Journal of Materials and Environmental Science
ISSN : 2028-2508
CODEN : JMESCN



J. Mater. Environ. Sci., 2020, Volume 11, Issue 10, Page 1574-1583

http://www.jmaterenvironsci.com

# Phytochemical analysis and comparative study of volatile compounds of *Aaronsohnia pubescens* subsp. *pubescens* aerial parts using hydrodistillation and headspace solid-phase microextraction (HS-SPME)

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*Received 22 May 2020,*
*Revised 21 Aug 2020,*
*Accepted 25 Aug 2020*

*Keywords*
- ✓ *Aaronsohnia pubescens subsp. pubescens,*
- ✓ *Essential oil,*
- ✓ *Volatile fraction,*
- ✓ *Hydrodistillation,*
- ✓ *Headspace solid-phase microextraction.*

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## Abstract

The main objective of the current research is to study is to report the comparative chemical analysis of essential oil (EO) obtained by hydrodistillation (HD) and volatile fraction (VF) detected by headspace solid-phase micro-extraction (HS–SPME) isolated from of *Aaronsohnia pubescens* subsp. *pubescens* (*APP*) using Gas Chromatography–Retention Indices (GC-RI) and GC–Mass Spectrometry (GC-MS). thirty-four volatile compounds identified in EO, representing 87% of the total oil, while HS-SPME revealed twenty-eight components constituting 78.7% of the volatile material. The chemical composition of the HS-SPME and HD extract comprised mainly of oxygenated monoterpenes and phenylpropanoids with 37.3 vs 31.9% and 26.5 *vs* 13.9%, respectively. The comparative analysis of two chemoprofiles obtained by two methods shows both qualitative as well as quantitative differences. The current study is the first report involving rapid analysis of volatile components of APP by HS-SPME.

## 1.Introduction

*Aaronsohnia pubescens* (Desf.) K. Bremer & Humphries subsp. *pubescens* (*APP*) is botanically a synonym of *Matricaria pubescens* (Desf.) Schultz (Asteraceae). It is a pleasant-scented annual plant endemic to North Africa [1] and the Canary Islands [2]. It is primarily found in non-saline wadis on preferably sandy-loamy soils. Parts of the plant are used in local folk medicine: In Morocco, this species, known locally as "*Taraght*", decoctions of the aerial flowering parts are used as mouthwash against toothache [3]. The plant is also used as a food additive for flavoring and preservation purposes [1,3]. In both Algeria and Morocco, infusions of the aerial parts are used against gastric ulcers and flatulence, respiratory disorders, dysmenorrhea, skin inflammation, fever and rheumatic diseases [4].

Nowadays, use of alternative and complementary therapies with mainstream medicine has gained the momentum. Sometimes it's called essential oil therapy. Aromatherapy uses aromatic essential oils

medicinally to improve the health of the body, mind, and spirit. It enhances both physical and emotional health. In other words, aromatherapy is one of the complementary therapies which use essential oils as the major therapeutic agents to treat several diseases [5-13].

Various methods are used for volatile extraction, such as hydrodistillation (HD), Soxhlet extraction, static headspace (HS) extraction, simultaneous distillation extraction (SDE) and supercritical fluid extraction (SFE). HD is a conventional method used to extract EOs, because it can be easily implemented in industry and has no chemical pollution. However, it has certain disadvantages, particularly, the consumption of energy and time, the deterioration of heat-sensitive compounds and the low extraction yields of EOs. Recently, Solid-phase microextraction (SPME) is an alternative technique to the traditional HD extraction, which has been successfully applied qualitatively and quantitatively for the analysis of various food samples, and flavors and aromas of many medicinal plants [14]. In this context, the aim of this work was to compare the volatile constituents of *APP* from Morocco (Figure 1), using the HD and HS-SPME extraction techniques.

**Figure 1:** *Aaronsohnia pubescens* subsp. *pubescens* in its native habitat in south-eastern of Morocco (Jorf-Errachidia)

## 2. Material and Methods

### *2.1. Plant material and EO isolation*

The aerial parts of *APP* were collected in May and June 2018 (full bloom) from Jorf-Errachidia (Morocco). Coupon specimens were deposited in the herbarium of the Faculty of Sciences and Technology of Errachidia. After that, 100 g of dried plant material was subjected to HD (3 hours) using a Clevenger-type apparatus according to the method recommended in the European Pharmacopoeia [15] and the EO yield was 0.7%.

### *2.2. Volatile compounds by HS-SPME*

The dried and pulverized aerial parts of *APP* were subjected directly to HS-SPME. The SPME fiber (Supelco) coated with divinylbenzene/carboxen/polydimethylsiloxane (DVB/CAR/PDMS, 2cm-50/30 µm) was used for extraction of the plant volatiles. Optimization of conditions was carried out using fresh aerial parts of the plant (3.5 g in a 20 mL vial) and based on the sum of total peak areas measured on GC-FID. The temperature and the equilibration time were selected, respectively, after three different experiments at 50, 70 and 90 °C, and after three different experiments at 60, 90 and 120 min. The extraction time was selected after three different experiments at 15, 30 and 60 min. After sampling, SPME fiber was inserted into the GC and GC-MS injection ports for desorption of volatile components (5 min), both using the splitless injection mode. Before sampling, each fiber was reconditioned for 5 min

in the GC injection port at 260 °C. HS-SPME and subsequent analyses were performed in triplicate. The coefficient of variation (9.6% < CV < 13.4%) calculated based on the total area obtained from the FID-signal for the samples indicated that the HS-SPME method produced reliable results. In the same way, the CV of the major compounds was always less than 15%.

### *2.3. GC-RI and GC-MS analysis*

The analysis and identification of volatiles compounds of *APP* were carried out using the methodology reported previously in our works [16-19].

## 3. Results and discussion

### *3.1. Essential oil analysis*

The chemical composition of *APP* EO was obtained from GC and GC-MS techniques. thirty-four components were characterized, which accounted to 87% of the total oil (Table 1). Hence, the dominant components of *APP* oil were established with mass percentage as follows: six hydrocarbon monoterpenes (17.3%), thirteen oxygenated monoterpenes (31%), one hydrocarbon sesquiterpene (0.6%), one oxygenated sesquiterpene (9.3%), seven nonterpenic oxygenated compounds (12.9%) and six phenylpropanoids (15.9%). Among them, Carvacrol (13.9%), α-Pinene (10.3 %), E-Anethole (10.1%), Ar-Turmerone (9.3%) and (E)-heptadeca-10. 16-dien-7-one (6.4%) were identified as major constituents of APS oil (Table 1).

**Table 1.** Chemical composition of EO and VF from *A. pubescens* subsp. *pubescens* aerial parts

| N [a] | Components | RI a [b] | RI p [c] | % HD [d] | % SPME [e] |
|---|---|---|---|---|---|
| **1** | **α-Pinene** | **931** | **1018** | **10.3** | **0.3** |
| **2** | β-Pinene | 971 | 1107 | 2.8 | 0.2 |
| **3** | α-Phellandrene | 998 | 1159 | 0.5 | - |
| **4** | P-Cymene | 1013 | 1262 | 2.3 | 0.2 |
| **5** | Limonene* | 1021 | 1195 | 1.1 | - |
| **6** | Cineole 1.8* | 1021 | 1205 | 3.9 | 0.2 |
| **7** | Z-β-Ocimene | 1026 | 1227 | 0.3 | - |
| **8** | Fenchone | 1069 | 1426 | 0.2 | 0.6 |
| **9** | Ethyl heptanoate | 1080 | 1325 | 0.4 | - |
| **10** | Linalool | 1085 | 1535 | 0.7 | 2.1 |
| **11** | Dihydrolinalol | 1117 | - | - | 0.3 |
| **12** | Cis-iso-pulegone | 1145 | 1564 | - | 1.0 |
| **13** | Camphre | 1122 | 1502 | 1.3 | - |
| **14** | Terpinen-4-ol | 1163 | 1588 | 1.1 | - |
| **15** | α-Terpineol | 1174 | 1687 | 2.3 | - |
| **16** | **Estragole** | **1176** | **1653** | **1.3** | **7.8** |
| **17** | **p-Propylanisole** | **1183** | **1644** | **-** | **9.6** |
| **18** | Ethyl octanoate | 1180 | 1426 | 0.3 | - |
| **19** | Cuminaldehyde | 1227 | 1762 | 2.0 | - |

| | | | | | |
|---|---|---|---|---|---|
| **20** | **Pulegone** | **1213** | **1630** | **-** | **12.4** |
| **21** | p-Anisaldehyde | 1220 | 2054 | 2.0 | - |
| **22** | Perillaldehyde | 1257 | 1760 | 1.5 | - |
| **23** | **Z-Anethole** | **1228** | **-** | **-** | **1.3** |
| **24** | **E-Anethole** | **1277** | **1807** | **10.1** | **7.8** |
| **25** | **Thymol** | **1269** | **2158** | **1.8** | **14** |
| **26** | 2-Undecanone | 1274 | 1590 | 1.2 | 0.3 |
| **27** | **Carvacrol** | **1277** | **2193** | **13.9** | **3.1** |
| **28** | 2-Undecanol | 1293 | 1693 | - | 0.7 |
| **29** | Piperitenone | 1310 | 1894 | - | 2.6 |
| **30** | α-Cubebene | 1348 | 1452 | - | 0.2 |
| **31** | α-Terpinyl acetate | 1331 | 1656 | 0.8 | 0.6 |
| **32** | p-Acetonylanisole | 1343 | 2100 | 1.3 | - |
| **33** | Acid Decanoic | 1353 | 1474 | 1.0 | - |
| **34** | Methyl Eugenol | 1366 | 1994 | 0.6 | - |
| **35** | Ethyl decanoate | 1382 | 1629 | 2.5 | - |
| **36** | α-Copaene | 1373 | 1484 | - | 0.8 |
| **37** | E-Caryophyllene | 1416 | 1588 | 0.6 | 1.1 |
| **38** | E-α-Bergamotene | 1430 | 1580 | - | 0.4 |
| **39** | Dill apiol | 1591 | 2350 | 0.6 | - |
| **40** | α-Curcumene | 1468 | 1757 | - | 1.3 |
| **41** | **6-Oxo-cyclonerolidol** | **1558** | **1936** | **-** | **8.4** |
| **42** | 6-Hydroxycyclonerolidol | 1627 | 2204 | - | 0.7 |
| **43** | t-Cadinol | 1623 | 2149 | 0.8 | 0.2 |
| **44** | β-Eudesmol | 1634 | 2243 | 0.7 | - |
| **45** | **Ar-Turmerone** | **1634** | **2229** | **9.3** | **0.5** |
| **46** | **(E)-heptadeca-10.16-dien-7-one** | **1825** | **2213** | **6.4** | **-** |
| **47** | Ethyl hexadecanoate | 1977 | 2230 | 1.1 | - |
| | | | **Total** | **87.0** | **78.7** |

[a] The numbering refers to elution order on apolar column (Rtx-1);

[b] **RI *a*** = retention indices measured on the apolar column (Rtx-1);

[c] **RI *p*** = retention indices measured on the polar column (Rtx-Wax) ;

[d] Relative percentages of components (%) are calculated on GC peak areas on the apolar column (Rtx-1) except for components with identical Ria* (concentration are given on the polar column).

[e] Relative percentages of components obtained by GC-FID (on RTX-1: apolar column) with peak-area normalization under optimized HS-SPME parameters: temperature: 70°C; equilibrium time: 90 min; extraction time:30 min

The EO was characterized by a large amount of monoterpenic fraction with 49.2% of the total oil, which the oxygenated monoterpenes (12 compounds) account 31.9% and monoterpene hydrocarbons (6 compounds) were scarcely represented (17.3%). This fraction was highly dominated by Carvacrol (**27**) and α-Pinene (**1**) accounting for 13.9 and 10.3%, respectively. However, the content of sesquiterpenic

fraction represent 11.4% of the total oil content, mostly attributed to 3 oxygenated sesquiterpene (**43-45**) with a percentage of 10.8% and one hydrocarbon sesquiterpene (**37**) represent only 0.6%. This fraction was characterized by a large amount of Ar-Turmerone (**45**) with 9.3% of the total oil. Also, it should be noted that this EO was characterized by the presence of 5 phenylpropanoids (**16, 24, 32, 34, 39**), representing 13.9% and 7 non-terpenic oxygenated compounds (**9**, **18**, **31**, **33**, **35, 46**, **47**) with a percentage 12.5% of the total oil. The chemical structures of these major compounds are presented in Figure 2.

These results disagreed with the findings of Makhloufi et al. who found β-ocimene-(Z), myrcene and α-pinene were the major components of essential oil of *M. pubescens*, collected from Bechar region (Algeria) [20] and while study by Tadrent et al. showed that the essentials of *M. pubescens* collected from the Ghardaia region(Algeria) shows the presence of geranyl isovalerate as major components [21].

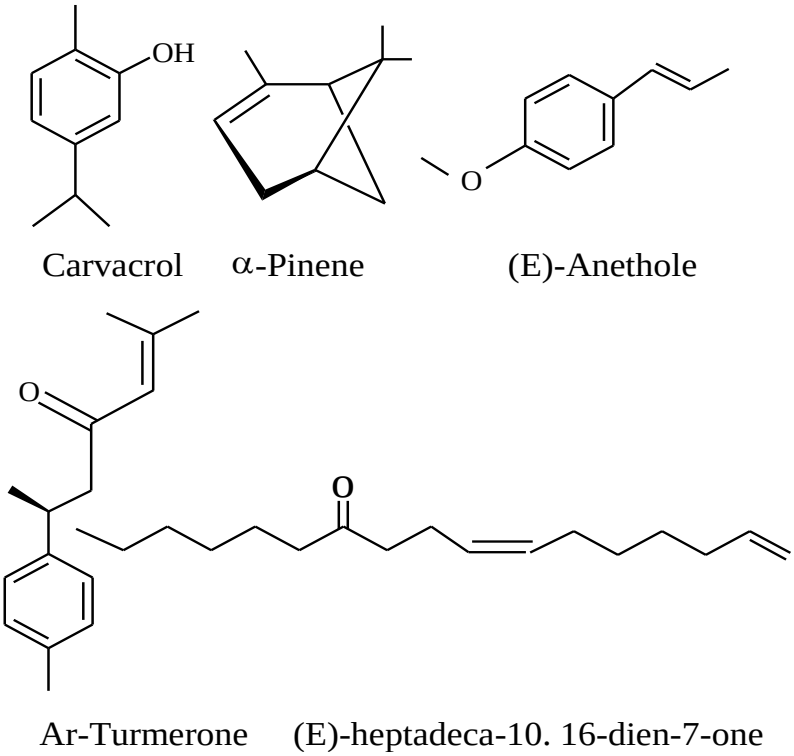


**Figure 2.** Chemical molecular structure of three major constituents of *A. pubescens subsp. pubescens* EO.

*3.2. Volatile fraction analysis*

The optimization of the HS-SPME sampling parameters was carried out using the aerial parts of *APP* and was based on the sum of the total peak areas obtained by GC-FID. the maximum sum of the total peak area was obtained at a temperature of 70 °C, an equilibrium time of 60 min, and an extraction time of 30 min. the sum of the total peak area increased according to the increase in the temperature until 70 °C. The GC-RI and GC-MS analysis allowed the identification of twenty-eight components in VF, representing 78.7% of the total composition (Table 1 and Figure 3).

The chemical composition of the VF was strongly dominated by oxygenated compounds representing 74.2% of the total VF composition, including 11 Oxygenated monoterpenes (**6**, **8**, **10**, **11**, **12**, **20**, **25**, **26**, **27, 29**) with a percentage of 37.3%, which Thymol (**25**) and Pulegone (**20**) were the major constituents with 14 and 12.4%, respectively, followed by 4 phenylpropanoids (**16**, **17**, **23, 24**) accounting 26.5%, which p-Propylanisole (**17**), (E)-Anethol (**24**) and Estragol (**16**) were the main components with 9.6, 7.8,

and 7.8%, respectively followed by 4 oxygenated sesquiterpenes (**41**, **42**, **43, 45**) which represent 9.8% predominately by 6-Oxo-cyclonerolidol (**41**) with 8.4%. However, the hydrocarbon fraction appeared in a small proportion (4.5%), comprising 5 sesquiterpenes hydrocarbons (**30**, **36**, **37**, **38, 40**) and 3 monoterpene hydrocarbons (**1**, **2, 4**).

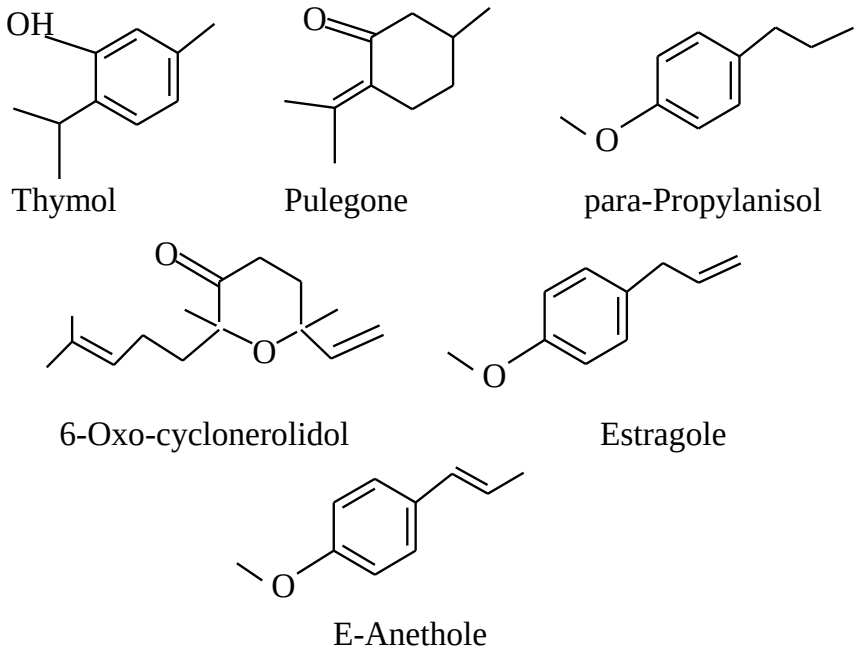


**Figure 3.** Chemical molecular structure of the major constituents of *A. pubescens subsp*. pubescens VF.

*3.3. Comparison of HD and HS-SPME methods*

In this study, dramatic qualitative and quantitative differences have been found in the composition of EO and VF obtained by HD and HS-SPME methods, respectively. Indeed, qualitatively, the total numbers of components in the case of HD were found to be more than that of HS-SPME. GC-FID and GC-MS analysis report of EO reveals 34 constituents, while as the HS-SPME extract reveals 28 constituents. Obviously, it should be noted that among the 28 compounds previously detected in the HS-SPME, only 15 of them were present in the EO. Conversely, among 34 identified compounds in the EO, only 14 of them were detected in the HS-SPME. Quantitatively, the data in Table 2 revealed that higher amounts of oxygenated monoterpenes are found in HS-SPME (37.3%) as compared to the EO obtained by HD (31.9%) while, phenylpropanoids were detected in much higher concentrations in the HS-SPME as compared to the HD (26.5% vs 13.9%, respectively), on the other hand, the non-terpenic oxygenated compounds contents of the FV (0.6%) were much lower than those of the EO (12.5%). Besides, the concentrations of oxygenated sesquiterpenes are quasi-similar in both samples (9.8% ((HS-SPME)) vs 10.8% (HD).

Besides, as shown in Table 1, the amounts of the main compounds differed between these two extraction methods. Indeed, Carvacrol (13.9%), α-Pinene (10.3 %) and E-Anethole (10.1%) were identified as the major compounds in HD, but were found in lower amounts in comparison to the HS-SPME extract at 3.1, 0.3 and 7.8 %, respectively. Also, (E)-heptadeca-10.16-dien-7-one (6.4%) wish identified in EO is not detected in HS-SPME. Conversely, Thymol (14%), Pulegone (12.4%), para-Propylanisol (9.6%), 6-Oxo-cyclonerolidol (8.4%) and Estragole (7.8%) were identified as the predominant compounds in VF, but except Thymol (1.8%) and Estragole (1.3%) identified at small content, Pulegone, para-Propylanisol and 6-Oxo-cyclonerolidol are absent in EO.

**Table 2.** Compound class distribution in the HS-SPME and HD of *A. pubescens subsp*. Pubescens aerial parts

| Compound class | HD | HS-SPME |
|---|---|---|
| **Monoterpene Hydrocarbons** | 17.3 | 0.7 |
| **Oxygenated monoterpenes** | 31.9 | 37.3 |
| **Sesquiterpene Hydrocarbons** | 0.6 | 3.8 |
| **Oxygenated sesquiterpenes** | 10.8 | 9.8 |
| **Phenylpropanoids** | 13.9 | 26.5 |
| **Oxygenated non-terpenic compounds** | 12.5 | 0.6 |

In general, it was difficult to establish a direct correlation between the chemical compositions of HD and HS-SPME techniques since the HD extraction technique is based on the liquid quasi-total extraction of plant volatiles and the HS-SPME technique is controlled by a solid/gas equilibrium step [22]. Thus, these differences were probably due to the solubilization and affinity of volatile compounds to the water and the fiber. Indeed, with HS-SPME extraction at 70 °C for 30 min, it is the fiber affinity of each compound that monitors the sampling of the volatiles limiting or favoring their extraction. Normally, the quantities of low boiling and high volatility compounds could be extracted by HS-SPME [23]. However, during HD (180 min at 100 °C), the most volatile compounds and water-soluble compounds are lost in the gaseous phase and the hydrosol under the effect of heat and acid pH, respectively [22]. EO with high solubility in water and susceptible to decomposition under temperature cannot be distilled. There is direct evidence of loss of some major and pharmacologically important minor chemical constituents from the hydrodistilled EO when compared to HS-SPME. In the same way, the amount of plant material used for sample preparation might probably be one of the major reasons which explain the difference between chemical HS-SPME and HD data. Indeed, the amount of plant material used for the HS-SPME analysis was smaller (3.5 g), while the production of hydrodistilled EO needed the use of 100 g of plant material (Table 3). HS-SPME analysis allowed a qualitative estimate of volatile compounds using a small quantity of material [24-29].

**Table 3.** Comparison of HS-SPME and HD for separation of the volatile components of *A. pubescens* subsp. *pubescens* aerial parts

| Characteristic | HD | HS-SPME |
|---|---|---|
| **Amount of sample required (g)** | 3.5 | 100 |
| **Extraction time (min)** | 30 | 180 |
| **Extraction temperature (°C)** | 70 | ~ 100 |
| **Separation time by GC-MS (min)** | 5 | 35 |
| **Major compound identified** | Carvacrol (13.9%) | Thymol (14%) |
| **Total number of components identified** | 34(87%) | 28(78.7%) |

## Conclusion

The chemical composition of HS-SPME extract obtained from the aerial parts of *A. pubescens subsp*. *pubescens* was compared with the composition of EO obtained by HD of the same plant. GC-RI and GC-MS analysis of the EO revealed thirty-four volatile compounds identified in hydrodistilled essential oil (HD), representing 87% of the total oil, while HS-SPME revealed, twenty-eight components constituting 78.7% of the volatile material. Carvacrol (13.9%), α-Pinene (10.3 %), E-Anethole (10.1%), Ar-Turmerone (9.3%) and (E)-heptadeca-10.16-dien-7-one (6.4%) were identified as the major compounds in HD. Whereas, Thymol (14%), Pulegone (12.4%), para-Propylanisol (9.8.4%), 6-Oxo-cyclonerolidol (8.4%), Estragole (7.8%) and E-Anethole (7.8%) could be identified as the predominant volatile compounds. According to our study, the chromatographic profiles obtained indicate that significant quantitative and qualitative differences between the chemical compositions of both analyzed samples were observed. Then, HS-SPME has been proved to be a complementary tool analytical tool with the hydrodistillation method for the investigation of volatile compounds from aromatic plants.

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