



Essential Oil Composition of Dry and Fresh Aerial Parts of the Dill (*Anethum graveolens* L.)^A

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Abstract: The study was conducted to determine the essential oil ratio and composition of fresh and dry aerial part of dill (*Anethum graveolens* L.), under the Harran Plain conditions (South-eastern Anatolia, Turkey), with three replicates, in 2014-2015 growing session. After harvesting, the dill samples were separated into leaf, stem and umbel. The essential oil ratios were determined at each fresh and dry samples. The essential oil ratios were varied between 0.08-0.59% in fresh dill and 0.11-2.07% in dry dill. Based on the CS-MS results, essential oil components varying between 17-23 in fresh dill and 16-19 in dry dill were determined in the aerial parts of the plant. The α -phellandrene was identified as the main essential oil compound of the samples analysed except for the dry stem. In dry stem, the dill ether was determined as the main component.

Keywords: *Anethum graveolens*, Volatile oil component, Leaf, Stem, Flower.

Dereotu (*Anethum graveolens* L.)'nun Taze ve Kuru Toprak Üstü Aksamlarının Uçucu Yağ Bileşenleri

Öz: Çalışma, dereotu (*Anethum graveolens* L.) taze ve kuru toprak üstü kısımlarının uçucu yağ oranı ve bileşenlerini belirlemek amacıyla, 2014-2015 yetiştirme döneminde, Harran Üniversitesi Ziraat Fakültesi

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Araştırma Alanında, 3 tekrarlamalı olarak yürütülmüştür. Hasattan sonra, dereotu örnekleri yaprak, sap ve çiçeklere ayrılmıştır. Her taze ve kuru örnekte uçucu yağ oranları belirlenmiştir. Uçucu yağ oranları taze dereotunda % 0.08-0.59 arasında, kuru dereotunda % 0.11-2.07 arasında değişmiştir. CS-MS sonuçlarına göre, bitkinin toprak üstü kısımlarında taze dereotunda 17-23 ve kuru dereotunda 16-19 arasında değişen uçucu yağ bileşeni belirlenmiştir. α -Phellandrene, kuru sap haricinde, analiz edilen numunelerin ana uçucu yağ bileşiği olarak belirlenmiştir. Kuru sapta ise dill ether ana bileşen olarak saptanmıştır.

Anahtar Kelimeler: *Anethum graveolens*, Uçucu yağ bileşenleri, Yaprak, Sap, Çiçek.

Introduction

In recent years, the importance and usage areas of essential oils have been increasing. Plants containing essential oils and essential oils are used in many fields such as in foods, cosmetics, perfumes, health (human and animal) and the animal feed industry (Curabay et al., 2020; Kirici et al., 2020). One of the important plants containing the essential oil is dill. Dill (*Anethum graveolens* L.) is an annual plant that belongs to Apiaceae family and is grown for its herb and seeds in many countries. While fresh dill herbs are used as a green vegetable, its fresh and dried herbs, and seeds are consumed as a spice and the volatile oils, extracted from its herbs and seeds are used in many fields of industry, including food, cosmetics and pharmaceuticals. Some studies showed that essential oils of dill have many functional properties such as antimicrobial (Stavri and Gibbons, 2005), anti-inflammatory, analgesic (Valady et al., 2010), gastric mucosal protective (Hosseinzadeh et al., 2002), hyperlipidaemic effect (Hajhashemi and Abbasi, 2008) and so on (Amin and Sleem, 2007; Hashemzadeh et al., 2013). In addition, it has been reported that dill has been used in traditional medicine due to its carminative, gastric and diuretic activity (Amin and Sleem, 2007).

The volatile oil ratio and volatile oil composition of dill are critical importance for the functional properties of the plant. Many factors including genotype, location, ecological factors and growth techniques affect the quantity and composition of essential oils of dill (Porter et al., 1983; Wander and Bouwmeester, 1998; Salmasi et al., 2006; Callan et al., 2007; Frąszczak, 2009; Vokk et al., 2011; Hashemzadeh et al., 2013; Barandozi and Borujeni, 2014; Khamssi, 2014; Rana and Blazquez, 2014). Also, the volatile oil content and composition varies according to the aerial parts of the plant such as leaves, stems, umbels (inflorescence) and seeds (Callan et al., 2007). Therefore, the different parts (i.e. leaf, stem and umbel) density in the dill herb can affect both concentration and composition of the volatile oils of dill (Radulescu et al., 2010; Kruma et al., 2011; Vokk et al., 2011). Several studies are conducted to determine the morphogenetic and ontogenetic variabilities of volatiles in dill plants (Huopalahti and Linko, 1983; Porter et al., 1983; Radulescu et al., 2010). It has been reported that the major compounds constituting essential oils of naturally dried parts of the dill plant were α -phellandrene in the leaves, limonene in the flowers, α -phellandrene and dill ether in the stems and carvone in the seed (Callan et al., 2007; Vera and Ming, 1998; Radulescu et al., 2010; Kruma et al., 2011; Vokk et al., 2011; Rana and

Blazquez, 2014). It has also been reported that the essential oil content and the essential oil composition of dill leaves and stems varied depending on the methods of drying (Kruma et al., 2011). Since dill is consumed both as fresh and in dried forms it is important to determine the amount and compositions of volatile oils in different fragments of the dry and fresh dills. Knowing the change in the rate of essential oil and the distribution of essential oil components after drying the plant may affect dill usage preferences.

The present work was conducted to determine the amount and compositions of the essential oils of different parts of fresh or dried dill.

Material and Methods

Plant Material

The population of dill (*A. graveolens* L.) seed, obtained from local growers, was used as the plant material. The dill plants were grown under the Harran Plain conditions, in 2014-2015 growing session. The research area was in the Harran Plain (South-eastern Anatolia region) where semi-arid climate conditions. The research field soil belonged to Harran I series and it had A, B and C horizons, flat and/or flat-like slope, main material was alluvial and a deep profile. According to the soil analysis made before sowing, it was observed that the texture of the study area soil had clayey (17%), neutral pH (7.84), low salt (0.08%) and organic matter (1.37%) levels.

The dill seeds were sown on December 5, 2014 with 30 cm inter-row space and the seed amount of 10 kg ha⁻¹. At the stage of sowing, the soil was fertilized with phosphorus (50 kg ha⁻¹) and nitrogen (50 kg ha⁻¹). Then, at the beginning of May, the nitrogen (50 kg ha⁻¹) was applied as top fertilizer to all research areas. The whole dill herbs were harvested at the full flowering stage. After harvesting, dill plant samples were separated into their organs: leaves, stems, and umbels (peduncle + flowers). Each sample was divided into 2 parts; one portion of each sample was naturally dried in the shade and stored at 4°C until analysis. The other part was taken into analysis for essential oil determination as fresh.

Essential oil isolation

Each 50 g sample taken fresh and dry was hydro distilled in water for 3 hours by Clevenger type apparatus (Anonymous, 2011). Essential oil ratios were determined in triplicate in each aerial part of the plant and averaged. The extracted essential oils were stored in a dark glass bottle and kept in the freezer until analysis. Essential oil component analysis was performed in single samples combined according to aerial parts of the plant (leaf, stem and flower).

Essential oil component analysis

Dill essential oils were analysed by means of GC-MS (Agilent 7890A) equipped with an electron impact quadrupole, mass spectrometer detector (Agilent 5975C). It had 70 eV electron ionization energy, 35-450 amu

scanning range, and 1 scan second⁻¹ scan rate. HP Innowax Capillary (A fused silica capillary column 5% phenyl-poly-dimethyl-siloxane) GC column was used. It was a length of 60 m, with the film thickness of 0.25 µm, and an internal diameter of 0.25 mm. Helium was used as carrier gas at 0.8 ml min⁻¹ flow rate. The inlet temperature was set as 250°C. The temperature program of the GC oven was used as follows; initial temperature 60°C and hold for 10 min, at 4°C min⁻¹ raised to 220°C and finally held on 10 min at 220°C. The sample is diluted one percent (v/v) with n-Hexane and injected with 1 µl. The 40:1 split ratio was used.

Identification of compounds was based on Kovats retention indexes. Identity of the compounds was made by comparison of the mass spectra with data from Adams, the US National Institute of Standards and Technology (NIST, USA), and the WILEY 1996 Ed mass spectrum library.

Results and Discussion

As seen in Figure 1, the ratio of volatile oils varied between 0.08% and 0.59% in different parts of the fresh dills and, 0.11% and 2.07% in different fragments of dried dills. The essential oil contents of dry parts of the plant, as expected, were higher than that of the fresh parts of the plant. The lowest essential oil ratio was determined on both fresh and dry stem of dill plants. Also, the highest essential oil levels were recorded in the fresh and dry umbels of the dill plants. The essential oil content of the leaves was lower than the essential oil content of the herbs. It may be due to that the dill plants were harvested during flowering periods, and the herbs included the umbels which contain a very high proportion of essential oils (Figure 1). These findings were in harmony with Radulescu et al. (2010) who showed that the ratio of essential oils in the flowers highest than in the leaves.

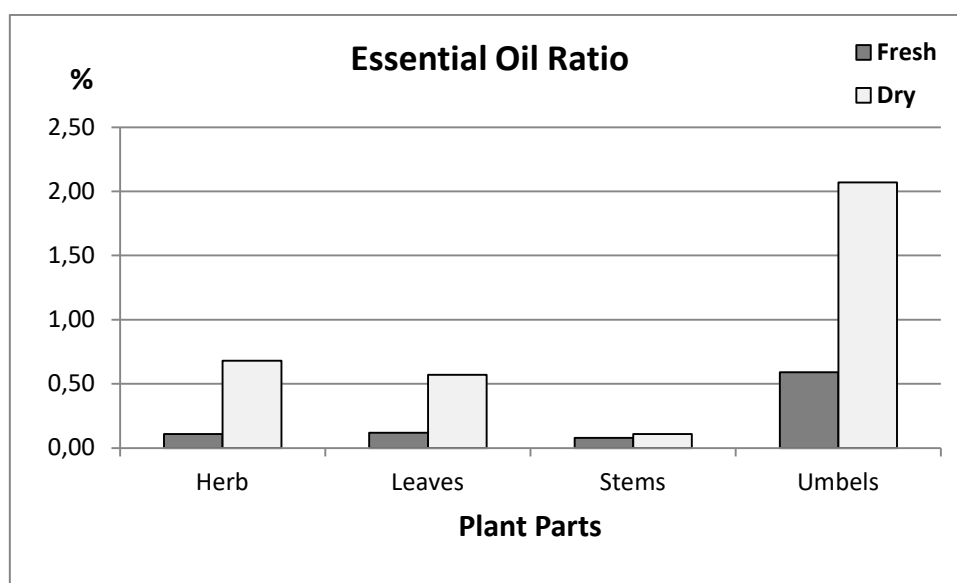


Figure 1. The essential oil ratios of different aerial parts of the fresh and dry dill plant

The composition of essential oils at different organs of fresh and dry dill plants was listed in Table 1. The number of compounds determined in different parts of fresh or dry dills was as follows: in fresh herb 20, in dry herbs 19, in fresh stem 17, in dry stem 16, in fresh and dry leaves 16, in fresh umbels 23 and in dry umbels 19 compounds. Total compounds ratio was varied between 95.64-99.98% of the total detected constituents in the fresh and dry plant parts.

Table 1. The composition of essential oils in different parts of fresh and dry dill plants.

R.T.	Compounds	Herb		Stem		Leaves		Umbels (flowers)	
		Fresh	Dry	Fresh	Dry	Fresh	Dry	Fresh	Dry
11.523	α -Pinene	1.266	0.844	1.534	0.868	1.806	1.425	1.033	0.967
11.590	α -Thujene	0.222	0.140	0.285	0.170	0.326	0.246	0.273	0.162
13.999	β -Pinene	0.073	-	-	-	0.156	-	-	-
14.333	Sabinene	0.100	0.125	0.176	-	0.167	-	0.126	-
15.495	Myrcene	0.606	0.588	0.695	0.611	0.742	0.719	0.545	0.530
15.741	α -Phellandrene	41.628	43.314	55.68	20.993	61.842	58.841	39.800	33.098
16.721	Limonene	10.701	6.277	3.469	3.959	3.405	4.092	13.378	17.052
17.055	β -Phellandrene	5.872	6.904	7.395	7.450	8.115	8.205	4.961	4.569
17.233	1,3,8-p-Menthatriene	-	-	-	-	-	-	0.087	0.128
18.905	p-Cymene	1.772	4.804	2.072	17.881	1.390	2.924	0.622	1.685
19.239	α -Terpinolene	0.150	0.185	0.421	-	0.181	-	-	-
21.542	Tyranton	0.448	-	1.838	-	-	-	1.647	0.100
25.891	Dill ether	24.147	26.637	24.643	37.64	20.694	19.626	24.235	21.418
28.200	Trans-Dihydrocarvone	0.129	-	-	-	-	-	0.105	0.530
28.464	Bicyclo[3.2.1]-3-octen-6-on,4,7-dimethyl (endo)	0.195	0.268	0.176	0.331	0.144	0.194	0.225	0.270
28.694	Cis-Dihydrocarvone	0.913	0.416	-	-	0.119	0.278	0.848	4.013
30.601	Germacrene	-	0.456	-	-	0.069	0.505	-	-
31.179	Carvone	5.919	1.289	0.228	-	-	0.502	6.098	7.214
31.849	Iso-Dihydrocarveol	-	-	-	-	-	-	-	0.150
32.411	Neoiso-Dihydrocarveol	0.452	-	-	-	-	-	0.189	0.213
32.490	Trans-Sabinol	-	0.285	-	0.539	-	0.187	-	-
32.821	p-Menth-1-en-9-yl acetate	-	0.109	-	0.481	0.491	-	0.192	0.173
34.554	Trans-Phytol acetate	-	-	-	0.251	-	-	-	-
35.601	Cis-Ocimene	-	-	-	-	-	-	0.067	-
36.162	Cinnamyl alcohol	-	-	0.086	0.231	-	-	0.099	-
39.844	p-Menth-1-en-9-ol acetate	0.780	0.427	0.193	1.905	0.161	0.547	-	-
40.306	Cinnamyl alcohol	0.174	1.016	0.474	1.727	-	0.656	0.146	0.451
40.831	p-Vinyl guaiacol	-	-	-	-	-	-	0.080	-
41.229	Elemicin	-	-	-	-	-	-	0.146	-
42.650	Myristicin	4.308	2.363	0.359	0.601	-	0.761	5.087	5.903
Total		99.86	96.44	99.72	95.64	99.81	99.71	99.98	99.51

The eight main components were identified in the essential oil obtained from different parts of the plant (fresh or dry). These are α -phellandrene, dill ether, limonene β -phellandrene, carvone, myristicin, p-cymene and α -pinene.

In all plant parts, the major essential oil compound, except dry stem was identified as α -phellandrene (Figure 2A-D). However, the dill ether was detected as the main constituent in dry stem essential oil (Table 1, Figure 2B). The ratio of macro components of essential oil was varied depending on the aerial parts of the dill plants and drying. Our findings are consistent with Saxena et al. (2018), who reported that the distribution of essential oil components varies depending on organs and drying.

In herb, along with drying of the plant material, α -phellandrene, β -phellandrene, dill ether and p-cymene ratios were increased while limonene and carvone ratios were decreased (Figure 2A).

Similar changes were observed between fresh and dry stems as well (Table 1). While α -phellandrene was the main essential oil component of the fresh stems, dill ether was found to be the dominant essential oil compound in dry stems. In dry stems, while the ratio of limonene, β -phellandrene, p-cymene and dill ether were increased, the ratio of α -phellandrene was decreased (Figure 2B). These findings were similar to Kruma et al. (2011) who reported that the major constituent of fresh stems' essential oils was the α -phellandrene while in the dry stems was the dill ether.

When the essential oil constituents of the leaves were investigated, it was found that the ratios of the α -phellandrene and dill ether were higher in the fresh leaves and decreased with drying. Limonene, β -phellandrene and p-cymene ratios were increased with drying (Figure 2C). Some researchers reported that the α -phellandrene was the main component of fresh and dry leaves essential oils and the α -phellandrene ratio of fresh leaves essential oils were varied such as 56.5% (Vera and Ming, 1998), 62.71% (Radulescu et al., 2010), 43.99% (Kruma et al., 2011) and between 47.74-62.49% (Vokk et al., 2011). Our figures for the α -phellandrene were similar to the findings of Radulescu et al. (2010).

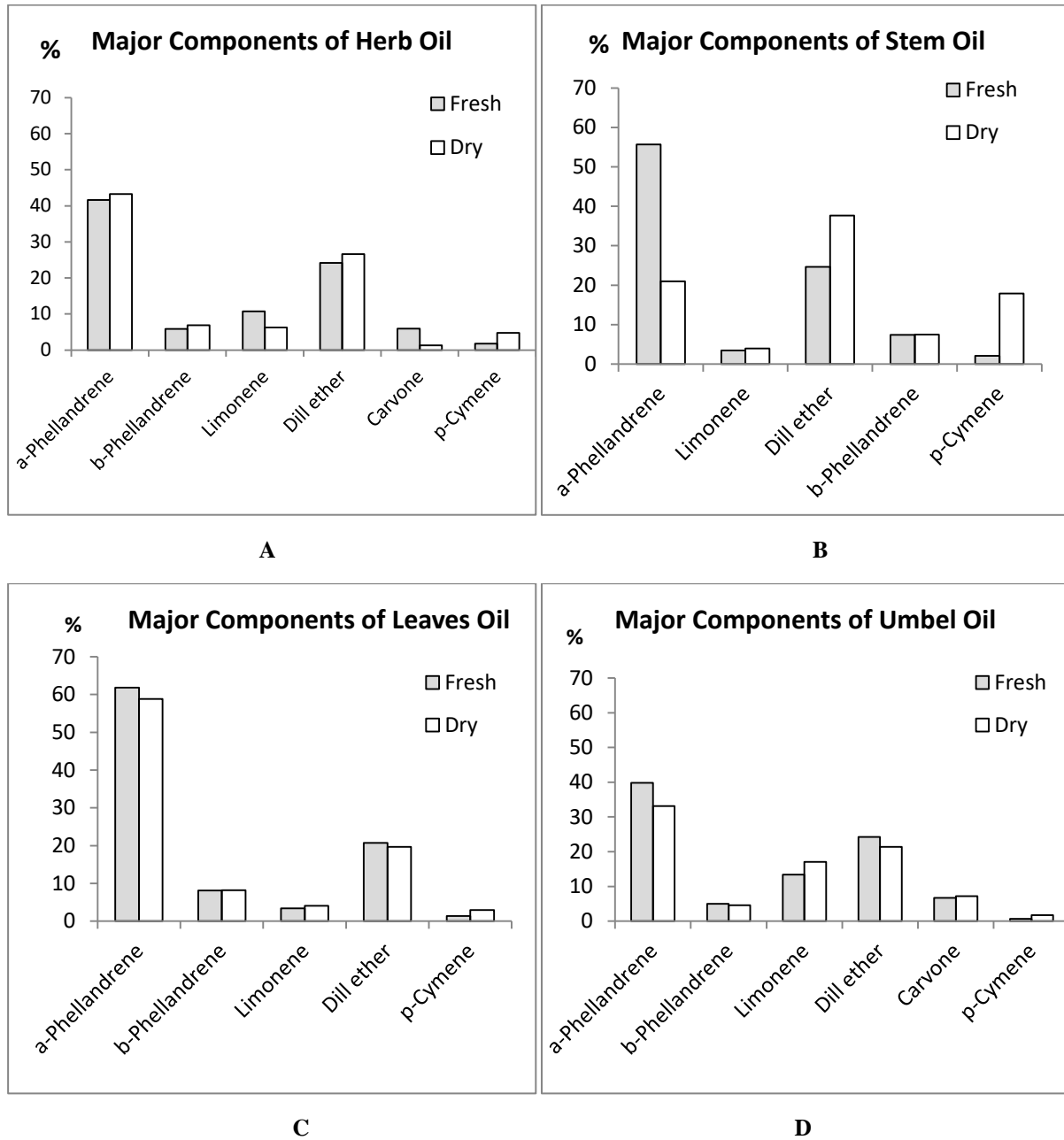


Figure 2. Major components of essential oils from different parts of the fresh and dry dill

In umbels essential oil components, α -phellandrene, β -phellandrene and dill ether ratios were higher in the fresh umbels and decreased with drying. Limonene, carvone and p-cymene ratios were increased with drying (Figure 2D). Our findings of umbels essential oil components were contradicted with Radulescu et al. (2010) who reported that the main component of dry umbels essential oils was the limonene. This may be due to differences in genotype, ecological and growing conditions.

Conclusions

The essential oil components of aerial parts of the fresh and dry dill plants varied according to the organ obtained. The α -phellandrene was determined the main essential oil component of all parts of the plant except the dry stem. In dry stem, the dill ether was found to be the predominant component. The ratio of α -phellandrene, β -phellandrene, dill ether and p-cymene increased, and limonene and carvone ratios decreased with the drying of the herbs. The ratio of limonene and p-cymene increased with drying in the leaves. The carvone was not detected in essential oils of leaves and stems. In contrast, it was detected in the herb which probably comes from the umbels. The high dill ether content in the dry herb was originated from the stem oil. The composition of the dill essential oil can be adjusted to a certain degree using plant parts, depending on the intended use.

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