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Chemical Composition of Two *Inula sp.* (Asteraceae) Species from Turkey

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ABSTRACT: The essential oil components of aerial parts of *Inula graveolens* Desf. and *Inula oculus-christi* L. was investigated by HS-SPME / GC-MS. Thirty six and thirty seven components were identified representing 96.9%and 97.4%oil, respectively. 1,8-cineole (22.4%), borneol (20.4%) and α-cadinol (11.8%) were detected main compounds of *I. graveolens*. Bornyl acetate (21.3%) *p*-cymene (16.6%) and β-pinene (14.8%) were found major components of *I. oculus-christi.* The results were discussed in terms of natural products, renewable resources and chemotaxonomy.

Key Words: *Inula*, essential oil, 1,8-cineole, borneol

Türkiye'de bulunan iki *Inula sp.* (Asteraceae) Türünün Kimyasal Kompozisyonu

ÖZET: *Inula graveolens* Desf. ve *Inula oculus-christi* L. topraküstü kısımlarının uçucu yağ içerikleri HS-SPME / GC-MS ile araştırıldı. *Inula graveolens* ve *Inula oculus-christi* türlerine ait %96.9 ve %97.4' lük toplam yağ miktarından sırasıyla otuzaltı ve otuzyedi bileşen tespit edildi. *I. graveolens*' in ana bileşenleri 1,8-sineol (%22.4), borneol (%20.4) ve α-kadinol (%11.8) olarak tespit edildi. *I. oculus-christi*' nin ana bileşenleri bornil asetat (%21.3) *p*-simen (%16.6) ve β-pinen (%14.8) olarak bulundu. Sonuçlar doğal ürünler, yenilenebilir kaynaklar ve kemotaksonomi açısından tartışıldı.

Anahtar Kelimeler: *Inula*, uçucu yağ, 1,8-sineol, borneol.

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INTRODUCTION

Asteraceae (Compositae) is one of the largest plant family and many genera and species have worldwide distribution comprising many useful plants, so it has been the subject of chemotaxonomical studies (Bremer, 2004) and comprising about 25.000 taxa in the world. Furthermore some Asteraceae taxa has been studied from the botanical (Bremer, 2004) and chemical (Kılıç and Bağcı, 2013; Kılıç, 2013; Kılıç and Bağcı, 2012; Kılıç et al., 2011; Bağcı and Kılıç, 2012) stand points.

The genus *Inula* L., is in the Asteraceae family, mostly perennial herbs or shrubs distributed in Turkey and comprises about 32 taxa (Davis, 1975; Ozhatay and Kultur, 2006). Some *Inula* taxa are used as traditional herbal medicines throughout the world. The roots of *I. hupehensis* Y.Ling have been used to treat many diseases, including bronchitis, diabetes and intestinal ulcers. The characteristic compounds of the *Inula* taxa are sesquiterpenes and monoterpenes (Bokadia et al., 2005). Further phytochemical studies of *Inula* sp. are needed to support classification efforts, which nowadays are based mainly on morphological traits. In some *Inula* species, such as *I. britannica* L., *I. salicina* L., *I. bifrons* L., *I. conyza* DC. and *I. spiraeifolia* L. thymol derivatives are the major root constituents of cited species (Bohlmann et al., 1978). Recently, much attention has been paid to thymol derivatives, because of their diverse biological and antibacterial activities (Stojakowska et al., 2005). The usefulness of thymol derivatives as insecticides and transdermal drug delivery enhancers has also been reported (Wagner et al., 2004).

The essential oil of *Inula* is known as the most effective oil for loosening, deep congestion, coughs, colds, sinusitis larygitis and bronchitis; moreover it suppots lymphatic circulation as well as the immune system, it also reduces acneic skin inflammation (Oshadi, 2005). Besides several *Inula* species have been used in various treatments: for calculus, for eye, as diuretic, position and sudorific. The species of *Inula* genus are lack detailed phytochemical investigation. Several studies on the chemical composition of the essential oil of *Inula* species have been reported; *I. viscosa* (L.) Aiton. from Turkey (Perez-Alonso et al., 1998), *I. viscosa* (L.) Aiton from Italian (De laurentis et al., 2002) and *I. macrocephala* Boiss. & Kotschy ex Boiss. from Turkey (Kılıç and Bağcı, 2013).

The aim of present study is to examine the chemical composition of the essential oils isolated from aerial parts of *I. graveolens* and *I. oculus-christi* from eastern part of Turkey by HS-SPME / GC-MS; and to evaluate the chemical data that might be helpful in potential usefulness and chemotaxonomy of these plants.

MATERIALS AND METHODS

Plant materials

Inula graveolens was collected from Bingol University Campus, Bingol / Turkey, on 23.09.2012, at an altidude of 1150-1200 m., by Omer Kılıç (4816). *I. oculus-christi* was collected from vicinity of Dikme village, Bingol / Turkey, on 24.06.2013, at an altidude of 1600-1650 m., by Omer Kılıç (5318). *I. graveolens* and *I. oculus-christi* General view of *I. graveolens* and *I. oculus-christi*are seen in Figure 1.

Inula graveolens

Inula oculus-christi

Figure I. General view of herbarium samples of *Inula graveolens* and *Inula oculus-christi*

HS-SPME Procedure

The extraction of dried aerial part powder of five grams plant samples were carried out by a (HS-SPME) head space solid phase microextraction method using a divinyl benzene / carboxen / polydimethylsiloxane fiber, with 50/30 lm film thickness; before the analysis the fiber was conditioned in the injection port of the gas chromatography (GC) as indicated by the manufacturer. For each sample, 5 g of plant samples, previously homogenized, were weighed into a 40 ml vial; the vial was equipped with a ‘‘mininert’’ valve. The vial was kept at 35°C with continuous internal stirring and the sample was left to equilibratefor 30 min; then, the SPME fiber was exposed for 40 min to the headspace while maintaining the sample at 35°C. After sampling, the SPME fiber was introduced into the GC injector, and was left for 3 min to allow the analytes thermal desorption. In order to optimize the technique, the effects of various parameters, such as sample volume, sample headspace volume, sample heating temperature and extraction time were studied on the extraction efficiency as previously reported by Verzera et al., (2004).

GC-MS Analysis

A Varian 3800 gas chromatograph directly inter faced with a Varian 2000 ion trap mass spectrometer was used with injector temperature, 260°C; injection mode, splitless; column, 60 m, CP-Wax 52 CB 0.25 mm i.d., 0.25 lm film thickness. The oven temperature was programmed as follows: 45°C held for 5 min, then increased to 80°C at a rate of 10°C/min, and to 240°C. at 2°C/min. The carrier gas was helium, used at a constant pressure of 10 psi; the transfer line temperature, 250°C; the ionisation mode, electron impact (EI); acquisit ion range, 40 to 200 m/z; scan rate, 1 us-1. The compounds were identified using the NIST library, mass spectral library and verified by the retention indices which were calculated as described by Van den Dool and Kratz (1963). The relative amounts were calculated on the basis of peak-area ratios. The identified constituents of *Inula* species are listed in Table-1.

RESULTS AND DISCUSSION

The essential oil components of aerial parts of *Inula graveolens* and *Inula oculus-christi* was investigated by HS-SPME / GC-MS. Thirty six and thirty seven components were identified representing 96.9%and 97.4%oil, respectively. 1,8-cineole (22.4%), borneol (20.4%) and α-cadinol (11.8%) were determined main compounds of *I. graveolens*. Bornyl acetate (21.3%) *p*-cymene (16.6%) and β-pinene (14.8%) were detected main constituents of *I. oculus-christi* (Table-1).

RRI*: Relative Retention Index.

The most important compounds leaves-stems-flowers and roots of *I. graveolens* from Tunisia, without flowers were τ-cadinol (9.2%), borneol (21.4%), bornyl acetate (33.4%); in the flowers oil, the main components found were camphene (5.5%), τ-cadinol (11.3%), borneol (19.3%), bornyl acetate (39.6%); the major constituents in the oil roots were found to be carvone (5.0%), bornyl acetate (5.3%), p-mentha-1(7)-2-dien-8-ol (5.3%), β-selinene (11.5%) (Harzallah-Skhiri et al., 2005). The main constituents of *Inula graveolens* (L.) Desf. from Corsica were bornyl acetate (56.8%), borneol (7.6%) and *τ*-cadinol (7.8%); bornyl acetate and borneol were always two major compounds of six different stages of *I. graveolens*; furthermore to determine the chemical variability of *I. graveolens*, 22 samples were analysed, obtained from several plants collected in a restricted area in different localities during the flowering stage. Bornyl acetate (43.1%–73.1%) and borneol (3.7%-32.2%) were the main compounds for 20 samples. The twenty-first sample exhibited borneol (41.9%) and bornyl acetate (16.6%) as the major components, while the composition of twenty-second was typical (borneol 41.9% and *τ*-cadinol 23.8%) (Blanc et al., 2004). Like these results bornyl acetate (8.9%- 21.3%) was detected the main compounds of *I. macrocephala* (Kılıç and Bağcı, 2013) and in this study with *I. oculus-christi* (Table-1) respectively. Borneol (20.4%) was detected the main compounds *I. graveolens* (Table-1). Borneol (3.72%- 3.26%) was also among the main components of *Anthemis pseudocotula* and *A. cretica* subsp. *pontica* (Asteraceae) from Turkey (Kılıç et al., 2011). *p*β-pinene (14.8%) *I. oculus-christi*; 1,8-cineole (22.4%) and α-cadinol (11.8%) in *I. graveolens* were detected as major components (Table-1).

The major components of *I. crithmoides* L. from central Italy were; *p*-cymene (30.1%), 1-methylethyl-trimethylbenzene (18.7%), scopoletin (15.3%) and α-pinene (13.1%) (Laura et al., 2010). Like this study, *p*-cymene (10.2%- 16.6%) was detected one of the major component of *I. macrocephala* (Kılıç and Bağcı, 2013) and *I. oculus-christi* (Table-1). Among the sesquiterpenes, β-caryophyllene (15.3%) was determined as one of the major constituents of *I. macrocephala* (Kılıç and Bağcı, 2013); it is noteworthy that, β-caryophyllene (1.9%- 0,6%) was reported low amounts in this study with *I. graveolens* and *I. oculus-*

Table I. Chemical profiles of *Inula* species (%)

Constituents	RRI*	*I. graveolens*	*I. oculus-christi*
2-Hexenal	855	0.4	0.5
α-pinene	935	2.3	1.8
Benzaldehyde	962	-	0.3
Sabinene	977	1.2	-
β-pinene	982	3.6	**14.8**
Myrcene	994	-	0.4
Santolinatriene	1001	0.2	-
α-phellandrene	1006	0.4	0.3
α-thujene	1015	0.3	0.5
p-cymene	1024	3.5	**16.6**
Limonene	1031	0.5	1.2
Camphene	1034	4.6	2.1
1,8-cineole	1035	**22.4**	5.3
γ-terpinene	1055	0.6	0.9
Benzene, 1- methyl - 2	1069	0.2	-
Terpinolene	1085	-	1.2
Linalool	1101	0.3	-
Trans-pinocarveole	1165	-	0.7
α-terpineol	1180	1.5	2.3
Camphor	1190	0.4	0.2
2-Cyclohexan-1-ol	1205	-	0.1
Thymol methyl oxide	1230	0.4	0.2
Thymol	1290	1.8	5.2
α-cubebene	1350	-	0.2
β-elemene	1360	0.6	0.3
α-copaene	1375	0.4	-
β-bourbonene	1385	-	0.1
β-cubebene	1390	0.1	-
β-caryophyllene	1418	1.9	0.6
α-humulene	1455	-	1.1
δ-cadinene	1460	0.6	-
γ-muurolene	1475	1.3	2.2
Bornylacetate	1478	5.3	**21.3**
Germacrene D	1480	-	0.9
β-selinene	1488	0.9	-
Bicyclogermacrene	1496	-	0.2
β-caryophyllene	1502	4.8	3.9
α-muurolene	1520	0.4	-
Spathulenol	1575	1.6	1.5
Borneol	1577	**20.4**	2.7
Caryophyllene oxide	1580	-	0.8
Ledol	1605	1.3	0.2
α-cadinol	1640	**11.8**	5.1
β-eudesmol	1650	0.1	-
Hexadecanoic acid	1690	-	0.5
Pentadecanal	1710	0.2	-
Nonadecane	1905	-	0.3
Tricosane	2295	-	0.6
Pentacosane	2495	0.2	0.3
Hexacosane	2600	0.4	-
	Total	**96.9**	**97.4**

christi (Table-1). Pentacosane (13.7%) was determined main constituent of the volatile oil of *I. oculus-christi* (Javidnia et al., 2006), whereas pentacosane (2.1%) was determined the minor compound of *I. macrocephala* (Kılıç and Bağcı, 2013); *I. graveolens* (0.2%) and *I. oculus-christi* (0.3%) (Table-1).

The findings showed that the genus *Inula* had a considerable variation in essential oil composition and this study demonstrates the occurrence of the 1,8-cineole / borneol / α-cadinol chemotype in *I. graveolens*; bornylacetate / *p*-cymene / β-pinene chemotype in *I. oculus-christi*.

Moreover this result is significant to chemotaxonomic evaluation and renewable resourches of the genus patterns.

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