mass range was from m/z 50-550. The ion source temperature was maintained at 230°C. The oil components identified based on their retention indices (determined with reference to homologous series of C8–C20 n-alkanes), by comparison of their mass spectra with those stored in NIST (National Institute of Standards and Technology) MS search 2.2 library. The amount (%) of separated volatile compounds was calculated in peak areas using the normalization method without correction factors.

Sesquiterpenic acids - sample preparation, sample analyses and content measurements

Methanolic extracts of powdered root samples were prepared by dissolving 1.2g sample into plastic centrifugal tube and 40 ml of methanol was added, and sample was incubated

in ultrasound bath for 45 min at 70°C. Samples were cooled, centrifugated for 10 min at 4400 rpm, and filtrated through a membrane filter with a nominal pore size 0.45 µm, and injected into LC system. The standard solution corresponding to 0.1 mg/mL valerenic acid was prepared by dissolving 236 mg of reference standard valerian dry extract (European Pharmacopoeia Reference Standard Y0000583, Batch: 3.2) in 10 mL of methanol. The solution was sonicated for 10 min and filtered through a membrane filter with a nominal pore size 0.45 µm. Chromatographic analyses were performed on a HPLC system Agilent 1290 Infinity II series (Agilent Technologies, Germany). LC separations were achieved based on the European pharmacopeia by using an Agilent Eclipse XDB-18 3.5 µm, 4.6 x 150 mm (Zorbax) column (25°C) with a mobile phase A, composed of acetonitrile and 5 g/L solution of phosphoric acid (20:80 v/v) and mobile phase B, composed of 5 g/L solution of phosphoric acid and acetonitrile (20:80 v/v) at a flow rate of 0.9 mL/min. The injection volume was 7 µL. Chromatograms were obtained on an Agilent WVD detector (Agilent Technologies, Germany) at a wavelength of 220 nm. The experimental data were handled using ChemStation 32 software (Agilent Technologies). For peak identification, retention times (t_R) for standard solution and analyzed samples were compared. The quantitative determination of sesquiterpenic acids in the samples was provided by inserting the related chromatographic peak areas in mathematical equation, based on EP.

Statistical analyses

Data analysis was carried out in the R version 3.5.3. The statistical significance of effect of harvest time on volatile compound composition was determined by analysis of variance.

Heat map was created with scaled data in R package pheat map [8].

Results and discussion

Essential oil content of valerian roots collected in wild

Five wild populations from Latvia were analyzed in this study. Concentration of essential oil from samples harvested in wild in autumn (2017) varied from 8.4 till 10.7 mL/kg and had greater essential oil in harvest in next spring (2018) – 11.5 – 14.2 mL/kg (Table 2). EP determines that the total content of essential oil in valerian dried roots should exceed 4 mL/kg and thus all studied wild populations meet the criteria. This is the first study to report essential oil content of wild-collected valerian roots in Latvia. Previous studies in Estonia and in commercial samples from retail pharmacies or health shops in different countries (Belgium, Czech Republic, France, Germany, Greece, Hungary, Latvia, Lithuania, Moldova, Russia, United Kingdom (Sctoland), Ukraine) reported essential oil content in valerian roots varies from 0.19 till 1.16 % [17,18]. Essential oil content in valerian samples cultivated in Lithuania were 0.55 % [1]. Essential oil obtained from valerian roots collected in Bulgaria yielded 0.40-0.42% [2]. Study in wild populations in Hungary observed essential oil variation from 0.32 ml/100g (3.2 mL/kg) to 0.60 ml/100g (6.0 mL/kg) [20]. Essential oil in wild roots harvested in mountainous area in Serbia yielded 1.88 % [14]. Variation of essential oil in different growing phases and harvesting times is in line with previous studies e.g. The oil content in valerian plants cultivated in Poland varied from 0.8% (the beginning of vegetation) to 1.3% (full bloom), and from 0.75% (after the first frost) to 1.25% (three weeks after seed harvest) [21]. In China root harvest in September and November has been found to be preferable [4].

Organic acids and terpenoids in roots of wild valerian

During the last years, gas chromatography–mass spectroscopy has been established as a fast efficient and relatively simple technique for separation and analysis of a mixture of volatile substances and nowadays this method is being extensively used for routine chemical screening analyses. Chemical composition of essential oil extracted from totally five *V. officinalis* root samples harvested in territory of Latviausing GC-MS method were evaluated. The identified compounds are presented in Table 3.

Totally thirty-seven different compounds have been identified and all the identified components have been reported previously in valerian root oil [1,2,12,14,17]. Of those, eight compounds (Figure 1) were dominant - bornyl acetate (18.44-36.94%), valerianol (8.69-22.89%), valeranone (7.17-13.59%), intermedeol (0-11.84%), camphene (1.04-11.18%), myrtenyl acetate (3.38-6.77%), agarospirol (3.5-8.71%), and γ-eudesmol (1.17-5.37%). In all investigated samples monoterpenic ester bornyl acetate was the most dominant compound. Bornyl acetate was predominant in valerian root oil in earlier studies in Estonia [18], Poland [12], Lithuania [1], France, Moldavia, and Russia[17]. Cyclopentanoid sesquiterpene valerianol is the next dominant compound in investigated samples. A high content of valerianol was found in oil of some valerian roots harvested in Estonia [18], Serbia [23], and in some samples harvested in the Netherlands [3]. Some of the compounds found in other studies like valerenal, 15-acetoxy valeranone, valerenic acid were not detected in this study. There is no consensus concerning impact of the harvesting time on quantity and quality of chemical compounds. The characteristics of herb are known to be affected by genetic, environmental factors, including precipitation, temperature and edaphic conditions, and their interaction effects [9] as well as its polymorphous form. Concentrations of valerenic acid, valerenal, and α-humulene increase in their roots as they age [3,9].

Sesquiterpenic acidsin roots of wild valerian

This is the first study to determine amount of valerenic (or sesquiterpenic) acids in wild valerian plants in Latvia. Results of sesquiterpenic acid amount in wild samples harvested in 2017 and 2018 are very similar and ranged from 0.002% till 0.014 % (Table 2) not meeting the 0.17% m/m threshold set by EP.

Chemical composition of valerian roots in organic farming system

Chemical variability of wild Latvian valerian was compared with commercial check varieties in field trials under organic farming system. Essential oil content in check varieties of valerian varied from 6.8 to 8.7 and in local populations from 8.5 to 11.3 and thus both wild accessions and control varieties grown in organic farming system exceeded the threshold of European pharmacopoeia (4 mL/kg). However, sesquiterpenic acid content was high in check varieties (0.28 – 0.35%) and exceeded the EP standard (0.17%), but local accessions had very low sesquiterpenic acid content (<0.02%). Latvian wild populations and commercial varieties showed contrasting chemical profiles (Figure 1). Main constituent of essential oil of local wild populations was bornyl acetate (21.6 – 28.8 %), they also had relatively high agarospirol, myrtenyl acetate, valerenal, intermedeol and camphene levels and higher total essential oil content. VAVRI23 was characterized with high valerianol content (6.7 %), but other Latvian wild accessions were valerianol free. Main con-

stituent of essential oil of check varieties was valerenal, they also had high isosphthulenol, valeranon, alloaromadendrene and sesquiterpenic acid content relatively to Latvian populations. Previous studies have employed main constituents of essential oils to determine chemotypes [25]. Houghton et al., [7] proposed three valerianplant essential oil chemotype profiles (A, B, and C), based on the amount of kessane derivatives, elemol, valerenal, and valeranone. The oil profile of wild samples in this study did not match any of the three Houghton profiles, because besides valerenal and valeranone samples were rich in bornyl acetate. This oil profile corresponds previous study by Raal, et.al., [17,18] who observed that bornyl acetate/valerenal chemotype was characteristic for 4 of the 5 samples cultivated in Estonia and 9 of the 15 samples of valerian roots from other European countries. Growing conditions in Latvia are suitable for quality production or valerian root, however wild genetic resources cannot be directly incorporated into commercial growing without further breeding. Latvian valerian can be exploited as donor of high bornyl acetate, essential oil, and valerianol.

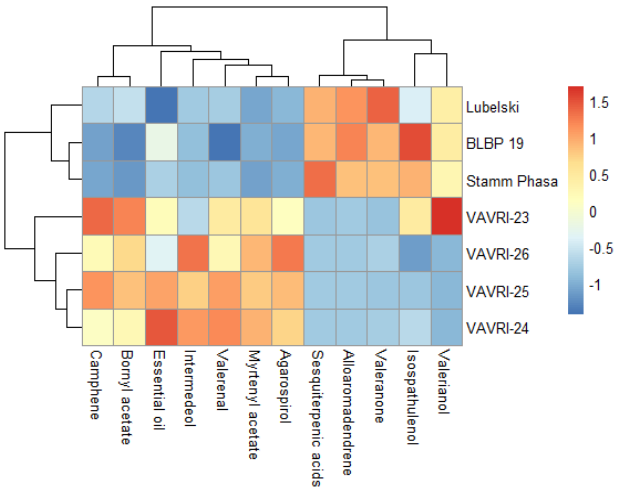

Figure 1: Relative amount of sesquiterpenic acid content, essential oil content and ten most dominant compounds of essential oil from *Valeriana officinalis L.* root harvested under organic farming conditions in October 2019.

Table 1: List of wild common valerian (*Valeriana officinalis L.)* samples collected on the territory of Latvia during 2017 and 2018.

Accession number	Accession name	Collecting data	Latitude, longitude of collecting site	Elevation of collecting site, meters above sea level	Description of collecting site
VAVRI 23	Saulriti, Cesis	18.08.2017., 10.04.2018.	57°17'22.7"N 25°18'33.6" E	120	Ditch edges, loamy soil
VAVRI 24	Renceni, Amatas county, Amatas parish	18.08.2017., 10.04.2018.	57°11'22.4"N 25°19'33.9" E	163	Permanent grassland, loamy soil
VAVRI 25	Doles, Amatas county, Amatas parish	18.08.2017., 10.04.2018.	57°08'15.9"N 25°26'23.2" E	187	Shaded forest edge, loamy soil
VAVRI 26	Plavu velki, Amatas county, Drabesu parish	18.08.2017., 10.04.2018.	57°13'35.65" N 25°8'26.519" E	126	Permanent grassland under powerline; loamy soil
VAVRI 29	Raunas-Smiltenes road, Raunas county	26.09.2017., 10.04.2018.	57°20'49.1" N 25°39'08.8" E	145	Clear-cut; moist, clay soil

Table 2: Mean values of Sesquiterpenic acid and essential oil content roots of wild-harvested valerian in 2017 and 2018.

Parameter	VAVRI 23		VAVRI 24		VAVRI 25		VAVRI 26		VAVRI 29	
	2017	2018	2017	2018	2017	2018	2017	2018	2017	2018
Sesquiterpenic acids, %	0.002	0.006	0.002	0.013	0.002	0.006	0.002	0.014	0.001	0.009
Essential oil content, mL/kg	8.5	14.2	10.7	13.1	9.8	11.5	8.8	11.5	8.4	13.2

Table 3: Essential oil composition (%) of the wild *V.officinalis L.* roots harvested in territory of Latvia from autumn (2017) and spring (2018) harvest.

No.	RI[a]	Compound[b]	VAVRI 23		VAVRI 24		VAVRI 25		VAVRI 26		VAVRI 29		Range	Median [c]	Median [c]
			2017	2018	2017	2018	2017	2018	2017	2018	2017	2018	n=10	2017	2018
1	937	α-Pinene	0.46	1.28	0.55	1.45	1.71	1.12	0.74	0.63	n.d.	0.44	0-1.71	0.55	1.12
2	950	α-Fenchene	0.33	1.27	1.55	2.66	n.d.	1.66	n.d.	n.d.	n.d.	n.d.	0-2.66	0	1.27
3	952	Camphene	3.5	7.47	2.91	8.86	11.18	9.69	5.72	4.85	1.04	5.08	1.04-11.18	3.50	7.47
4	979	β-Pinene	0.59	1.06	0.43	1.28	1.76	1.01	0.91	0.79	n.d.	0.56	0-1.76	0.59	1.01

5	1030	Limonene	0.5	0.63	0.26	1.04	0.67	0.63	n.d.	n.d.	n.d.	n.d.	0-1.04	0.26	0.63
6	1167	Borneol	0.97	2.33	1.23	1.84	0.85	1.69	0.36	0.59	2.41	1.91	0.36-2.41	0.97	1.84
7	1177	Terpinen-4-ol	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	0.54	n.d.	n.d.	n.d.	0-0.54	0	0
8	1195	Myrtenol	0.55	1.17	1.08	1.46	0.65	1.38	0.31	n.d.	0.95	1.18	0-1.46	0.65	1.18
9	1285	Bornyl acetate[d]	30.44	37.44	18.44	32.07	29.25	33.24	30.36	37.57	25.63	36.94	18.44-37.57	29.25	37.44
10	1327	Myrtenyl acetate	5.76	3.92	4.01	6.58	6.26	6.77	4.54	6.73	3.38	6.30	3.38-6.77	4.54	6.58
11	1391	7-Epi-sesquithujene	n.d.	n.d.	0.22	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	0-0.22	0	0
12	1419	Caryophyllene	0.7	1.03	0.96	n.d.	0.49	0.58	0.68	1.06	0.68	0.51	0-1.06	0.68	0.58
13	1422	2,5-Dimethoxy-p-cymene	n.d.	n.d.	n.d.	n.d.	0.39	0.45	n.d.	n.d.	n.d.	n.d.	0-0.45	0	0
14	1432	β-Gurjunene	n.d.	n.d.	0.82	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	0-0.82	0	0
15	1443	Guaia-6,9-diene	n.d.	n.d.	0.39	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	0-0.39	0	0.49
16	1454	α-Caryophyllene	0.96	0.39	1.18	0.49	0.84	0.75	n.d.	1.53	0.58	n.d.	0-1.53	0.84	5.00
17	1480	γ-Curcumene	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	1.29	0.81	n.d.	n.d.	0-1.29	0	0
18	1481	Germacrene D	1.55	0.94	2.36	0.75	1.14	1.06	1.05	n.d.	0.79	n.d.	0-2.36	1.14	0.75
19	1492	Valencene	0.59	n.d.	n.d.	n.d.	0.5	4.05	0.33	1.19	n.d.	n.d.	0-4.05	0.33	0
20	1495	Bicyclogermacrene	2.74	2.08	4.3	2.32	2.51	n.d.	6.01	6.45	3.54	2.46	0-6.45	3.54	2.32
21	1509	β-Bisabolene	0.57	n.d.	0.59	n.d.	n.d.	n.d.	0.32	0.55	n.d.	n.d.	0-0.59	0.32	0
22	1515	Cubebol	1.41	0.48	1.52	0.59	1.31	0.69	1.01	1.88	1.18	0.50	0.48-1.88	1.31	0.59
23	1537	Kessanyl acetate	n.d.	n.d.	0.64	0.52	n.d.	n.d.	0.36	0.38	n.d.	n.d.	0-0.64	0	0
24	1554	Pacifigorgiol	n.d.	n.d.	0.29	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	0-0.29	0	0
25	1549	Elemol	0.40	n.d.	1.63	0.55	n.d.	n.d.	n.d.	n.d.	0.60	1.95	0-1.95	0.40	0
26	1557	Germacrene B	0.85	0.69	0.62	0.56	0.78	0.66	0.84	0.48	0.43	0.55	0.43-0.85	0.78	0.56
27	1574	Germacrene D-4-ol	0.57	n.d.	0.66	0.40	0.68	0.36	0.35	0.40	n.d.	n.d.	0-0.68	0.57	0.36
28	1574	Maali alcohol	n.d.	n.d.	1.86	n.d.	n.d.	n.d.	n.d.	n.d.	0.62	n.d.	0-1.86	0	0
29	1576	Spathulenol	n.d.	n.d.	0.91	0.47	0.75	0.36	0.41	n.d.	n.d.	n.d.	0-0.91	0.41	0
30	1600	Rosifoliol	1.37	n.d.	n.d.	n.d.	n.d.	n.d.	0.31	n.d.	n.d.	n.d.	0-1.37	0	0
31	1619	10-epi-γ-Eudesmol	0.84	0.38	0.55	0.46	0.71	n.d.	0.65	0.39	0.71	n.d.	0-0.84	0.71	0.38
32	1638	γ-Eudesmol	4.37	5.37	4.45	4.19	2.82	1.88	2.11	1.17	4.02	2.30	1.17-5.37	4.02	2.3
33	1645	Agarospirol	7.23	5.44	5.98	3.5	4.61	4.28	6.37	3.70	8.71	6.03	3.5-8.71	6.37	4.28
34	1661	Valerianol	16.62	19.46	17.05	11.59	12.21	11.26	15.29	8.69	22.89	15.04	8.69-22.89	16.62	11.59
35	1667	Intermedeol	4.88	n.d.	7.13	5.41	4.97	5.20	8.22	11.84	9.9	4.66	0-11.84	7.13	5.2
36	1677	Valeranone	10.31	7.17	14.28	10.95	12.08	11.22	10.59	7.64	11.94	13.59	7.17-14.28	11.94	10.95
37	1723	Vetiselinenol	0.96	n.d.	0.51	n.d.	0.85	n.d.	0.32	0.66	n.d.	n.d.	0-0.96	0.51	0

[a] Retention indexes (RI) determined on HP-5MS capillary column
[b] According to NIST data base
[c] Confidence level 99%
[d] Harvest time has significant effect on concentration (p<0.05 ANOVA)
[n.d.] not detected

Conclusions

Valerianhas long been used for its pharmaceutical properties. Chemical composition of wild valerian is not much explored, particularly in organic farming system. A total of 37 compounds were identified in essential oil of five valerian populations harvested in wild and grown in organic farming system. Major components of essential oil are bornyl acetate, valerianol, valeranone, intermedeol, camphene, myrtenyl acetate, agarospirol, and γ-eudesmol. Results indicate that roots of wild-harvested valerian and organically cultivated local wild valerian populations have sufficient essential oil content (>4 mL/kg), but not sesquiterpenic acid content (<0.17%). Therefore, quality herbal product cannot be wild-harvested in Latvia and wild accessions cannot be directly used for commercial growing. Varieties of valerian can be successfully cultivated in temperate climate of

Latvia under organic farming system and quality criteria of European pharmacopoeia can be met.

Acknowledgments

The study was funded by the European Regional Development Fund roject No.1.1.1.1/16/A/307 “Growing Genetic Diversity of Medicinal and Aromatic Plants (MAPs)”.

Conflict of interests

The authors declare that there is no conflict of interests regarding the publication of this paper.

References

1. Baranauskiene R. Essential oil composition of Valeriana officinalis ssp. officinalis grown in Lithuania. Chem. Nat. Compd. 2007; 43: 331–333.
2. Bos R, Hendriks H, Pras N, St. Stojanova A, GeorgievEV. Essential oil composition of valeriana officinalis ssp. Collina cultivated in Bulgaria. J. Essent. Oil Res. 2000; 12: 313–316.
3. Bos R, Woerdenbag HJ, Van PuttenFMS., HendriksH, Scheffer JJC. Seasonal variation of the essential oil, valerenic acid and derivatives, and valepotriates in Valeriana officinalis roots and rhizomes, and the selection of plants suitable for phytomedicines. Planta Med. 1998; 64: 143–147.
4. Chen H-W, Wei B-J, He X-H, Liu Y, Wang J. Chemical Components and Cardiovascular Activities of Valeriana spp. Evidence-Based Complement. Altern. Med. 2015: 947619.
5. Committee on Herbal Medicinal Products. Assessment report on Valeriana officinalis L, radix and Valeriana officinalis L, aetheroleum. 2016.
6. Dumur J, Csajka C, Pavec O, Messaoudi S, Cretignier T, et al. Which alternative to benzodiazepines, Z-pills and other hypnotics for aged people ? Melatonin, valerian, or clomethiazole. Rev. Med. Suisse. 2018: 14; 2018–2023.
7. Houghton PJ. Valeriana, in: Medicinal and Aromatic Plants – Industrial Profiles: Harwood Academic, Amsterdam. 1997.
8. Kolde R. pheatmap : Pretty Heatmaps. R Packag. 2015.
9. Letchamo W, Ward W, Heard B, Heard D. Essential oil of Valeriana officinalis L. cultivars and their antimicrobial activity as influenced by harvesting time under commercial organic cultivation. J. Agric. Food Chem. 2004: 52; 3915–3919.
10. Miyasaka LS, Atalla A.N, Soares BGO. Valerian for anxiety disorders. Cochrane Database Syst. Rev. 2006.
11. NavarreteA, Avula B, Choi Y-W, Khan IA. Chemical fingerprinting of valeriana species: Simultaneous determination of valerenic acids, flavonoids, and phenylpropanoids using liquid chromatography with ultraviolet detection. J. AOAC Int. 2006: 89: 8–15.
12. Nurzyńska-Wierdak R. Biological active compounds from roots of Valeriana officinalis L. cultivated in south-eastern region of Poland. Farmacia. 2014.
13. Patočka J, Jakl J. Biomedically relevant chemical constituents of Valeriana officinalis. J. Appl. Biomed. 2010.
14. Pavlovic M, Kovacevic N, Tzakou O, Couladis M. The Essential Oil of Valeriana officinalis L. s.l. Growing Wild in Western Serbia. J. Essent. Oil Res. 2004: 16; 397–399.
15. PileroodSA, Prakash J. Nutritional and medicinal properties of valerian (Valeriana officinalis) herb: A review. Int J Food Sci Nutr Diet. 2013: 1; 25–33.
16. Priedītis N. Latvijas augi. Gandrs, Rīga. 2014.
17. Raal A, Arak E, Orav A, Kailas T, Müürisepp M. Variation in the composition of the essential oil of commercial valeriana officinalis L. Roots from Different Countries. J. Essent. Oil Res. 2008: 20; 524–529.
18. Raal A, Orav A, Arak E, Kailas T, Müürisepp M. Variation in the composition of the essential oil of Valeriana officinalis L. roots from Estonia. Proc. Est. Acad. Sci. Chem. 2007: 56; 67–74.
19. SAM. Statistics on medicines consumption. 2018.
20. Sárosi S, Tulok M, Ferenczy A, Repcák M. Comparative evaluation of different valerian (Valeriana officinalis L.) lines. Int. J. Hortic. Sci. 2005: 11.
21. Seidler-Lozykowska K, Mielcarek S, Baraniak M. Content of Essential Oil and Valerenic Acids in Valerian (Valeriana offcinalis L.) Roots at the Selected Developmental Phases. J. Essent. Oil Res. 2009: 21; 413–416.
22. Smith T, Gillespie M, EcklV, Knepper J, Reynolds CM. Herbal supplement sales in US increase by 9.4% in 2018. Herbalgram. 2019.
23. Tori M, Yoshida M, Yokoyama M, Asakawa Y. A guaiane-type sesquiterpene, valeracetate from Valeriana officinalis. Phytochemistry. 1996: 41; 977–979.
24. Zlatanović IJ, Pajić MB, Rančić DV, Stevanović ZD, Dudić D. Drying kinetics and shrinkage analysis of Valeriana officinalis roots. FME Trans. 2017: 45; 142-148.
25. ZouariN. Essential Oils Chemotypes: A Less Known Side. Med. Aromat. Plants. 2013: 2; 145.