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Chemical composition of essential oils from the aerial parts and underground parts of Iranian valerian collected from different natural habitats

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ABSTRACT

Valeriana sisymbriifolia Vahl., as an Iranian endemic plant belongs to the family Valerianaceae, is widely distributed in the alpine regions of Iran. This study was done to study phytochemical characteristics of essential oils from the aerial parts (leaves, stem, and flowers) and underground parts (roots and rhizomes) of *V. sisymbriifolia* collected from four natural habitats in Southwestern Iran. The essential oils from both parts of the plant analyzed by GC and GC/MS. Results indicated that there was no significant difference among various populations for essential oil yield, while there was significant difference ($p \leq 0.01$) among different parts for oil yield. The essential oil yield of the roots and rhizomes of *V. sisymbriifolia* (0.25 ml/100 g dry matter) was higher than the aerial parts of the herb (0.08 ml/100 g dry matter). For interaction effects of population × organ, the highest essential oil yield was obtained from the underground parts of the Choobin population with 0.32 ml/100 g dry matter. The major compounds in the essential oil from the aerial parts of *V. sisymbriifolia* were derivatives of phenol (*p*-cresol) and valeric acid (*n*-valeric acid and 3-methylvaleric acid). While, hydrocarbon monoterpenes (α–pinene and camphene), oxygenated monoterpenes (borneol and bornyl acetate), and hydrocarbon sesquiterpenes (*cis*-α-bisabolene) were the main components identified in the roots of rhizomes of *V. sisymbriifolia*. In conclusion, the main source of variability in chemical composition and oil yield of the studied populations of *V. sisymbriifolia* seemed to be due to differences in harvested parts of the plant. In total, the essential oil from the aerial parts and roots of *V. sisymbriifolia* could be serving a potential source of borneol, camphene, derivatives of valeric acid, and phenol, especially *p*-cresol and for use in food, cosmetic, and pharmaceutical industries.

1. Introduction

Valeriana L., is one of the important genera of the family Valerianaceae which frequently is used as medicinal plant. The underground parts (roots and rhizomes) of *Valeriana* genus (valerian) have been used as an herbal drug to encourage sleep, improve sleep quality, and reduce blood pressure. The underground parts of the plant are perceived as a sedative, mild anodyne, hypnotic, antispasmodic, carminative, and antiepileptic (Mills and Bone, 2000). In addition, the antidepressant, antihypertensive, and anti-broncho spastic effects of the herb have been reported (Miyasaka et al., 2006). Modern interest in valerian preparations is focused on their use as a sedative and hypnotic (Barnes et al., 2002). The alcoholic extract and the essential oil of valerian showed neuro-protective properties and antimicrobial activity (Malva et al., 2004; Letchamo et al., 2004). Valerian ranks at the 8th place among the top-selling herbal supplements (Blumenthal, 2001), making very interesting the research about the chemical constituents of the species belonging to the same genus. Earlier studies have been identified the chemical compositions of the essential oil from different species of *Valeriana* (Hendriks et al., 1977; Tori et al., 1996; Bos et al., 1997, 2000; Letchamo et al., 2004; Pavlovic et al., 2004; Safaralie et al., 2010; Verma et al., 2011, 2013; Huynh et al., 2013; Thusoo et al., 2014). For example, valerian contains various compounds, including essential oil and its sesquiterpenoids (valerenic acid), iridoid (valepotriates: isovaltrate and valtrate), amino acids (GABA, tyrosine, arginine), alkaloids, phenolic acids,

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and flavonoids (Upton, 1999; Aman Zadeh et al., 2002). Generally, the main constituents of their were valeric and isovaleric acid, hydrocarbons monoterpenes (α-pinene, α-fenchene, and camphene), monoterpenic esters (bornyl acetate, myrtenyl acetate, and myrtenyl isovaleriate), oxygenated sesquiterpenes, and valerian cyclopentanoid sesquiterpenes such as valerenal, valerenone, valerenol, valerenyl acetate, valerenic acid, and valerenyl isovalerate.

Of the six species of *Valeriana* L. that grow in many remote areas of Iran (Mozaffarian, 2008), *Valeriana sisymbriifolia* Vahl. (Syn: *V. cardamines* Bieb.) has gained botanical, food, and pharmaceutical interest due to a characteristic aroma, and application in folk medicine (Ghasemi Pirbalouti, 2010). Known as "Sonboletib-e-Kohestani" or "Sonboletib-e-Irani" in Persian, *V. sisymbriifolia* is distributed in Zagros mountain range from northwestern to southwestern Iran. In addition, the plant is found in many parts of Iran, Anatolia, Turcomania, Transcavcasia, and Afghanistan (Rechinger, 1963–2005; Mozaffarian, 2008). *V. sisymbriifolia* is a perennial plant with 30–80 cm tall, oval–circular leaves and pink flowers.

The characteristics of herb are known to be affected by genetic, environmental factors, including precipitation, temperature and edaphic conditions, and their interaction effects (Letchamo et al., 1995; Ghasemi Pirbalouti et al., 2013a,b,c). Bioclimatic preferences along with geographic distances play a major role in ecotype differentiation (Rahimmalek et al., 2009) that affect plant constituency. Yet, to our knowledge, there are no documents on variation of essential oil composition and yield of wild *V. sisymbriifolia* populations due to the growth environment and/or the parts of the plant. However, other researchers have reported chemical compositions and antioxidant activity of the essential oil from the aerial parts and roots of *V. sisymbriifolia* collected from different regions of Iran (Javidnia et al., 2006; Ekhteraei Tousi et al., 2010; Ansari Dugaheh et al., 2013). Therefore, this study measured variation in chemical composition and oil yield of populations of *V. sisymbriifolia* aerial parts and underground collected from various geographical regions of southwestern Iran.

2. Materials and methods

2.1. Plant material

The aerial parts and underground parts (roots and rhizomes) four populations of *V. sisymbriifolia* were collected from Chaharmahal va Bakhtiari province, Southwestern Iran. The chosen collection areas were in different geographic areas and included areas in which differences in physical characteristics of the plant accessions were observed. Each sample was labeled and the location was recorded using a global positioning system (GPS, Vista Garmin) receiver. The physical and chemical characteristics of the soil, such as pH, electrical conductivity (EC), organic carbon, and texture, at the sample collection sites were determined (Table 1) along with climatic conditions as recorded by the nearest meteorology station. Plant identities were confirmed by Dr. H. Shirmardi (Research Center for Medicinal Plants & Ethno-Veterinary, Islamic Azad University, Shahrekord, Iran) and a representative voucher specimen (No. 2563) has been placed in the Herbarium of the Research Center of Natural Resources of Chaharmahal va Bakhtiari, Iran. The aerial parts of three replicate samples of six plants were harvested at the early flowering stage on May 2012. The roots and rhizomes of different populations were harvested after wilting of the plant on September 2012.

2.2. Sample preparation

The aerial and underground parts were dried at room temperature for six to twelve days in a shaded room at 25 ± 5 °C. Dried plant materials were grinded, and 100 g of tissue was distillated with 1 L water for 3 hrs using a Clevenger-type apparatus according to the method recommended in British Pharmacopoeia (British Pharmacopoeia, 1988). The separated oil was dried over anhydrous sodium sulfate, and stored in dark glass bottles at 4 ± 1 °C prior to use.

2.3. Identification of the oil constituents

Composition of the essential oils was determined by gas chromatography (GC) and mass spectrophotometry (GC/MS). The GC analysis was done on an Agilent Technologies 7890 GC (Agilent Technologies, Santa Clara, CA) equipped with a single injector and a flame ionization detector (FID). An apolar HP-5 capillary column (30 m × 0.25 mm, 0.25 μm film thicknesses) coated with 5% phenyl, 95% methyl polysiloxane was used. The flow of the carrier gas (N_2) was 0.8 ml/min. Initial column temperature was 60 °C and programmed to increase at 4 °C/min to 280 °C. The injector temperature was set at 280 and 300 °C. Split injection was conducted with a ratio split of 1:40. Essential oil samples of 0.1 μL were injected neat.

GC–MS analyses of aromatic oil samples were performed on an Agilent Technologies 7890 gas chromatograph coupled to Agilent 5975 C mass selective detector (MSD) and quadrupole EI mass analyzer (Agilent Technologies, Palo Alto, CA, USA). A HP–5MS 5% column (coated with methyl silicone) (30 m × 0.25 mm, 0.25 μm film thicknesses) was used as the stationary phase. Helium was used as the carrier gas at 0.8 mL/min flow rate. The temperature was programmed from 60 to 280 °C at 4 °C/min ramp rate. The injector and the GC-MS interface temperatures were maintained at 290 °C and 300 °C, respectively. Mass spectra were recorded at 70 eV. Mass range was from *m*/*z* 50-550. The ion source and the detector temperatures were maintained at 250 and 150 °C, respectively.

Oil constituents were identified based on their retention indices (determined with reference to homologous series of C5–C24 *n*-alkanes), by comparison of their mass spectra with those reported in the literature (Adams, 2007) and stored in NIST 08 (National Institute of Standards and Technology) and Willey (ChemStation data system) libraries. The peak area percentages were computed from HP-5 column without the use of FID response factors.

2.4. Statistical analyses

Simple and interaction effects of experimental factors were derived from two-way analysis of variance (ANOVA) based on the GLM procedure of the SAS statistical package (SAS/STAT® v.9.2.

Table 1
Geographical and climate of natural habitats of *Valeriana sisymbriifolia* populations.

No.	Region	Altitude (m a.s.l.)	Latitude (UTM)	Longitude (UTM)	*P*[a]	*T*	pH	E.C.	O.C	Sand	Silt	Clay
1	Tang-e-sayad (Shahrekord)	2430	0510449	3563716	394[†]	11.4	7.69	0.707	0.799	29	46.0	25.0
2	Sheyda (Ben)	2650	0460500	3604950	342	11.3	8.20	0.371	0.527	87	6.0	7.0
3	Sabz-e-kooh (Ardal)	2725	0495131	3513131	596	15.1	7.55	0.569	1.287	29	36.0	35.0
4	Choobin (Farsan)	2903	0445205	3585388	497	12.5	7.68	0.676	1.365	21	46.0	33.0

[a] *P*: annual precipitation (mm), *T*: average temperature (°C); E.C.: electrical conductivity (dS m^{-1}); O.C.: organic carbon (%), and sand, silt and clay in %.
[b]Meteorological information was obtained from weather stations located within the study area and the surrounding zone; each value in the mean of 10–15 years data.
[c]Soil characteristics are based on average of samples taken from three farms in each region.