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COMPOSITION OF ESSENTIAL OIL OF *CUMINUM CYMINUM* L. ACCORDING TO HARVESTING TIMES

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Abstract

The cumin (Cuminum cyminum L.) is an annual plant of Apiaceae. It is generally used as a food additive for increasing the flavour of different food preparations. The fruits (often called seed)are rich in essential oil with the main constituents at different harvesting time being cumin aldehyde (19.9–23.6%), p-mentha-1,3-dien-7-al (11.4-17.5 %) and p-mentha-1,4-dien-7-al (13.9-16.9). The results of GC and GC/MS analysis showed that the fruits should be harvested at the ripe stage for ideal volatile oil yield and composition. The effect of different harvesting times on the yield and ratio of main components of the essential oil are evaluated.

***Key Words**: Apiaceae, Cumin, Cuminum cyminum, Essential oil*

Hasat Zamanlarına Göre Kimyon Meyvelerinin Uçucu Yağ Bileşenleri

Kimyon (Cuminum cyminum L.) Apiaceae familyasına ait tek yıllık bir bitkidir. Kimyon genellikle koku ve tad düzeltici olarak yemeklerde baharat olarak kullanılır. Kimyon meyveleri(ekseriyetle tohum olarak anılır) uçucu yağ bakımından zengin olup meyvelerinin farklı hasat zamanlarına bağlı olarak ana bileşenlerinin kumin aldehit (19.9–23.6%), p-menta-1,3-dien-7-al (11.4-17.5 %) ve p-menta-1,4-dien-7-al (13.9-16.9)olduğu bulunmuştur. GC ve GC/MS analiz sonuçları, uçucu yağ verimi ve bileşenleri bakımından kimyon meyvelerinde hasadın meyvelerin tam olgun olduğu dönemde hasat edilmesi gerektiğini göstermiştir. Bu çalışmada farklı hasat zamanlarının kimyon uçucu yağ verim ve bileşenleri üzerine etkisi değerlendirilmiştir.

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INTRODUCTION

Cuminum cyminum L. is an annual plant of the family Apiaceae. The medicinal component of the plant is Cumin oil extracted from the ripe fruit. In folk medicine, cumin is used as a carminative for stomach disorders, diarrhea, and colic, as well as particularly in veterinary medicine (1). The oil of cumin is especially used as a carminative and astringent (2). It is widely cultivated in Turkey, Syria, Sudan, Iran, India, Pakistan, Argentina, China and other countries. The fruits of *C. cyminum* L. are used as a traditional flavouring in a number of ethnic cuisines and food industries. Moreover, cumin oil shows a high antifungal activity against various pathogenic fungi, and effective high antibacterial activity. Therefore it is also used as a fumigant or additive in the storage of foodstuffs (3). The composition of the essential oil of *C. cyminum* depends on many factors, such as plant part, harvest-time, extraction method, type of cultivar, geographic origin, and storage conditions (4). The fruits are known as "Kimyon" and are traded and used as a spice in Turkey. Cumin is mostly cultivated in the Central Anatolian region where it is used as condiment and as an ingredient of sucuk (a kind of spicy Turkish sausage) (5,6).

The cumin fruits contain volatile oil (2–5 %) that impart the characteristic aroma to the fruits (7). The yellow-coloured fresh oil contains cuminaldehyde and two p-menthadienals as its chief components (8). The composition of cumin oil has previously been studied by several workers. Principal constituents of the oil were found to differ in their cuminaldehyde and *p*-menthadienal contents (3,9).

When cumin fruits are harvested at different times, their physical and chemistry properties may change considerably. Accordingly it is necessary to determine optimum harvesting time for essential oil yield and composition. Object of this study was to investigate composition of cumin oil at different harvesting times.

EXPERIMENTAL

Plant Material

The cumin fruits were cultivated in the experimental farm of Selçuk University, Faculty of Agriculture in ecological conditions of Konya. Cumin fruits were harvested at three different periods (unripe, ripe and fully ripe) at 2-week intervals during the harvest season in 2005. These three materials were used in the present study.

Essential Oil Distillation

The dried and powdered samples (100 g) were subjected to hydrodistillation for 3 h using a Clevenger-type apparatus to produce essential oil. The yield of oils is increased by crushing of the fruits prior to distillation.

GC analysis

The GC analysis was carried out with a Hewlett-Packard HP6890, equipped with a HP-Innowax silica capillary column (60 m x 0.25 mm, film thickness 0.25 μm) and a flame ionization detector. Nitrogen was used as carrier gas with a flow rate of 0.8 ml/min. Injector and detector temperatures were both set at 250 ^{0}C. Column temperature was programmed to 60 ^{0}C for 10 min, gradually increased to 220 ^{0}C at 4 ^{0}C/min, held for 10 min and then increased to 240 ^{0}C at 1^{0}C/min. Split ratio was 50:1 and one microliter of sample (dissolved in hexane as 20 % v/v) was injected into the system.

Gas chromatography-mass spectrometry (GC-MS) analysis

The GC-MS analysis of the oil was carried on an Agilent 6890N Network GC system combined with Agilent 5973 Network Mass Selective Detector (GC-MS). The capillary column used was an Agilent 19091N-136 (HP Innowax Capillary; 60.0 m x 0.25 mm x 0.25 μm). Helium was used as carrier gas at a flow rate of 1.0 ml/min with 1 μl injection volume. Samples were analyzed with the column held initially 60 ºC after injection with 10 min hold time, then increased to 220 ºC with 4 ºC/min heating ramp and kept at 220 ºC for 10 min. Then final temperature was increased to 240 ºC with 1 ºC/min heating ramp. The injection was performed in split mode (50:1). Detector and injector temperatures were 230 °C and 280 °C, respectively. Run time was 80 min. MS scan range was (*m/z*): 35-450 atomic mass units (AMU) under electron impact (EI) ionization (70 eV).

Identification of the volatile oil components

The components were identified by comparing their relative retention times with those of authentic samples and mass spectra with the data from the Baser library of essential oil constituents as well as Wiley and Nist Library. Relative content of % components were determined with area under peaks using Agilent software. The results are expressed as an average of three determinations in all cases. GC and GC/MS analysis were both conducted at the Faculty of Pharmacy at Ankara University.

RESULTS AND DISCUSSION

In the course of the present study, 41 components amounting to 97.0–97.2% of the oils were identified in three samples which were harvested at different times (Table I). The volatile oils of dried cumin fruits were obtained by water distillation with yields of 1.9 % for unripe fruit, 2.4 % for ripe fruit and 2.3 % for fully ripe fruit. The major components were cumin aldehyde, *p*-mentha-1,3-dien-7-al, *p*-mentha-1,4-dien-7-al, γ-terpinene and *p*-cymene. Comparison of the cumin oils of three samples has shown that the cumin aldehyde content of the unripe fruit oil was lower than those of ripe and excessive ripe samples examined. The impact compounds of the volatile oil of cumin unripe, ripe and fully ripe fruits were found to be 14.1, 17.5 and 11.4 % for *p*-mentha-1,3-dien-7-al, 16.9, 13.9 and 16.0 % for *p*-mentha-1,4-dien-7-al, respectively. The results of our analysis are in good agreement with those reported by Baser *et al* (6,9). Harvesting at fully ripe stage caused an increase in the amount of γ-terpinene and *p*-cymene. But β-phellandrene content decreased when fruits ripened. Our results showed that observed differences in the composition of the oils besides other factors appear to be the maturity of the cumin fruit.

Table 1. Chemical composition of the essential oils of cumin fruits at different harvesting times

RRI	Compound	Unripe fruit oil content %	Ripe fruit oil content %	Excessive ripe fruit oil content %
1032	-Pinene	0.4	0.3	0.4
1035	-Thujene	0.2	0.1	0.2
1118	**-Pinene**	**8.7**	**7.4**	**9.0**
1132	Sabinene	0.4	0.3	0.1
1176	**-Phellandrene**	**8.5**	**4.1**	**5.4**
1188	-Terpinene	0.1	0.1	0.2
1203	Limonene	1.0	0.8	1.0
1213	1,8-Cineole	0.1	0.1	0.1
1218	-Phellandrene	1.2	0.8	1.0
1255	**-Terpinene**	**10.3**	**11.5**	**13.6**
1280	***p*-Cymene**	**7.7**	**9.6**	**11.6**
1290	Terpinolene	0.1	0.1	0.2
1504	Daucene	0.5	0.7	0.6
1519	2-Acetyl furan	0.1	0.1	0.2
1571	*trans*-*p*-Menth-2-en-1-ol	0.1	0.1	tr
1583	*cis*-Isopulegone	0.4	0.3	0.3
1590	Bornyl acetate	0.1	0.1	0.1
1594	*trans*--Bergamotene	0.1	0.2	0.2
1611	Terpinen-4-ol	0.1	0.1	0.2
1612	-Caryophyllene	0.3	0.4	0.4
1638	*cis*-*p*-Menth-2-en-1-ol	0.1	0.1	tr
1648	Myrtenal	0.1	0.1	0.1
1668	(*Z*)--Farnesene	0.7	0.8	0.8
1670	*trans*-Pinocarveol	0.1	0.1	0.1
1697	**Carvotanacetone**	**0.2**	**0.1**	**0.1**
1706	-Terpineol	0.1	tr	0.5
1708	-Acoradiene	0.8	0.7	0.3
1740	Valencene	0.2	0.2	0.1
1741	-Bisabolene	0.2	0.2	0.2
1744	Phellandral	0.1	0.1	0.1
1802	Cumin aldehyde	19.9	23.6	20.4
1811	***p*-Mentha-1,3-dien-7-al**	**14.1**	**17.5**	**11.4**
1816	***p*-Mentha-1,4-dien-7-al**	**16.9**	**13.9**	**16.0**
2008	Caryophyllene oxide	0.1	0.2	0.1
2045	Carotol	1.5	1.4	1.2
2050	(*E*)-Nerolidol	0.1	0.1	tr
2073	*p*-Mentha-1,4-dien-7-ol	0.5	0.4	0.4
2113	Cumin alcohol	0.2	0.2	0.3
2144	Spathulenol	0.5	tr	0.1
2250	-Eudesmol	0.1	0.1	0.1
2931	Hexadecanoic acid	0.1	0.1	0.1
	Total	**97.0**	**97.1**	**97.2**

RRI: relative retention indices calculated against n-alkanes
% calculated from FID data
tr: trace (< 0.1 %)

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