

# SUPERCRITICAL CO<sub>2</sub> EXTRACTION OF TURKISH MOUNTAIN TEA (*Sideritis arguta* Boiss.et Heldr.)

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## ABSTRACT

*Sideritis arguta* is widespread over in Turkey. The essential oil of *Sideritis arguta* are used for flavoring of foods and in the pharmaceutical, cosmetic and perfumery industries and in folk medicine.

The use of supercritical fluids for extraction can be considered one of the most potentially useful new methods in isolation of volatile compounds.

The supercritical carbondioxide extraction (SCCO<sub>2</sub>) of aroma compounds from *Sideritis arguta* was carried out by homemade apparatus having 2.5 l inner volume for 350 g sample. The extraction time was 120 minutes and dry matter of the sample was 7.28 %. Extraction was conducted at temperature of 34 C and pressure of 110 kg/cm<sup>2</sup> by homemade apparatus using 6 kg CO<sub>2</sub>. Supercritical CO<sub>2</sub> extract of sample was collected in a glass jar in water bath. The extraction yield was found 0.43 %. The extract was analyzed by gas chromatography (GC) and gas chromatography–mass spectrometry (GC-MS).

Some identified compounds are sabinene, beta-pinene, carvacrol, alpha-humulene, germacrene-D, ar-curcumene, zingiberene, alpha-bisabolol, beta-caryophyllene, caryophyllene oxide, palmitic acid, 9-octadecen-1-ol, 9,12,15-octadecatrienoic acid methyl ester, n-eicosane, 1,1'-binaphthalene, n-docosane.

*Keywords* : *Sideritis sp*; Supercritical CO<sub>2</sub>; Aroma extract;

## INTRODUCTION

*Sideritis* species are a group of plants known as “mountain tea” in Turkey. Local names are “dag çayı” or “yayla çayı” in Turkish. *Sideritis sp* is widespread over Turkey. The essential oil of *Sideritis sp* can be interesting for food flavoring and in the pharmaceutical, cosmetic and perfumery industries. Infusion of aerial parts of a number of *Sideritis* species are used as tonics, carminatives, as anti-inflammatory agents, antispasmodics, diuretics, and digestives, and in the treatment of colds [1].

Supercritical fluid extraction (SFE), mainly using carbon dioxide is the most popular process to extract volatile compounds from natural products. Carbon dioxide is the preferred solvent in the food industry because it is nontoxic, non-corrosive, low-cost, non-flammable, readily available and has low critical temperature and pressure. There is considerable interest in replacing steam distillation and solvent extraction processes traditionally used to obtain aromatic extracts, thereby avoiding thermal degradation of aroma compounds and the presence of solvent residues in the extracts. The extraction of essential oils and oleoresins by SFE may provide higher quality products [2].

Many studies involving the extraction of natural aroma compounds by this procedure are reported.

The composition of the essential oils of several species of *Sideritis* grown in Turkey have been studied [3-18].

The essential oil components of aerial parts from *Sideritis bilgerana*, *Sideritis tmolea* and *Sideritis congesta* were investigated by Özcan et al. [1] using GC and GC/MS. The oil yields of dried plants obtained by hydro-distillation were 0.26, 0.33, and 0.83 (v/w), respectively. Fifty compounds representing 94.6% of *S. bilgerana* oil were identified. The main ones were  $\beta$ -pinene (51.2%) and  $\alpha$ -pinene (30.2%). Thirty-six components, representing 79.7% of the *S. congesta* oil were determined with high contents of muurol-5-en-4-a-ol (11.7%) and muurol-5-en-4-b-ol (33.0%). Forty-four components were identified accounting for 89.6% of the *S. tmolea* oil. Major constituents were  $\alpha$ -cadinol (21.9%),  $\beta$ -caryophyllene (10.6%), calamenene (7.05%), muurol-5-en-4-b-ol (7.05%) and  $\alpha$ -pinene (5.1%). All oils consist of monoterpene hydrocarbons, oxygenated monoterpenes and sesquiterpenes. Muurol-5-en-4-a-ol and muurol-5-en-4-b-ol, at high percentages, were distinct components of *S. congesta*.

The main objective of this work was to study the composition of extract obtained by supercritical CO<sub>2</sub> extraction of *Sideritis arguta* collected in Turkey.

## I-MATERIALS AND METHODS

### 1. Plant material

*Sideritis arguta* Boiss. et Heldr. was collected from plants growing wild in the Antalya region of Southern Turkey.



Figure 1. *Sideritis arguta* Boiss. et Heldr.

### 2. Carbon dioxide

The carbon dioxide was purchased from Habas in Izmir.

### 3. Supercritical CO<sub>2</sub> extraction apparatus



**Figure 2. Home-made supercritical CO<sub>2</sub> extraction apparatus**

#### **4.Extraction**

Air-dried aerial parts of the plant was subjected to extraction by SCCO<sub>2</sub>. The supercritical carbon dioxide extraction (SCCO<sub>2</sub>) of aroma compounds from *Sideritis arguta* was carried out by homemade apparatus having 2.5 l inner volume for 350 g sample (**Figure 2**). The extraction time was 120 minutes and dry matter of the sample was 7.28 %. Extraction was conducted at temperature of 34 C and pressure of 110 kg/cm<sup>2</sup> by homemade apparatus using 6 kg CO<sub>2</sub>. Supercritical CO<sub>2</sub> extract of sample was collected in a glass jar in water bath. The extraction yield was found 0.43 %. The extract was dissolved with methylene chloride.

#### **5.GC and GC-MS analyses**

The extract was analyzed by gas chromatography (GC) and gas chromatography–mass spectrometry (GC-MS).

##### *Gas Chromatography:*

GC analysis was performed on a Hewlett-Packard 5890 gas chromatograph equipped with a FID and a HP-Ultra 2 column (cross-linked 5% diphenyl, 95% dimethylsiloxane, 25 m, 0.32 mm i.d., film thickness 0.52 µm). The oven temperature was held at 50 C, then programmed at 3 C/min. to 250 C; and then held at 250 for 40 min. Injector and detector temperatures were 250 C. Carrier gas, H<sub>2</sub> was adjusted to a flow of 1 ml/min. The samples were injected using the split mode (split ratio 1:50) the injection volume being 1 µl.

##### *Gas chromatography–mass spectrometry:*

The GC–MS unit consisted of a Hewlett-Packard 5973 mass selective detector operating in the electron impact mode (70 eV) coupled to a Hewlett-Packard 6890 gas chromatograph. A HP-5MS capillary column (30 m, 0.25 mm i.d., film thickness 0.25 µm) was used. The oven temperature was programmed at 50-250 C at 3 C/min. and then held at 250 C for 40 min. Injector temperature was 250 C. Sample was injected using the split mode (split ratio 1:100). The injection volume was 1 µl. Carrier gas, He, was adjusted to a linear velocity of 1 ml/min.

The quantification of the components was made on the basis of their GC/MS peak areas.

## RESULTS AND DISCUSSION

### 1.Extraction yield

Supercritical CO<sub>2</sub> extract of sample was collected in a glass jar in water bath. Dry matter of the sample was 7.28 %. The extraction yield was found 0.43 %. The extract was dissolved with methylene chloride and injected into GC and GC/MS.

### 2.Chromatograms

Figure 3 and 4 show the gas chromatogram and the total ion chromatogram (TIC) of the extract obtained using SCCO<sub>2</sub>. Volatile compounds of SCCO<sub>2</sub> extract of *Sideritis arguta* identified from TIC was shown in Table 1.

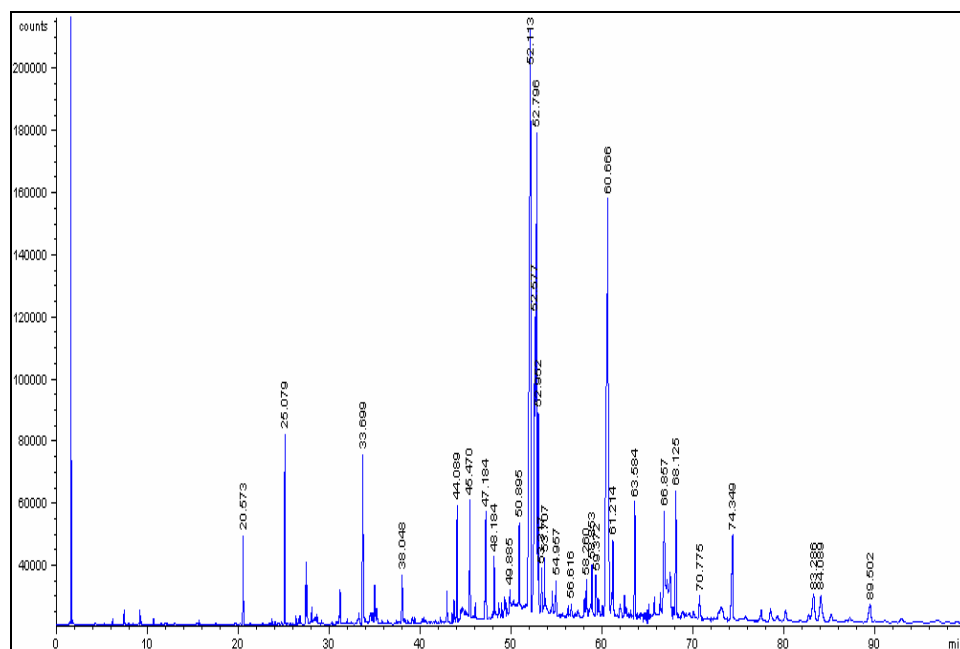
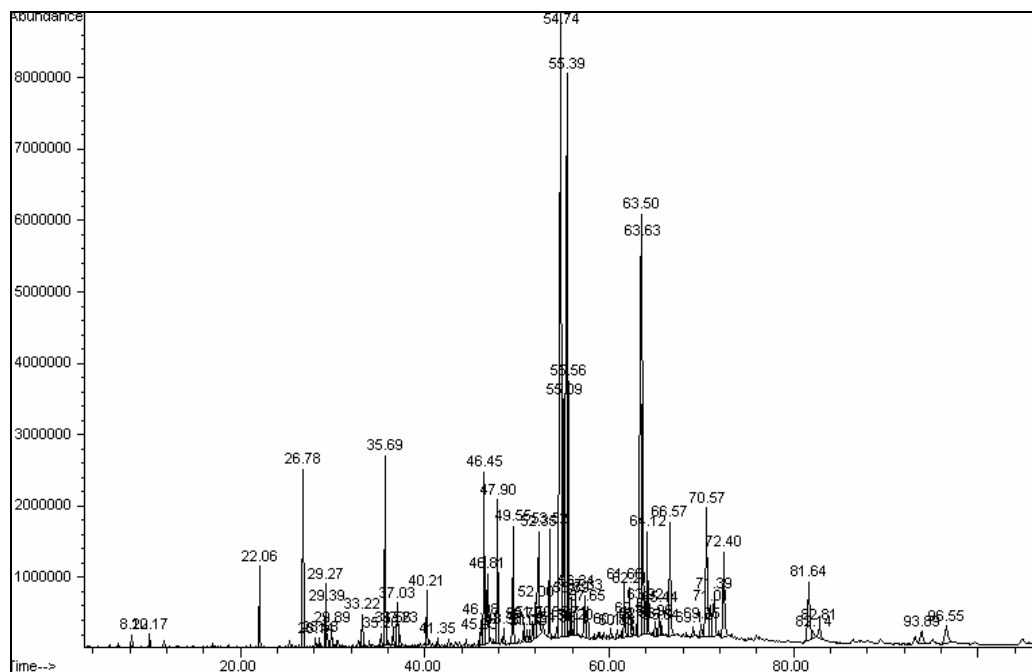


Figure 3. Gas chromatogram of SCCO<sub>2</sub> extract of *Sideritis arguta*



**Figure 4. Total Ion Chromatogram of SCCO<sub>2</sub> extract of *Sideritis arguta***

**Table 1. Volatile compounds of SCCO<sub>2</sub> extract of *Sideritis arguta* (from TIC)**

RT (min.)	Area %	Compound
8.22	0.08	β-pinene
10.16	0.14	Sabinene
22.06	0.82	Carvacrol
26.78	1.81	β-caryophyllene
28.14	0.08	α-humulene
29.27	0.61	Germacrene-D
29.39	0.38	Ar-curcumene
29.89	0.23	Zingiberene
33.22	0.39	Caryophyllene oxide
37.03	0.56	α-bisabolol
46.81	1.88	Palmitic acid
49.55	1.61	9-octadecen-1-ol
52.36	3.23	9,12,15-octadecatrienoic acid methyl ester
71.01	1.42	1,1'-binaphthalene
72.40	2.08	Eicosane
96.55	0.94	n-docosane

## CONCLUSION

In this work, the extract was obtained by supercritical CO<sub>2</sub> extraction of *Sideritis arguta* collected in Turkey and was studied its composition and 16 compounds were identified.

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