



Volatile constituents of *Polylophium Involucratum*, a wild plant grown in Iran , extracted by HS-SPME

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Abstract

The volatile constituents of *Polylophium Involucratum* , which grown wild in the north of Iran, were extracted by head-space solid phase microextraction using a 65 µm dimethyl siloxane- divinyl benzene (PDMS-DVB) fiber in order to determine compounds. The extracted compounds were analyzed by means of gas chromatography (GC) and gas chromatography- mass spectroscopy (GC-MS). □- terpinene (20.71%), sabinene (14.86%), p-cymene (12.91%) and bornyl angelate (10.2%) were the major components in volatile fraction of *P. Involucratum*. Monoterpene. Hydrocarbons were the major compounds in head space of *P. Involucratum*. The main aim of this study is the extraction of volatile constituent of plants with an affordable method that is suitable for heat sensitive components. HS-SPME method, which needs no solvent, can be used as a Superseded method instead of hydrodistillation, due to less chemical changes in volatile composition, especially in the case of oxygenated ones, of plants.

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Introduction

Essential oils are the volatile portion of plant chemicals which have been used as folk and nowadays medicine due to their biological activity such as, antibacterial, antifungal. Essential oils are extracted by several methods such as hydrodistillation, cold expression, solvent extraction enzymatic extraction and super critical fluid extraction. Head-space solid phase micro extraction is simple and rapid technique to extraction the volatile compounds in sample which uses a fused silica fiber coated by a solid sorbent that contacted by head-space above the sample. The volatiles have been absorbed on the sorbent and desorbed thermally in gas chromatograph injection port.

The genus *Polylophium* (Umbelliferae family) has only one species in Iran, *Polylophium involucreatum*, which endemic to Iran. This plant is growing in high altitude of Northern Alborz Mountains (Mozaffarian, 1998). *P. involucreatum* is used widely as spice in cooking in north of Iran. Literary survey in two last decade revealed that only one study had been done on chemical composition of *P. involucreatum* essential oil composition. Limonene (60.39%), aldehyde and alcohol derivatives of Limonene (34.45%) were the main constituents of *P. involucreatum* essential oil. Larvicidal activity test against the *Anopheles stephensi* and *Culex pipiens* showed strong larvicidal activity of the oil (Verdian Rizi, 2007).

SPME was used to extraction and identification of volatile constituent of a plant with minimum structural changes which could accrued during extraction of essential oils by hydrodistillation method such as hydrolysis and oxidation components due to extreme heat in presence of water.

This study deals with the comparison of extraction and analysis of volatile components of *P. involucreatum* which used as an aromatic herb used in folk cooking as a spice by head space solid phase micro extraction (HS-SPME) followed by GC and GC-MS.

Material and methods

Plant material

The seeds of *P. involucreatum* were collected from Javaherdeh village, Ramsar, province of Mazandaran, Iran in July 2012. The plant material was dried in shadow at room temperature (25°C). Voucher specimen was deposited at the herbarium of Research Institute of Forest and Rangelands (TARI), Tehran, Iran.

Volatile Compounds (VCs) Extraction

volatile constituents of *P. involucreatum* were extracted by head-space solid phase microextraction (HS-SPME). For this propose, 1.5 g of powdered seed of *P. involucreatum* was placed in a 20 ml glass vial that seal with rubber septum caps perched from Suplco (Bellefonte, USA). The vial was remained at 50 °C temperature 15 min to volatile compounds released. Adsorption of released components was done using a manual SPME holder equipped with a 65µm PDMS-DVB (poly dimethyl siloxane- divinyl benzene) fiber (Suplco, Bellefonte, USA) for 15 min (Aberoomand Azar, 2013) VOCs desorption was done at 250 °C for 3 min in a splitless GC and GC-MS injector.

Volatile Compounds analyses:

GC analysis

GC analysis was done using a Shimadzu 14A gas chromatograph equipped with a DB-5 capillary column (30m × 0.25 mm, film thickness 0.32 µm) , a split/splitless injector (30:1) (250 °C) and flame ionization detector (250°C). The initial temperature of column was set at 60 °C (3 min hold time) and reached to 230 °C with a 6°C/min rate and kept for 5 min in 230°C. N₂ was used as carrier gas (1 ml/min). The retention times and area of each peak was recorded using a Shimadzu CR5 chromatopack.

GC-MS analysis

GC-MS analysis of the samples was performed by a Hewlett-Packard 6890 gas chromatograph equipped with a HP-5MS capillary column (30m × 0.25 mm, film thickness 0.32 µm) that coupled to a quadropole HP 5973 mass selective detector with 70 eV ionization

energy (mass range 40-500 amu and scan time was 1 s. The temperature programming was as like as GC condition. He (99.999%) was used as carrier gas (1 ml/min). The Chemstation software used for processing of achieved data

Identification of VCs components

characterization of VCs was done by comparison of their e mass spectra and relative retention indices with those given in authentic references (Adams, 2004). The relative retention indices were calculated

by C₈-C₂₀ normal hydrocarbons series retention time at the same GC condition.

Result and discussion

The percentage composition of *P. Involucratum* VOCs which extracted by HS-SPME is listed in table 1. Twenty four compounds were identify in HS-SPME sampling, representing 99.18% of the total sample. $\square\square$ -terpinene (20.71%), sabinene (14.86%), p-cymene (12.91%) and bornyl angelate (10.2%) were the major components in volatile fraction of *P. Involucratum*.

Table 1. Percentage composition of *P. Involucratum* volatile compounds by HS-SPME isolation methods.

Compounds	KI	HS-SPME(%)
α - thujene	930	0.88
α - pinene	939	8.48
Camphenene	954	0.48
Sabinene	975	14.86
β - pinene	979	0.87
Myrcene	991	2.33
α - terpinen	1017	0.37
p-cymene	1025	12.91
Limonene	1029	0.78
z- \square - Ocimene	1037	0.24
\square - terpinene	1060	20.71
α -terpineolene	1089	0.34
Citronellal	1153	0.69
z -anethol	1253	5.56
Geranial	1267	0.23
bornyl acetate	1289	0.31
γ - therpinal	1291	6.31
acetanisol(meta)	1299	0.34
citronelly acetate	1353	0.77
nery acetate	1362	0.31
geranyl acetate	1381	10.24
z- \square - Franesense	1443	0.23
isobornyl-3-methyl	1524	0.74
bornyl angelate	1566	10.2
Total		99.18%

As seen in table 1, hydrocarbonated monoterpene (63.25%), oxygenated monoterpenes (35.7%) and sesquiterprne hydrocarbon (0.23%) were the compound classes in the volatile fraction of *P. Involucratum* (figure 2).

By comparison of the obtained result in our research and which was done by Verdian Rizi, M.R *et al*, it revealed that the plant essential oil which obtained by water distillation was characterized by hydrocarbonated monoterpens (59.66%), oxygenated monoterpens (25.12%) and sesquiterprne

hydrocarbon (9.96%) (Verdian Rizi & Hadjiakhoondi, 2007). Similar compounds are extracted by water distillation and HS-SPME, although the relative amounts of these compounds are different. The oxygen-containing compounds obtained from the HS-SPME method is more abundant in essential oil obtained by hydrodistillation one and when the purpose of separation is study of oxygenated compounds and hydrocarbonated monoterpenes the HS-SPME method is more efficiently than water distillation.

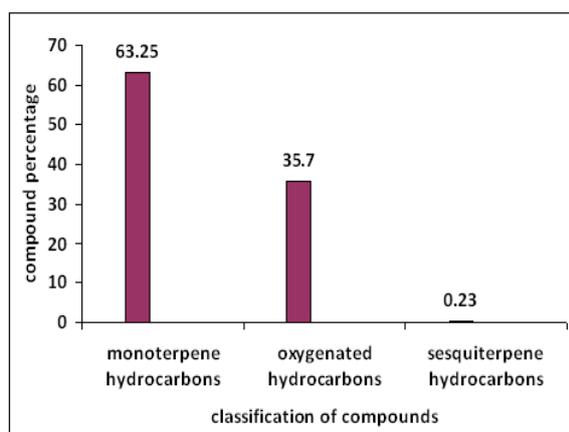


Fig. 2. Classification of volatile compounds identified in *P. Involucratum*.

Conclusion

As shown in this study, HS-SPME is a rapid, eco-friendly and cheap method to extraction and characterized the volatile fraction which are responsible of smell in an aromatic plant, mainly used as spice in food preparation.

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