

**EFFECT OF DIFFERENT DRYING AND STORAGE METHODS
ON THE QUALITY OF SOME MEDICINAL PLANTS:
2- EFFECT OF STORAGE METHODS AND PERIODS OF
FENNEL FRUITS (*Foeniculum vulgare*, MILL.) ON THE
QUALITY OF ESSENTIAL OIL**

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ABSTRACT

This study was carried out on fennel fruits at the Experiment Station of Agriculture Faculty, Mansoura University during June 1993 and 1994 seasons. The investigation was conducted to study the effect of two keeping methods (brown and transparent bottles) on the storage of powder and whole fruits of fennel for different intervals. Essential oil percentage and its physical and chemical properties were studied. The highest percentage of essential oil (1.80%) was obtained in powdered fruits stored in brown bottles for 3 months, while the lowest (0.66%) was obtained in whole fruits, stored in transparent bottles for 12 months. The lowest acid value and refractive index were detected in powdered fruits, stored in brown bottles for 3 months (1.87 and 1.37), respectively, while the highest values were of whole fruits, stored in transparent bottles for 12 months (2.50 and 1.55) respectively. The highest anethole percentage of (51.5%) was in powdered fruits, stored in brown bottles for 3 months, while the lowest of (40.3%) was in powder fruits, stored in transparent bottles for 12 months.

INTRODUCTION

Fennel (*Foeniculum vulgares*, Mill) is a member of Apiaceae family and was known by the Romans, Greeks and Babylonians. It was used to freshen the air, repel insects, increase nursery women milk, and depress appetite (Arslan *et al.*, 1990). Fennel is an important aromatic crop for flavouring foods, liquers and as an essence in cosmetics and perfumery. It is used in folk medicine for its balsamic, cardiogenic, digestive, lactogogue and tonic properties (Marotti *et al.*, 1994). Guenther (1961) reported that the fennel seeds must be crushed prior to distillation immediately, to avoid loss of oil by evaporation.

Akgul (1986) identified the essential oil constituents and quantified by GC and that trans - anethol contributed 75.68 % of the oil. Gurdip *et al.* (1990) examined the essential oil extracted from crushed fennel seeds by hydrodistillation, GLC and identified 20 components. The major component was found to be anethole (68%). Massoud (1992) found that, the oil percentage of the essential oil from Egyptian strain of fennel was 2.71% and the major constituent was anethole (40.63%). Venskutonis *et al.* (1996) using capillary GLC and found that the main constituent of the essential oil of fennel fruits was anethole (50.1%).

The storage of plant material offers some hazard in the way of ultimate loss of essential oil. Gradual evaporation results in some loss in essential oil content. Guenther (1949) stated that the plant material must be

stored in dry and low temperature atmosphere and in a room free from air circulation to avoid evaporation. He added that the major sources of losses represented by oxidation and resinification of the essential oil.

Fehr (1980) studied the changes of the essential oil contents of fennel, anise and caraway fruits during storage period. He found that anise and caraway fruits contain an essential oil which decreases by 1% and 2.8 % per month, respectively. In case of fennel, it was difficult to make a conclusion, because of the differences of their ripeness degree. The decrease of essential oils content in fennel samples ranged from 0.01 % to 0.153 ml per month.

The aim of this investigation was to study the effect of different storage methods and storage periods on percentage and quality of fennel fruits oil.

MATERIALS AND METHODS

The fruits of fennel (*Foeniculum vulgare*, Mill) was obtained from Experimental Station Faculty of Agricultural, Mansoura University and carried out during 1993 and 1994 seasons. The samples were randomized collected and divided in three replicates.

The experiment included:

- 1- Forms of the samples (whole, and fruits powder).
- 2- Keeping methods (transparent and brown bottles).
- 3- Storage periods (0, 3, 6, 9 and 12 months).

The samples were (1 kg) finely powdered and (1 kg) left as whole fruits. 100 g was taken from each sample to determine the oil percentage as control. 1.8 kg was taken from powdered and whole fruits, kept in brown or transparent bottles at room temperature for four storage periods (3, 6, 9, and 12 months). After the storage periods, 24 samples were chosen for essential oil extraction.

Oil determination:

The essential oil percentages were determined in the air dried fruit in both seasons by distillation in clevenger apparatus according to the method described by British Pharmacopoeia (1963).

Physical and chemical properties :

Physical and chemical characteristics of essential oil were specified by many scientists. The most frequent properties tested are refractive index, chemical properties as acid number. The chromatographic analysis was the most important technique for studying the essential oil constituents.

1- Acid number:

The acid number was determined according to the directions given by Guenther (1972)

2- Refractive index:

The refractive index of the essential oil was determined according to the method mentioned by Guenther (1949) the refractometer of Carl Zeiss, Germany was used at 20 °C.

3- Essential oil composition:

the essential oil obtained from whole and powder fruits was analysed using GCVPYE unicom Gas Liquid Chromatography (GLC) equipped with a flame ionization detector for separation of volatile oil constituents. This is undoubtedly the most important technique for the study of essential oils. The oil samples obtained from different treatments were subjected to GLC analysis. The percentages of main components (α -pinene, limonene, myrcene, fenchone, estragol and anethole) were calculated.

The use of GLC in the quantitative determination could be achieved by Bunzen *et al.*, (1969) and Harborne, (1973) methods which are based on peak area measurements. The percentage of component was the ratio between its peak area to the total area multiplied by 100.

Statistical analysis:

The obtained data were subjected to statistical analysis using technique of factorial experimental in randomized complete design according to Gomez and Gomez (1984).

RESULTS AND DISCUSSION

1. Oil percentage:

Data in Table (1) shows the effect of storage methods and periods and their effect on essential oil of powder and whole fennel fruits.

Grinding the seeds prior to distillation was important as it increased the oil percentage. Guenther (1961), realized the importance of grinding the seeds prior to distillation, as it increased the oil yield.

It was clearly seen that the oil percentage of fennel powder was higher than that of the whole fruits (1.82 and 1.65%) respectively. The highest percentage of essential oil (1.80%) was obtained from stored powder in brown bottles for 3 months, while the lowest percentage (0.66) was obtained by the whole fruits stored in transparent bottles for 12 months. The loss of essential oil was due to the long of storage periods (Marotti *et al.*, 1994).

The differences in essential oils percentage due to increment of storage periods were significant in most cases (Table 1). It was obvious that essential oil percentage of fennel powder and fruits decreased according to increase of storage periods. While certain types of essential oils, especially those containing alcohol as that of coriander are quite stable against prolonged storage. These results were obtained by Abd El-Aal (1990).

Georgiev and Khadzhiiki (1969) studied changes in oil of carium ajwan fruits during storage for 3 years at 14-25 °C. and concluded that essential oils decreased by 14% in 3 years at 14-25 °C, the quality of the oils worsens in storage of the fruits over a period of 3 years.

Guenther (1961) studied the effect of the light on the physical and chemical properties of sweet mrjoram oil during the storage for one year. He stated that light as a factor causing deterioration of essential oils is less important than moisture.

Table 1: Averages of essential oil percentage as affected by interaction between storage methods and periods of powder and whole fruits of fennel.

Storage treatments Methods Period (months)	Essential oil (%)			
	Powder fruits		Whole fruits	
	Brown bottles	Transparent bottles	Brown bottles	Transparent bottles
0	1.82		1.65	
3	1.80	1.70	1.60	1.50
6	1.70	1.64	1.50	1.40
9	1.67	1.33	1.40	1.20
12	1.27	0.90	0.90	0.66
LSD at 5 %	0.17		0.12	

2. Acid number:

Most essential oils contain only little amount of free acid, which can be calculated as acid number.

The acid number increases as the oil ages especially if it is improperly stored. Table (2) showed that, the acid number increased in case of whole fruits compared with powder fruits (1.88% and 1.86%) respectively. In case of storing fruits in transparent bottles compared with in brown bottles the acid number was higher to some extent in the former than in the latter. This was true in either the whole or powder fruits. The long storage periods led to more increment in acid number, due to the hydrolysis and oxidation of essential oil. The highest value was (2.50%) after 12 months in transparent bottles by whole fruits. The same results were found by El-Tamimi (1962) who found gradual increments in the acid value during the storage.

Lukyanov and Berestovaya (1973) reported that 20 months storage of coriander essential oil in air - tight containers in darkness or in light did not affect on the qualitative indices of the oil. However, storage in open containers caused deterioration of the oil as soon as after 4 - 5 months. Increases in the acid number were observed.

Table 2: Acid number of essential oil as affected by interaction between storage methods and storage periods of powder and whole fennel fruits.

Storage treatments Methods Period (months)	Acid number			
	Powder fruits		Whole fruits	
	Brown bottles	Transparent bottles	Brown bottles	Transparent bottles
0	1.86		1.88	
3	1.87	1.89	1.90	1.95
6	1.89	1.92	1.95	1.97
9	1.90	1.94	1.97	1.99
12	1.93	1.98	2.10	2.50
LSD at 5 %	NS		NS	

El-Ramal (1976) said that the increase in acidity of cumin oils during storage were generally considered indicative of the degree of deterioration. The increase in acid number is noted with oils rich in their ester content.

3. Refractive index:

Data in Table (3) showed that, the increment in refractive index was in linear relationship with the increase of storage period. In addition it should noted that refractive index was less in all cases in brown bottles than in transparent to some extent. Also the values in this respect were higher in general when whole fruits was compared with powder fruits. The changes which occurred in refractive index was caused by the deterioration of essential oil with storage increase. The same results were found by El-Zahwey (1978).

Table 3: Averages of refractive index as affected by interaction between storage methods and storage periods of powder and fennel fruits.

Storage treatments Methods Period (months)	Refractive index			
	Powder fruits		Whole fruits	
	Brown bottles	Transparent bottles	Brown bottles	Transparent bottles
0	1.37		1.37	
3	1.37	1.38	1.39	1.40
6	1.38	1.39	1.40	1.43
9	1.38	1.40	1.42	1.45
12	1.39	1.42	1.45	1.55
LSD at 5 %	0.05		0.03	

El-Tamimi (1962) studied the effect of temperature on the physical and chemical properties of nerolibigrade and petitgrain oil during storage. He found gradual increments in the refractive index and acid value during the 12 days of storage.

Oda (1982) stored geranium and cumin oils for two years to study the effect of the material of containers on the oil properties during the storage periods. He found that the oil samples which stored in glass containers well preserved, no major changes took place in refractive index.

Badei *et al.* (1991) studied the effect of different storage containers and conditions on the quality of Egyptian coriander fruits essential oil, and the following results were obtained: specific gravity, refractive index, acid number and ester number increased by storage of the oil for 12 months, while solubility in diluted ethyl alcohol decreased with storage of the oil. Also, they found changes occurred in chemical composition of coriander fruits conditions detected by Gas Liquid Chromatography. This means that, a great deterioration in quality of coriander fruits essential oil could happen with prolonged storage.

4. GLC study:

The results from GLC study of fennel oil during the second season are shown in Tables (4 and 5). The identified compounds were α -pinene,

myrcene, lemonene, fenchone, estragole, α -terpinol, anisaldehyde and anethole as main components. These results were in agreement with the results obtained by Guether (1961). The fenchone percentage in fennel oil considerably affected by the two different storage methods in brown and transparent bottles during 12 months storage period (Tables 4 and 5) in the whole and powder fruits. All treatments decreased fenchone percentage in the volatile oil. The highest fenchone percentage (8.75% and 8.57%), (7.63% and 7.73%) was determined in the oil distilled from whole and powder fruits after 3 months stored in brown and transparent bottles, respectively. The least fenchone percentage (6.63% and 7.00%), (6.01% and 6.25%) from whole and powder fruits after 12 months stored in brown and transparent bottles, respectively.

Also from data in Tables (4 and 5) it was noticed that all treatments increased the estragole percentage in the volatile oil of fennel compared with control. The highest estragole percentage (18.5%, 21.6% and 21.6%, 22.6%) was detected in oil distilled from whole powder and powder fruits, respectively in brown and transparent bottles for 12 months. The control samples had the least estragole percentage (15.2% and 15.8%) from whole and powder fruits, respectively if compared with the other treatments. This is due to rupture of vittae on powdering concerning the effect of storage treatments on the anethole percentage, data in Tables (4 and 5) showed that, the highest percentage of anethole was from the control treatment (52.4% and 52.2%) from whole and powder fruit oil. The main component anethole is produced in higher percentage from powdered fennel fruits (Table 5), (51.5% and 50.8%) after 3 months stored in brown and transparent bottles, respectively. Than that from whole fruits oil (50.5% and 46.5%) after the same storage period and method (Table 4).

Kapelev (1981) studied the essential oil obtained from *Foeniculum vulgare*, Mill. and found that the highest content was anethole (51.7 %).

Table (4): Effect of storage methods and periods on the percentage of main components in the essential oil of *Foeniculum vulgare* (whole fruits).

Storage methods Periods Component	Oil components percentage								
	Cont.	Brown bottles				Transparent bottles			
		3	6	9	12	3	6	9	12
α -pinenene	0.37	0.46	0.36	0.31	0.28	0.42	0.35	0.28	0.25
Myrcene	0.31	0.26	0.22	0.17	0.13	0.31	0.27	0.23	0.19
Limonene	0.25	1.09	1.04	0.71	0.70	1.22	1.23	1.04	0.82
Fenchone	8.85	8.75	8.41	7.51	6.63	8.57	8.28	7.65	7.00
Estragole	15.2	15.6	16.5	17.8	18.5	19.3	20.6	21.3	21.6
α -Terpinole	3.99	2.86	2.37	3.54	4.53	3.36	3.16	3.80	5.15
Anethole	52.4	50.5	50.5	48.7	48.6	46.5	45.0	44.3	41.3
Anisaldehyde	10.9	11.6	9.79	9.76	8.63	11.3	10.4	9.70	9.39
Unknown	7.73	8.88	10.8	11.5	12.0	9.02	10.7	11.7	14.3

Table (5): Effect of storage methods and periods on the percentage of main components in the essential oil of *Foeniculum vulgare* (powder fruits).

Storage methods Periods Component	Oil components percentage								
	Cont.	Brown bottles				Transparent bottles			
		3	6	9	12	3	6	9	12
α -pinenene	0.33	0.57	0.54	0.51	0.47	0.44	0.36	0.34	0.33
Myrcene	0.34	0.34	0.65	1.14	1.57	0.44	0.37	0.34	0.17
Limonene	0.65	1.09	1.04	0.81	0.84	1.03	1.13	1.01	0.86
Fenchone	8.35	7.63	6.57	6.12	6.01	7.73	6.72	6.57	6.25
Estragole	15.8	17.6	19.1	19.5	21.6	18.8	19.8	21.4	22.6
α -Terpinole	3.92	3.55	2.59	3.80	3.89	3.56	2.62	3.87	4.15
Anethole	52.5	51.5	50.0	47.6	40.9	50.8	48.9	43.3	40.3
Anisaldehyde	9.11	9.25	9.21	8.62	8.33	9.10	9.58	9.23	9.14
Unknown	9.00	8.27	10.3	11.95	16.3	8.10	10.5	14.0	16.3

Marotti *et al.* (1994) studied the chemical composition of fennel oils deyhrolistilled from the fruits of *F. vulgare.*, and noticed that main contituents were anethole, limonene, fenchone.

Venskutonis *et al.* (1996) using capillary GLC, found that the main constituents of the essential oil of fennel fruits were anethole (50.1 %).

Storing of the drug in brown bottles is better than storing in transparent ones. Anethole is produced in higher concentration on keeping the whole and the powder fruits in brown bottles than keeping the whole and powder fruits in transparent bottles (Tables 4 and 5). This may be due to the sensitivity of anethole to light and conversion to p,p, di-methoxystilbene (called photo-anethole). The latter is quite insoluble, and stilbene (called photo-anethole). The latter is quite insoluble, and therefore freshly prepared anethole upon standing becomes turbid and loses its ability to crystallize giving a viascid consistency, a bitter disagreeable taste, a yellow color. This is accompanied by an increase in the specific gravity. The mechanism of this conversion may be due to conversion to anisaldehyde as an intermediary product which is changed on polymerization into photo-anethole (Balbaa, *et al.*, 1976).

Soliman (1972) stored rosemary oil in both colour-less and brown bottles for five months at room temperature and found that the chemical changes in the sample kept in colourless bottles after two months were nearly similar to those which occurred in the sample kept in brown bottles for three months.

Georgiev (1972) studied the changes in oil during storage of fruits of coriander and fennel. They observed changes in content of carvone, α -pinene, β -pinene, anethole, limonene, camphene, p-thymol, cineole and phellandrene.

Khatab (1985) found that menthol content was increased in the samples of *Menth piperita.* oil which were stored in glass bottles at room

temperature for one year, but it was stable in the aluminium containers, while menthol content was almost stable.

Manova *et al.* (1986) stated that the changes in the physical and chemical properties of lavender oil were more pronounced when the samples were kept in metal containers. He added that glass containers were more suitable for the preservation of oil.

The storing of whole fruits as well as the powdered ones for longer periods lead to the loss of the oil content and the oil constituents especially anethole and estragole. So, it is more significant to prepare the oil directly after short time from collection to avoid the loss of essential oil or deterioration of its sensitive components especially the oxygenated ones or the unsaturated hydrocarbons. But, if it is urgent to store the fennel drug, colored bottles should be used and the drug should be in a powder form.

Marotti *et al.*, (1994) mentioned that powder fruits of fennel had the highest yield than whole fruits. Trans-anethole content decreased with degree of grinding.

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تأثير طرق تجفيف وتخزين مختلفة على خواص بعض النباتات الطبية :
٢- تأثير معاملات التخزين المختلفة على صفات الجودة للزيت الطيار في ثمار
نبات الشمر

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تم إجراء البحث على الثمار الكاملة والمطحونة لنبات الشمر وتخزينها لمدة ١٢ شهر في زجاجيات بنية اللون وشفافة اللون خلال موسمين متتاليين ١٩٩٣ و ١٩٩٤ في معمل قسم البساتين بكلية الزراعة جامعة المنصورة بهدف دراسة تأثير نوع الثمار على نسبة الزيت وصفاته الطبيعية والكيميائية وتأثير نوع التخزين (زجاجيات بنية أو شفافة اللون) ومدة التخزين حيث تم أخذ عينات كل ثلاثة شهور لتحليلها كيميائياً وطبيعياً وقد أعطت الدراسة النتائج الآتية:
- أعطت معاملة المقارنة (بداية التخزين) أعلى نسبة للزيت وأقل رقم الحموضة ومعامل الانكسار، حيث كانت ١,٨٢%، ١,٨٦، ١,٣٧ على الترتيب يليها معاملة التخزين في زجاجات بنية اللون لمدة ٣ شهور في الثمار المطحونة فكانت ١,٨٠%، ١,٨٧، ١,٣٧ على الترتيب.
- قلت نسبة الزيت الطيار وارتفع رقم الحموضة ومعامل الانكسار بإطالة فترة التخزين لمدة عام كامل باستخدام الثمار الكاملة والمحفوظة في زجاجات شفافة حيث كانت ٠,٦٦%، ٢,٥، ١,٥٥ على الترتيب.
- كانت أعلى نسبة للمركب الرئيسي للزيت الطيار (الأنيثول) ٥٢,٥%، ٥٢,٤% في بداية التخزين (معاملة المقارنة) في الثمار المطحونة والكاملة على الترتيب قبل التخزين وتم الحصول على المكونات الفعالة بطريقة الفصل الكروماتوجرافي.
- بعد التخزين أعطت الثمار المطحونة والمخزنة في زجاجيات بنية اللون لمدة ثلاثة شهور أعلى نسبة للأنيثول (٥١,٥%) أما أقل نسبة للزيت الطيار (٤٠,٣%) فكانت في الثمار المطحونة والمخزنة في زجاجيات شفافة اللون لمدة سنة.
ثبت أن ثمار الشمر المطحونة والمخزنة في زجاجات بنية اللون في جو الغرفة العادي لمدة ثلاثة شهور تعطي أفضل النتائج وعلى ذلك لا ينصح بزيادة مدة التخزين عن ٣ شهور.