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Determination of Volatile Organic Components of *Achillea millefolium* L. using Microwave Distillation Technique

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ABSTRACT

The Microwave-assisted hydrodistillation (MAHD) was applied for the first time for the extraction of volatile organic compounds of *Achillea millefolium* L from Iran. The oil obtained was analyzed by GC–MS. The extraction time while using the MAHD is no more than 24 min using a microwave power of 300W. The major component by MAHD was Nerol. Differences were observed both in the composition of the essential oil and from the energetic point of view. The essential oil obtained with microwave methods contained substantially higher amounts of oxygenated compounds and lower amounts of monoterpenes than conventional method. The in situ microwave heating is safe and versatile. It presents time and energy saving advantages, and therefore it can also be considered useful for industrial applications.

Key words: Volatile Organic compounds, *Achillea millefolium* L. Microwave Distillation.

INTRODUCTION

Achillea millefolium L. is a perennial herb from 0.6 to 1.2m high, which grows from underground horizontal rhizomes. The stem is simple, erect and hairy and leaves are lanceolate, multipinnate with short acute tips. White flowers arranged in dens cymes with small capitulate. Bracts are imbricate, long, thorn-tipped and taper to appoint. The disc florets are tubular, yellowish, white and androgynous. Fruit is 1.5 to 2 mm long 1. The genus *Achillea* (Family: Asteraceae, Section: Santolinoidea) is represented by about 85 species mostly found in Europe and Asia and a handful in North America[1]. Forty species of *Achillea* are widely distributed in Turkey[2]. As far as ethnopharmacologic background is concerned, *Achillea millefolium* is a well-known species amongst the members of *Achillea* (Asteraceae)[3]. It is known as "Bomadran" and used in folk remedies as an appetizer, wound healer, diuretic, carminative or menstrual regulator[4]**.** *Achillea* which is native to Europe, Asia, North America and distributed in low temperate zones of the world[5]. It is a commonly used herb both in the ethno-pharmacology and in the up-to-date phytotherapy, they assurea valuable source of

natural remedies[6]. It is used as an anti-inflammatory, antispasmodic, anti-pyretic, anti-septic, and as anti-dandruff in topical form (6)**.** *Achillea* essential oil, which is used in aromatherapy, is extracted by steam distillation from the dried herbs and uses for acne, burns, eczema, scars, circulation problems, high blood pressure, arthritis and thrombosis, it can also be used for constipation, cramp, hemorrhoid, common cold, fever, flu, hypertension, insomnia and stress related disorders. In recent years, therapists have used the oil on patients suffering with cancer. A wide variety of analytical methods is used to extract the volatile compounds from plant material. Techniques commonly used to extract the essential oils include steam distillation, hydrodistillation, dynamic and static headspace, supercritical fluid extraction and solvent extraction (7-10). In 2003, Chemat et al. invented the novel technique of microwave distillation (MD). (11), which is a combination of microwave heating and dry distillation at atmospheric pressure. MD was conceived for the laboratory scale extraction of essential oils from aromatic plants. Based on a relatively simple idea, MD involves placing plant material in a microwave reactor, without any added solvent or water. The internal heating of the water within the plant material distends the plant cells and leads to rupture of the glands and oleiferous receptacles. This frees essential oils which are evaporated by the water of the plant material. A cooling system outside the microwave oven condenses the distillate. The excess of water was refluxed to the extraction vessel in order to restore the water to the plant material. In this work, for the first time, microwave distillation (MD), and developed for the rapid analysis of *Achillea millefolium* L essential oil components.

MATERIAL AND METHODS

Plant material and microwave oven

Achillea millefolium L leaves were collected from Zagrous maintain in May 2010 and dried at 25–30 °C for 3 days without applying any heat treatment to minimize the loss of active components. The microwave oven with a maximum delivered power of 1000 W and 2450 MHz was purchased from Samsung Company (Korea).

MADH apparatus and procedure

Microwave-assisted hydrodistillation (MAHD) was carried out with a Samsung microwave apparatus. The multimode microwave reactor has a twin magnetron (1000 W, 2455MHz) with a maximum delivered power of 1000W variable in 10W increments. A rotating microwave diffuser ensures homogeneous microwave distribution throughout the plasma coated cavity is 35 cm × 35 cm × 35cm. Temperature was controlled by feedback to the microwave power regulator.

The experimental MADH variables have been optimized by the university method in order to maximize the yield of essential oil. In a typical MADH procedure performed at atmospheric pressure, 60 g of fresh plant material was heated using a fixed power of 600 W for 24 min without added any solvent or water. A cooling system outside the microwave cavity condensed the distillate continuously. Condensed water was refluxed to the extraction vessel in order to provide uniform conditions of temperature and humidity for extraction. The extraction was continued at 100 °C until no more essential oil was obtained. Microwave-assisted hydrodistillation is based on the combination of microwave heating and distillation and is performed at atmospheric pressure. This method involves placing vegetable material in a microwave reactor. The internal heating of the in situ water within the plant material distends it and makes the glands and oleiferous receptacles burst. This process thus frees essential oil which is entrained by the in situ water of the plant material by azeotropic distillation. The vapor then passes through a condenser outside the microwave cavity where it condensed. The distillate is collected continuously in the receiving flask. The excess of water was refluxed and recycled to the extraction vessel by cohobating in order to restore the moisture of the plant material. The essential oil is collected directly and dried without added any solvent extraction step.

Optimization of microwave power

An appropriate microwave irradiation power is important to ensure the essential oil is extracted quickly; however, the power should not be too high otherwise loss of volatile compounds would result. Different microwave irradiation power, 180, 300, 450, 600, 800, and 1000 W, were examined for MAHD extraction of essential oils. The total extraction time

(it was until no more essential oil was obtained) in relation with the microwave irradiation power was studied. A microwave irradiation power of 600 W for 60 g of plant material was the optimum microwave power density because this power permits in only 24 min to extract the essential oil completely and avoid loss of volatile compound.

Chemicals and Reagents

Helium, 99.999%, used as carrier gas was purchased from Roham Gas Company (Tehran, Iran). Alkane mixture consisting of the C8-C20 alkanes (concentration of 40 mg/mL in hexane) was purchased from Fluka. All other chemicals were of the highest purity available from Merck or Fluka. Doubly distilled deionized water was used.

GC analysis

GC analyses were carried out on a Shimutzu 17A gas chromatograph equipped with a FID and a BP-5 capillary column (30 m × 0.25 mm; 0.25 µm film thickness). The oven temperature was held at 60 °C for 3 min then programmed at 5°C / min to 300 °C. Other operating conditions were as follows: carrier gas, He with a flow rate of 5 ml/min; injector temperature, 230; detector temperature, 300 °C; split ratio, 1:8.

GC-MS analysis

GC/MS analyses were performed on a Shimutzu 17A GC coupled with Shimutzu QC5050 Mass system and a BP-5 capillary column (30 m × 0.25 mm; 0.25 µm film thicknesses). The operating conditions were the same conditions as described above but the carrier gas was He. Mass spectra were taken at 70eV. Mass range was from *m/z* 50–500amu. Quantitative data were obtained from the electronic integration of the FID peak areas. The components of the oils were identified by comparison of their mass spectra and retention indices with those published in the literature (12) and presented in the MS computer library (WILEY229.L and NIST 1988).

RESULTS AND DISCUSSION

Identification of compounds

Retention indices were calculated by using retention times of n-alkanes that were injected after the oils under the same chromatographic condition. The components were identified by comparison of their mass spectra with the Wiley library, or with the published mass spectra. The quantification of each compound was based on peak area method without using correction factor.

Qualitative and quantitative analyses

Most constituents were identified by gas chromatography through comparison of their retention indices (RIs) with those of the literature (12), or with those of authentic compounds available in our laboratories. The retention indices (RIs) were determined in relation to a homologous series of n-alkanes (C8–C24) under the same operating conditions. Further identification was made by comparison of their mass spectra on both columns with those stored in NIST 98 and Wiley 5 Libraries or with mass spectra from literature (12). Component relative concentrations were calculated based on GC peak areas without using correction factors.

Table 1: Chemical composition of the essential oil from *Achillea millefolium* L

No	Compound	RI*	MD(%)**
1	α-pinene	939	0.65
2	Limonene	1037	2.60
3	1,8-Cineole	1042	1.29
4	Linalool	1108	2.83
5	UNKNOWN	1117	4.95
6	Nerol oxide	1161	0.14
7	Camphor	1167	0.19
8	α -Terpineol	1212	0.49
9	Nerol	1240	39.65
10	Z-Citral	1282	0.30
11	α-Terpinyl acetate	1359	0.69
12	Neryl acetate	1367	34.52
13	β-Caryophyllene	1438	0.39
14	UNKNOWN	1489	0.67
15	Germacrene d	1500	1.55
16	Bicyclogermacrene	1515	0.35
17	Nerolidol E	1571	1.31
18	E-sesquilavandulol	1642	3.74
19	10-epi-gamma-eudesmol	1659	0.78
20	Farnesyl acetate	1733	1.98

*RI= retention indices in elution order, ** MD%=microwave distillation area %

DISCUSSION

The aerial parts from the flowering plants of *Achillea millefolium* L., on MAHD gave 0.14% (v/w) of oil on fresh weight basis. GC and GC-MS analysis of the oil resulted in the identification of 18 constituents, representing 97% of the oil (Table 1). Nerol (39.65%) and Neryl acetate (34.52%) were the major components.

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REFERENCES

1. Konemann, Botanica: The Illustrated A-Z of over 10,000 Garden Plants and How to Cultivate Them. Gordon Cheers Publication, Hong Kong, pp. 51–53 (1999).
2. Davis P. H., Flora of Turkey and the East Aegean Islands, vol.5. University Press, Edinburgh, p. 244, (1982).
3. Candan F., Unlu M., Bektas Tepe, Dimitra Daferera, Moschos Polissiou, Atalay Sökmen, H.Askýn Akpulat, Antioxidant and antimicrobial activity of the essential oil and methanol extracts of Achillea millefoliumsubsp. millefoliumAfan. (Asteraceae), *Journal of Ethnopharmacology*, **87**: 215-220 (2003).
4. Nemeth E., Essential oil Composition of species in the Genus Achillea, *J. Essent. Oil Research*, **17**(5): 501-512 (2005).
5 Zargari A., Medicinal plants.7th edition, Tehran University publications. pp: 161-167 (1997).
6. Lewis M., What is yarrow-*Achillea millefolium* essential oil?, Jeobp, **11**(6): 628-633 (2006). (available in; Alternative Medicine, Complementary Health Directory and Resources, United Kingdom (www.worldwidehealth.com).
7. Iranian Herbal Phrmacopoeia Food and drug organization, Ministry of Health and Medical Education, 161-167 (2002).
8. Karlova K., Essential oil from yarrow (*Achillea millefolium* complex), the content and the composition. Proceeding of 9th international conference of Horticul ture ,September 3rd-6th. Lednice, *Czech Republic*, **2**: 339-342 (2001).
9. Paulo S., Effect of the essential oil of *Achillea millefolium* L. in the production of hydrogen peroxide and tumor necrosis factor -??in murrain macrophases. RBCF, Brazilian *Journal of Pharmaceutical Sciences*, **41**(3): 401-405 (2005).
10. Massada Y., In Analysis of essential oil by Gas Chromatography and Spectrometry. Wiley New York (1976).
11. Chemat, F; Smadja, J; Lucchesi, M. E; European Patent Application. EP, 1.439.218.A1 (2003).
12. Adams R. P., Identification of Essential Oil Components by Gas Chromatography/ Quadrupole Mass Spectroscopy, Allured Publishing Corporation. Carol Stream, Illinois (2004).