International Journal of Plant Science and Ecology
Vol. 1, No. 3, 2015, pp. 93-97
http://www.aiscience.org/journal/ijpse

# Volatile Oil Composition of *Anethum graveolens* Affected by Harvest Stage

**Hussein A. H. Said Al Ahl[1, *], Atef M. Z. Sarhan[2], Abou Dahab M. Abou Dahab[2], El-Shahat N. Abou Zeid[1], Mohamed S. Ali[1], Nabila Y. Naguib[1]**

[1]Department of Medicinal and Aromatic Plants, National Research Centre, 33 El Bohouth St. (former El Tahrir St.), Dokki, Giza, Egypt
[2]Department of Ornamental Horticulture, Faculty of Agriculture, Cairo University, Cairo, Egypt

## Abstract

A field experiment was carried out to study the effect of harvest stage on essential oil and its constituents of dill plants at three stages of development. Essential oil percentage from fresh herb was the lowest (0.08%) in the vegetative stage and increased to (1.10%) in the flowering stage. Maximum essential oil percentages (3.20%) were achieved from dill plants in the seeds.The major components of the essential oil were α-phellandrene (46.33%), limonene (13.72%), β-phellanrene (11.7149%) and p-cymene (17.88%) in the vegetative stage; while, three main compounds were identified such as p-cymene (33.42%), carvone (13.10%) and dill ether (19.63%) of volatile oil distilled from fresh herb in the flowering stage. The maincompounds of the dill fruits essential oil in seeds were carvone (62.48%), dill apiole (19.51%) and limonene (14. 61%) in fruiting stage.

## Keywords

*Anethum graveolens* L., Essential Oil, Chemical Composition, Harvest Stage

Received: March 31, 2015 / Accepted: April 11, 2015 / Published online: April 20, 2015



## 1. Introduction

*Anethum graveolens* L. “Dill” is an annual herb, belonging to the family Apiaceae (umbelliferae). It originates from the Mediterranean and West Asia. Dill was widely distributed in antiquity and is now grown worldwide. It is one of the first-known multi-purpose plant which has been used as a spice and medicine. Dill fruits are used as a carminative, antispasmodic, sedative, lactagogue, diuretic, stimulant and to treat haemorrhoids, bronchial asthma, neuralgio, remal colic, dysurea, genital ulcers and dysmenorrhoea (Mahran et al., 1992). Dill herb is marketed fresh or dried for culinary use; chopped green herbage is sprinkled on foods and may also be used to flavor pickles. Herb oil is mainly used in the food industry for flavoring and seasoning, perfumes, cosmetics, soaps, creams, lotions and in detergents. Also, herb and seed oil have been widely investigated in respect of their antiseptic ability and exhibits anticarcinogenic, antimicrobial and antioxidant activity, although dill is also economically important. After oil extraction the residue contains approximately 15% protein and 16% fat and can be used as cattle fodder. (Weiss, 2002).

The biosynthesis of secondary metabolites, although controlled genetically, is strongly affected by the environmental influences of a particular growing region, and also by the agronomic conditions, harvesting time and the type of processing (Miguel et al., 2004; Baydar et al., 2004). Harvesting time of medicinal and aromatic plants is essential to obtain higher essential oil content and better quality (Filho et al., 2006), the oil composition is altered during harvesting process. In addition, for maximum oil production, long days and high light intensities are required during the maturation period (Green, 1985). However, Murray et al. (Murray et al., 1988 and Court et al., 1993) noticed that harvest date of the

*Correspondingauthor
E-mail address: hussein_saidalahl@yahoo.com (H. A. H. S. A. Ahl)

peppermint is a major factor in the composition of the essential oil and optimizing the date of harvest is essential for maximizing the production of oil of suitable quality.

No information has been cited in the literature concerning variation of aroma composition in dill volatile oil extracted from different various developing stages in Egypt. However, production information for dill herb essential oils and their compositions is lacking. Furthermore, there is poor information regarding the essential oil and its composition of dill herb at vegetative and flowering stages. Our experiment involving three development stages showed the great differentiation in aroma composition between each stage. Dill has been grown throughout theworld as an essential-oil-producing crop and a culinary herb with a large portionof the industry in Egypt, there has been no evaluation of essential oil productivity andquality of dill in this region. The objective of this research was to determine the potentialto grow dill as an essential oil crop in Egypt and toobserve the effect of harveststage on essential oil composition. It is possible to select the stage of the best aroma quality.To meet theseobjectives field experiments and laboratory analyses werecarried out in 2001 and 2002.

# 2. Materials and Methods

## 2.1. PlantMaterial and Experimental Procedure

The experiment of this study was carried out at the Farm Station of National Research Centre, at Shalakan Kalubia Governorate during the season 2001/2002. Seeds of dill (Anethum graveolens L.) were obtained from Medicinal and Aromatic plants Dep., National Research Centre, Dokki, Giza, Egypt. The dill seeds were sown on 15thOctober in the two seasons. The distance between each row was 60cm a part and 20cm between the hills. The seedlings were thinned one month after sowing to leave two plants per hill. The fresh herb was harvested at different dates. Data for essential oil and chemical constituents were obtained during three stages of development as follows; at the vegetative stage (90 days after sowing); at the flowering stage (135 days after sowing), and at the fruiting stage (210 days after sowing).

## 2.2. Essential OilandGLC Analysis

Essential oil percentage of dill at the three stages of development was determined according to Guenther (1961). The essential oil was dehydrated over anhydrous sodium sulphate and stored in freezer till used for gas liquid chromatographic (GLC) analysis.The GLC analysis of the essential oilsamples was carried out in the secondseason using gas chromatography instrumentstands at the Central Laboratory ofthe National Research Center with the followingspecifications. Instrument: Hewlett Packard 6890 series, Column: HP (Carbwax 20M) 25m length × 0.32mm I.D, Film thickness: 0.3Mm, Sample size: 1µl, oven temperature: 60-190 °C, Program: 60 °C/2min, 8 °C/min, 190 °C/25min. Injection port temperature: 240 °C, Detector temperature (FID): 280 °C, Carrier gas: nitrogen, Flow rate: N2 30 ml/min; H2 30 ml/min; air 300 ml/min. Main compounds of the essential oils were identified by matching their retention times with those of the authentic samples injected under the same conditions. The relative percentage of each compound was calculated from the area of the peak corresponding to each compound.

# 3. Results and Discussion

## 3.1. Essential Oil Percentage

The results recorded in the Table(1) showed that volatile oil percent in dill herb during vegetative stage was the lowest (0.08%) comparing to volatile oil in herb during flowering stage (1.10%), whereas the highest volatile oil percent was obtained in dill fruits (3.20%). Said-Al Ahl (2005) concluded that volatile oil in dill was (0.05 to 0.07%); (0.6 to 1.0%) and (2.6 to 3.4%)at vegetative, flowering and fruiting stages, respectively.Essential oil content of dill seeds ranges between 0.2 and4.6 % (Embong et al. 1977; Kruger and Hammer1996; Bailer et al. 2001; Kapoor et al. 2002; Santos et al. 2002),whereas the content of essential oil in dill herbage varies from0.09 to 0.34 % (fresh) (Wall and Friesen 1986; Santoset al. 2002). Essential oil product from the *Dracocephalum moldavica* plant varies depending on the date of harvest (Said-Al Ahl et al., 2015).

## 3.2. Essential Oil Constituents

Data presented in Table (1) pointed out variable changes in the percentages of different ingredients which constituted the constituents of volatile oil distilled from dill under different harvest dates. It can be remarked that 19, 17 and 13 compounds were identified from volatile oil of herb at vegetative, herb at flowering and seeds at fruiting stages, respectively. The main constituents of the essential oil distilled from fresh herb during vegetative stage was α-phellandrene (46.33%), limonene (13.72%), β-phellanrene (11.7149%) and p-cymene (17.88%). Three main compounds were identified such as p-cymene (33.42%), carvone (13.10%) and dillether (19.63%)of volatile oil distilled from fresh herb during flowering stage.Whereas, the main compounds were carvone (62.48%), dillapiole (19.51%) and limonene (14. 61%)of volatile oil distilled from dill seeds at fruiting stage.

From Table (1) we found that the highest percentages of both

of α-phellandrene and β-was found in essential oil distilled from herb at vegetative stage and less in herb at flowering stage and these compounds were disappeared completely in the essential oil distilled of seed at fruiting stage. While the highest percentage of both carvone and dillapiole was found in the essential oil distilled from seed, followed by essential oil distilled from herb at flowering stage and lower percentages in the vegetative stage as well as limonene compoundwas of the highest percentage in the seed followed byessential oil distilled from herb at vegetative stage and the lowest rate in essential oil distilled from herb at the flowering stage. But p-cymene and dillether had the highest percentages of both in essential oil distilled from herb at flowering stage and the lowest percentages was found in essential oil distilled from fruits and herb at vegetative stages in p-cymene and dillether, respectively.Said-Al Ahl and Omer (2009) found that essential oil content and its composition of coriander differ with different development stages.Said-Al Ahl et al. (2010) reported that the major compounds in essential oil extracted from dill herb at flowering stage were p-cymene,dill ether and dillapiole. The content andcomposition of an essential oil of *Anethum graveolens* are dependent on many factors,such as plant part, harvest time, extractionmethod, type of cultivar, storageconditions, growth conditions, geographicalorigin and state of maturity, etc. (Leopoldet al., 2003). Babri et al. (2012) found that carvone (38.899%), apiole (30.812%), limonene (15.938%) and dihydrocarvone (10.999%) were the main components of dill fruits essential oil growing in Pakistan. Ashraf et al. (1977) analyzed the dill seed essential oil by GLCand found the oil to be rich in carvone (52.25%), dill apiole (28.28%) and limonene (9.34%). Singh et al. (2005) reported carvone (55.2%), camphor (11.44%), limonene (16.6%) and dill apiole (14.4%) to be the key components present in theessential oil extracted from the seeds of *A.graveolens* L.Also, Jirovetz et al. (2003) who reported carvone (50.1%) andlimonene (44.1%) as the major constituents of the*A.graveolens* essential oil from Bulgaria.

The essential oil composition of dill is different in theherbage and seeds. Santos et al. (2002) reported carvone (67%) and limonene (23%) as the main components of dillseed oil and α-phellandrene (62%), dill apiole (10%), and myristicin (7%) as the main components of the herbage oil.Similar seed oil composition, limonene (46.3%) and carvone (49.5%) were reported by Delaquis et al. (2002). Incontrast, another analysis of dill seed oil determined dill apiole (61.6%) and carvone (23%) as the main components (Kapoor et al., 2002); furthermore (Wall and Friesen, 1986) reported the main components of dill seed oil to be carvone, limonene, and α-phellandrene. Dill seeds of a Bulgarian cultivarcontain limonene (43.7%), carvone (41.2%), dihydrocarvone(3.1%), and myristicin (11.7%) (Kruger andHammer, 1996). The North American dill seed oil trade isbased on carvone content in the essential oil. The minimumlevel of acceptable carvone is 30% (Wall and Friesen, 1986). Embong et al. (1977) reported a significant increase in carvonecontent as the dill plant matures, and a decrease inα-phellandrene.The essential oil quality and productivity of dill and otheressential oil crops depend on many factors such as climate, cultivar, seeding date, harvest date, weed pressure, plant disease, and management practices (Frank et al., 1987; Kothariet al., 1989; Zheljazkov and Zhalnov, 1995; Rangappa et al., 1997; Gil et al., 2002).

**Table 1.** Principal constituents of dillessential oil at three harvest dates

| Compound | First Harvest (herb at vegetative herb) | Second Harvest (herb at flowering herb) | Third Harvest (fruits at fruiting) |
|---|---|---|---|
| α-Pinene | 1.63 | - | - |
| β-pinene | 0.27 | - | - |
| α-phellandrene | 46.33 | 0.59 | - |
| Limonene | 13.72 | 0.48 | 14.61 |
| β-phellandrene | 11.01 | 2.70 | - |
| γ-terpinene | 0.36 | - | - |
| P-cymene | 17.88 | 33.42 | 0.30 |
| Linalool | - | 0.35 | 0.01 |
| Dillether | 0.45 | 19.63 | 1.64 |
| Dihydrocarvone | 0.22 | 0.73 | 0.07 |
| Sabinol | 1.04 | 0.31 | 0.03 |
| Carvone | 2.11 | 13.10 | 62.48 |
| Piperitone | 0.23 | 4.60 | 0.21 |
| Carveol | 0.66 | 3.24 | - |
| Nerolidol | 0.71 | 1.48 | 0.01 |
| Eugenol | 0.79 | 1.55 | - |
| Thymol | 0.59 | 0.85 | 0.01 |
| Carvacrol | 0.24 | 1.62 | 0.02 |
| Myristicin | 1.07 | 0.64 | 0.05 |
| Dillapiole | 0.59 | 4.16 | 19.51 |
| Total Identified compounds | 99.90 | 89.45 | 98.95 |
| Essential oil % | 0.08% | 1.10% | 3.20% |

## 4. Conclusion

It can be concluded that dill plant has a highest value of essential oil in fruits followed by essential oil of fresh herb at flowering stage and then essential oil of fresh herb at vegetative stage. It also appears likely that the major components of the essential oil were α-phellandrene, P-cymene, limonene and β-phellandrene in the vegetative stage; while, P-cymene, dilletherand carvonein theflowering stage. In the fruiting stage,carvone, dillapioleand limonene was the main compound of the essentialoil in seeds.

## References

[1] Babri, R.A., Khokhar, I., Mahmood, Z. and S. Mahmud (2012).Chemical composition and insecticidal activity of the essential oil of *Anethum graveolens* L. Sci. Int., (Lahore), 24(4): 453-455.

[2] Bailer, J., Aichinger, T., Hackl, G., de Hueber, K. and M. Dachler (2001). Essential oil content and composition in commercially available dill cultivars in comparison to caraway. Ind. Crops Products, 14: 229–239.

[3] Baydar, H., Sagdic, O., Ozkan, G. and T. Karadogan (2004). Antibacterial activity and composition of essential oils from Origanum, Thymbra and Satureja species with commercial importance in Turkey. Food Control, 15:169-172.

[4] Court, W.A., Roy, R.C. and R. Pocs (1993). Effect of harvest date on the yield and quality of the essential oil of peppermint. Can J Plant Sci., 73:8l5-824.

[5] Delaquis, P. J., Stanich, K., Girard, B. and G. Mazza (2002). Antimicrobial activity of individual and mixed fractions of dill, cilantro, coriander and eucalyptus essential oil. Int. J. Food Microbiol., 74: 101–109.

[6] Embong, M. B., Hadziyev, D. and S. Molnar (1977). Essential oils from spices grown in Alberta, dill seed oil (*Anethum graveolens* L. (Umbelliferae)). Can. Inst. Food Sci. Technol. J., 10: 208 214.

[7] Filho, J.L.S.C., Blank, A.F., Alves, P.B., Ehlert, P.A.D., Melo, A.S. andS.C.H. Cavalcanti (2006). Influence of the harvesting time, temperature and drying period on basil (*Ocimum basilicum* L.) essential oil. Brazilian Journal of Pharmacognosy, 16(1):24-30.

[8] Frank, J. R., Baron, J. J. and R. T. Guest (1987). Pesticides for use in herbs, spices and medicinal plants. Herb Spice Medicinal Plant Dig., 5: 1–12.

[9] Gil, A., De La Fuente, E. B., Lenardis, A. E., Pereira, M. L., Suarez, S. A., Arnaldo, B., Van Baren, C., Di Leo, L. P. and C. M. Ghersa (2002). Coriander essential oil composition from two genotypes grown in different environmental conditions. J. Agric. Food chem., 50: 2870–2877.

[10] Green, R.J. (1985). Peppermint and spearmint production in the Midwest. The Herb. Spice and Medicinal Plant Digest., 3(l):l-7.

[11] Guenther, G. (1961). The essential oils V.III. Robert E.D. Nastrand Comp. Inc. New York.

[12] Jirovetz, A., L, Buchbauer, G., Stoyanova, A.S., Georgiev, E.V. and S.T. Damianova (2003). Composition, quality control and antimicrobial activity of the essential oil of long time stored dill (*Anethum graveolens* L.) seeds from Bulgaria. J. Agric. Food Chem., 18: 3854-3857.

[13] Kapoor, R., Giri, B. and K. G. Mukerji (2002). Glomus macrocarpum: a potential bioinoculant to improve essential oil quality and concentration in Dill (*Anethum graveolens* L.) and Carum (*Trachyspermum ammi* (Linn.) Sprague). World J. Microbiol Biotechnol., 18: 459–463.

[14] Kothari, S. K., Singh, J. P. and K. Singh (1989). Chemical weed control in Bulgarian coriander (Coriandrum sativum L.) Trop. Pest Mgt., 35: 2–5.

[15] Kruger, H. and K.Hammer (1996). A new chemotype of *Anethum graveolens* L. J. Essen. Oil Res., 8: 205–206.

[16] Leopold, J., Buchbauer, G., Stoyanova, A. S., Gergiev,E. V. and S. T. Damianova (2003). Composition, quality control, and antimicrobial activity of the essential oil of long-time stored dill (*Anethum graveolens* L.) seeds from Bulgaria. J. Agric.Food Chem., 51:3854-3857.

[17] Mahran, G.H., Kadry, H.A., Thabet, C.K., El-Olemy, M.M., Al-Azizi, M.M., Schiff, P.L. and L.K.N. Wong (1992). GC/MS analysis of volatile oil of fruits of *Anethum graveolens.* Int. J. of Pharmacognosy, 30 (2) 139-144.

[18] Miguel, M.G., Guerrero, C., Rodrigues, H., Brito, J.C., Duarte, F. andF. Venâncio (2004). Main Components of the Essential Oils from Wild Portuguese *Thymus mastichina* (L.) L. ssp. mastichina in Different Developmental Stages or Under Culture Conditions. Journal of Essential Oil Research, 16:111-114.

[19] Murray, M.J., Marble, P., Lincoln, D. and F.W. Hefendehl (1988). Peppermint oil quality differences and the reasons for them. Flavors and Fragrances: Proceeding of the 10th International Congress of Essential oils, Fragrances and Flavors, Washington, DC, U.S.A., 189-208.

[20] Rangappa, M, Bhardwaj, H. L., Showhda, M. and A. A. Hamama (1997). Cilantro response to nitrogen fertilizer rates. SAS Institute, Inc. 2000. SAS version 8. SAS Institute Inc., Cary,NC. J. Herbs Spices Medicinal Plants, 5: 63–68.

[21] Said- Al Ahl H.A.H. (2005). Physiological studies on growth, yield and volatile oil of dill (*Anethum graveolens* L.). Ph. D. Thesis, Fac. Agric. Cairo Uni. Egypt.

[22] Said-Al Ahl, H. A. H. and E. A. Omer (2009). Effect of spraying with zinc and / or iron on growth and chemical composition of coriander (*Coriandrum sativum* L.) harvested at three stages of development. Journal of Medicinal Food Plants, 1(2): 30-46.

[23] Said-Al Ahl, H. A.H., Sabra, A.S., El Gendy, A.G., Aziz, E.E. and K. G. Tkachenko (2015). Changes in content and chemical composition of *Dracocephalum moldavica* L. essential oil at different harvest dates. Journal of Medicinal Plants Studies, 3(2): 61-64.

[24] Said-Al Ahl, H.A.H., Sarhan, A.M.Z., Abou Dahab, M.A., Abou-Zeid, E.N., Ali, M.S. and N.Y. Naguib (2010). Effected of nitrogen and/or biofertilizer on the essential oil of dill at the flowering stage. Journal of Medicinal Food Plants, 2(2): 21-26.

[25] Santos, P. A. G., Figueiredo, A. C., Lourenco, P. M. L., Barroso, J. G., Pedro, L. G., Oliveira, M. M., Schripsema, J., Deans, S. G. and J. J. C. Scheffer (2002). Hairy root cultures of *Anethum graveolens* (dill): establishment, growth, time-course study of their essential oil and its comparison with parent plant oils. Biotechnol. Lett. 24: 1031–1036. SAS Institute, Inc. 2000. SAS version 8. SAS Institute Inc., Cary, NC.

[26] Singh, G., Maurya, S., Lampasona, M.P.D. and C. Catlan (2005). Chemical constituents, antimicrobial investigations, and antioxidative potentials of *Anethum graveolens* L. essential oil and acetone extract: Part 52. Journal of food science, 70 (4):208 - 215.

[27] Wall, D. A. and G. H. Friesen (1986). The effect of herbicides and weeds on the yields and composition of dill (*Anethum graveolens* L.) oil. Crop Prot., 5: 137–142.

[28] Weiss, E.A. (2002).Spice Crops. CABI Publishing, 10E 40th street, Suite 3203, New York, NY 10016 U.S.A.

[29] Zheljazkov, V. and I. Zhalnov(1995). Effect of herbicides on yields and quality of *Coriandrum sativum* L. J. Essen. Oil. Res., 7: 633–639.