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DIFFERENT METHODS TO EXTRACT VOLATILE OIL FROM LEAVES OF DIFFERENT MEDICAL PLANTS

Ebaid. M. T.
Agric. Eng. Res. Inst. (AEnRI), Giza.

ABSTRACT

The object of the present study was to investigate the effect of different drying methods on percentage and components of volatile oil distilled from some aromatic plants leaves. The natural sun drying at 30°C ±1 and the artificial drying using artificial batch dryer are the two different methods used to dry the leaves. Drying methods were carried out under three different levels of air temperature (35, 40 and 45 ^{o}C), and air velocity of (2 m/s), for different plants spearmint (*Mentha viridis* L.), sage (*Salvia officinalis* L.), and sweet basil (*Ocimum basilicum*, L. var. basilicum).

The obtained results indicated that:-

1- In natural drying, moisture content decreased from 88.7 to 13.7 %, 85.6 to 14.1 % and 89.2 to 13.9 % at drying time of 42, 46 and 44 hours for spearmint, sage and sweet basil, respectively.
2- In artificial drying the recorded drying times at drying air temperature of 35, 40 and 45 ^{o}C were 18, 16 and 14 hours for spearmint 20, 18 and 16 hours for sage, and 20, 18 and 16 hours for sweet basil moisture content decreased from 88.7 to 13.7 %, 85.6 to 14.1 % and 89.2 to 13.9 % for spearmint, sage and sweet basil, respectively.
3- Percentage of volatile oils distilled from the leaves of the studied aromatic plants, varied and it decreased as the drying air temperature increased over 40 ^{o}C.
4- The optimum distillation time to distillate volatile oils for dry leaves from spearmint, was one hour, while it was two hours for sage and sweet basil.
5- The maximum percentage of volatile oil components were obtained from artificial drying at 40 ^{o}C and sir velocity of 2 m/s.
6- Artificial drying of the studied plants at drying air temperature of 40 ^{o}C is recommended for shorter drying time, higher percentage of volatile oil, and getting dried plants without contamination.

INTRODUCTION

The cultivated area of Medicinal and aromatic plants in the world and recently in Egypt increased as worldwide and local demand increased. The operation of drying in Egypt is done by using natural sun and wind to dry aromatic plants, but low quality and high losses occur because during the drying process the aromatic plants are contaminated by dust, rain, birds, and insects. Industrial drying maintains the oil percentage and components of aromatic plants. Volatile oil is considered one of the product plant organic foods, and it is important as a secondary product. These volatile oil characteristic by easy separation from the plants by distillation and extracting methods, therefore called volatile oil. The post harvest processes are of crucial important in case of medicinal plants. Among these processes, drying which determines the oil percentage of the final products in terms of feature and active ingredients content. Hazra *et al*. (1990) indicated that drying in

direct sunlight needs 24 % less steam than required during distillation. Nedkov and Georgiev (1991) said that spearmint, is a major essential oil plant. Dried leaves are used for various blends of tea which are reputed to have medicinal properties. Mujumdar (1995) mentioned that relatively seldom used in the manufacture of final pharmaceutical products, band dryers find wide use in drying of raw materials, especially herbs and medicinal plants, usually several bands in one above another configuration are used. Bands are made of stainless steel screens, or perforated plates. Speed from several centimetres to about 0.5 m/min are used. Band widths vary from as low as 0.5 up to 2 m, drying air temperature in the range 80-100 ^{o}C, initial moisture contents of 45-100 %, and drying rate of 5-18 kg/m^{2} h are usual in industrial practice. Ozguven and Tansi (1999) found that in trials on Marjoram (origanum marjoram) in Cukurova, Turkey, the highest fresh (1077.2 kg/day) and dried herb yields (492.9 kg/day) and essential oil yield (77.7 litters/day) were obtained at the post flowering stage. The main components of the oil were gamma-terpinen, P-Cyomol and terpineol. Arafa, (2001) mentioned that, the specific quantity of essential oil (1m/100g) distillated from dried Marjorom 0.51 1ml/100g was higher than that distillated from both peppermint and M.pulegium (0.37 and 0.34 1m/100g) respectively, regardless of drying air temperature. The essential oil ratio represents the quantity ratio of essential oil distillated from dried leaves to that from the fresh leaves. CAGMC (2002) reported that Peppermint is one of the major aromatic plants in Egypt, one faddan producing about 15-20 ton fresh herbs and they give about 25-30 kg volatile oil. Ocimum, needed from 2-3 h. to distillate volatile oil by steam-distillation, one faddan produce about 15 ton fresh herbs and they give about 15 kg volatile oil. Also Salvia is one of the most important of aromatic plants, one faddan produce about 10-12 ton fresh herbs and 1 ton, dry herbs give about 10-12 kg volatile oil. Kassem *et al*. (2006) studded the effect of solar energy and other drying methods on quality of some medicinal plants. The solar drying (35^{o}C), natural drying (sun drying 30^{o}C), and artificial drying (in oven at 45^{o}C) are the three different systems used for drying Lemongrass (cymbopogon citrates), Oregano (Origanum vulgare), Spearmint (Mentha virdis) and Peppermint (Mentha pepperita). Arafa (2007) reported that the effect of drying air velocity on moisture content is not a pronounced when the air velocity increased from 1.1 to 2.0 m/s.

This research was conducted to investigate the effect of different drying methods (natural sun drying and artificial drying) on percentage and components of volatile oil distilled from some aromatic plants leaves.

MATERIALS AND METHODS

A) Materials:

In the present investigation the natural sun drying (at 30°C ±1) and the artificial drying using artificial batch dryer were used for drying aromatic plants of spearmint (*Mentha viridis* L.), sage (*Salvia officinalis* L.), and sweet basil (*Ocimum basilicum,* L. var. basilicum). For artificial drying method, a batch dryer was constructed at a private workshop in El-Bagour district,

Minofiea Governorate, Egypt. While, for natural sun drying method, the plant leaves were spreated over 1 x 1 m screen tray sited under sun shine and covered over night to prevent changes in moisture content during this period. The samples of plants were taken from the farm of Medicinal and Aromatic plants Research Department, Dokki, Giza, in 2008 at initial moisture content of 88.7, 85.6 and 89.2 % w.b. for spearmint (*Mentha viridis* L.), sage (*Salvia officinalis* L.), and sweet basil (*Ocimum basilicum*, L. var. basilicum), respectively. The oil percentage and its components were distillated from the plants leaves in the laboratory of the same department.

1) Dryer specification and description:

Artificial batch dryer consists of centrifugal fan, air heating unit and drying bed as shown in fig. (1). A centrifugal fan of 1 hp (0.75 kW), and 1200 rpm., was used for air supply to the drying chamber through a control valve. The heating unit consists of two electric heaters (1.5 kW) installed inside an insulated box and connected with an electric switch and a digital thermostat to control the drying air temperature. Drying bed consists of a perforated stainless-steel sheet fixed over an iron frame constructed of iron angles of half-meter long and half-meter wide with a four sides of 10 cm high covered with 1 mm galvanized iron sheets. The frame was located over a plenum chamber constructed of iron angle frame with a height of 25 cm and covered with double layers steel sheets that contain fibber glass in between as an insulation material to reduce heat losses from the plenum chamber.

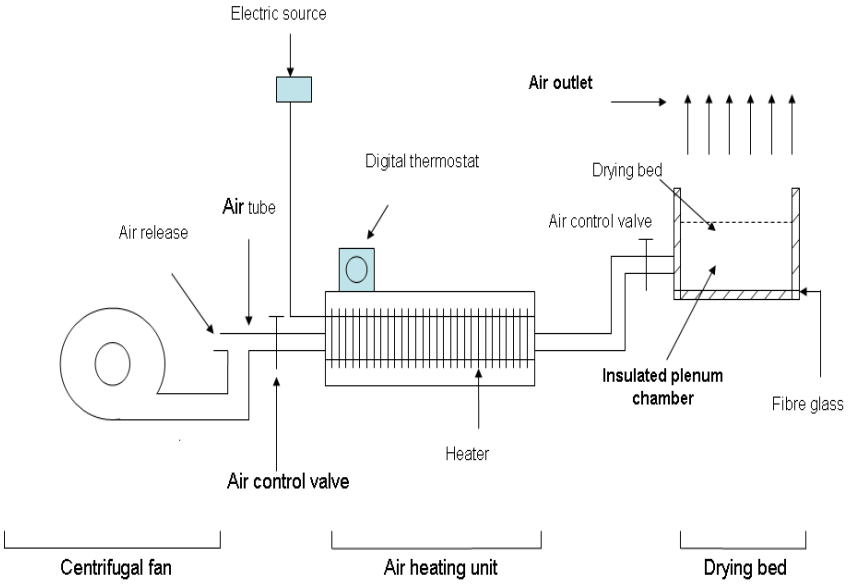

Fig. (1): Schematic diagram of the experimental dryer.

2) Distillation unit:

Distillation of the volatile oil was preceded as described in the British Pharmacopoeia (1963). The volatile oil obtained from the herbs was analyzed using Dschrom 6200 Gas Chromatograph equipped with a flame ionization detector for separation of volatile oil constituents.

3) Instrumentation:

A hot wire anemometer model (sp-51) was used for measuring the air speed inside the air supply tube, ranging from 0 to 50 m/s. A 12 channel digital thermometer with thermocouples (type T) model T.M-201 was used to measure temperature, with a range of 0 to 100 ^{o}C and accuracy of 0.01 ^{o}C. A digital balance with accuracy of 0.2 g was used, to measure the changes of samples mass (before and after drying).

4) Experimental treatments:

The following variables were tested to show their effects on required drying time, percentage and components of volatile oil distilled from the studied aromatic plants leaves:

1-Drying air temperature: three different levels of air drying temperature were functioned (35, 40 and 45 ^{o}C), in comparison with natural sun drying method at 30 ^{o}C.

2-Air speed: air speed of 2 m/sec was used as recommended by (Arfa 2007).

3-Distillation time:6 different times were used (0.5, 1, 1.5, 2, 2.5 and 3 h).

5) Experimental procedure.

The samples of aromatic plants were, washed to remove the mud before entering into the drying unit. The initial moisture content was determined by standard oven method (drying the sample of 10 g at 105 ^{o}C for 3 h.). Before the start of the drying test, the dryer was allowed to run for 30 min using a dummy samples. The air flow was regulated using the control valve installed between the heating unit and the centrifugal fan, while the drying air temperature was adjusted by the digital thermostat. The actual drying tests were continued until the moisture content was in equilibrium with the temperature of the drying air. The air temperature and moisture content was recorded every hour. At the end of the drying test, the moisture content of the dried samples was also determined as described by AOAC (1990). The oil content and components of plants were analyzed at the laboratory of the Medicinal and Aromatic plants Research Department, Horticulture Research Institute.

6) Experimental measurements:

6-1) Moisture content (M.C. w.b. %):

The moisture content in wet basis was measured for the tested aromatic plant by taking random samples (10 g) from leaves of each plant and drying it in electric oven at 105 ^{o}C for three hours to measure its moisture content according to AOAC (1990). The moisture content was calculated using to the following equation:

$$\mathbf{Mc_{w.b}} = \frac{M_w - M_d}{M_w} x\,100 \qquad \text{--------(1)}$$

Where: $Mc_{w.b}$ = Moisture content, wet basis %; M_d = Mass of dry samples, g; and M_w = Mass of wet samples, g.

6-2) Plant bulk temperature:

The plants bulk temperature was measured by installing the thermocouples at 6 different points represents all the drying area and the hourly average of readings were taken for illustrating the changes of bulk temperature with drying time.

6-3) Volatile oils:

Distillation of the volatile oil was preceded as described in the British Pharmacopoeia (1963). The percentage of volatile oil of different plants was measured at the end of drying process and calculated as follows:-

$$\text{Volatile oils \%} = \frac{volum\ of\ distillation\ volatile\ oils}{weight\ of\ leaves\ sample} \times 100 \quad \textbf{--(2)}$$

RESULTS AND DISCUSSION

1) Natural drying method:

Fig. (2) shows the change in moisture content % (w. b.) for natural drying method as a function of drying time for different studied plants. The moisture content was found to decrease as the drying time increased. The moisture content decreased from 88.7 to 13.7 %, 85.6 to 14.1 % and 89.2 to 13.9 % for spearmint, sage and sweet basil at drying times of 42, 46 and 44 hours respectively.

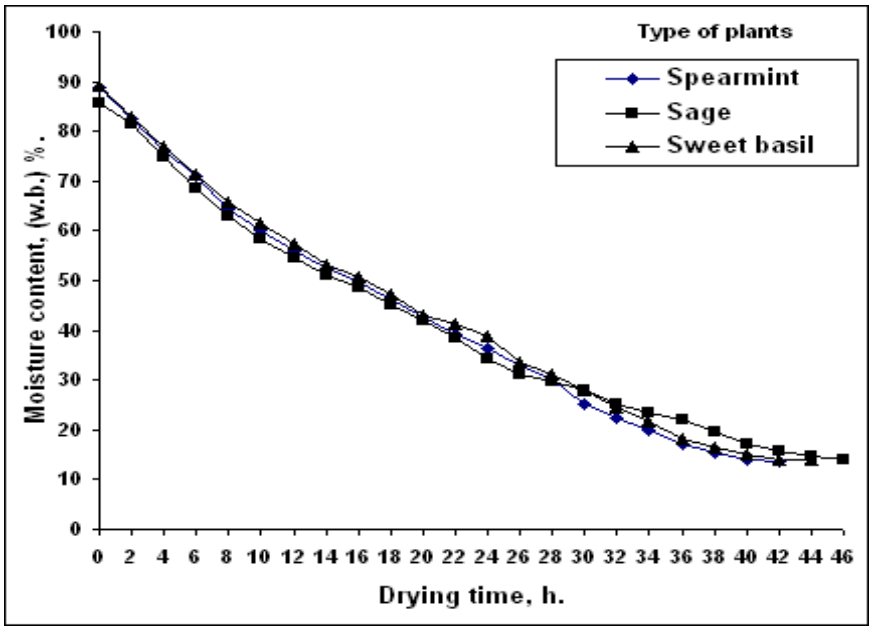

Fig. (2): Change of moisture content as related to drying time for natural drying method

2) Artificial drying method:

2-1 Bulk temperature of the studied plants:

Fig. (3) illustrate the change in bulk temperature as a function of drying time for spearmint, sage and sweet basil respectively. As shown in the figure, for all studied plants, the bulk temperature increased during the early stage of drying and starts to decrease again due to the evaporative cooling during the drying process and approached levels very close to the drying air temperature during the end period of drying. Also, the plants bulk temperature varied with the kind of plant and the level of drying air temperature. The average bulk temperatures of different plants at drying air temperatures of 35, 40 and 45 ^{o}C were 32.37, 35.02 and 39.01 ^{o}C for spearmint, 32.64, 35.46 and 37.87 ^{o}C for sage, and 33.87, 37.76 and 40.77 ^{o}C for sweet basil respectively.

2-2 Moisture content of the studied plants:

Fig. (4) illustrate the change in moisture content (% w.b.) as a function of drying time for spearmint, sage and sweet basil plants, respectively. As shown in the figures for all studied plants, the moisture content decreased in higher rate during the early stage of drying and the drying rate starts to decline with the progress of drying time. Also, the drying rate was increased with the increase of drying air temperature and consequently the drying time decreased. The recorded drying times at drying air temperatures of 35, 40 and 45 ^{o}C were 18, 16 and 14 hours for spearmint, 20, 18 and 16 hours for sage, and 20, 18 and 16 hours for sweet basil, respectively.

3) Effect of drying methods on percentage of volatile oils:

Data in table (1) presents the influence of two different methods of drying on percentage of volatile oils at different drying air temperature. The percentage of distillate volatile oils from naturally dried herbs under sun shin (30°C ±1) were 0.329, 1.351 and 0.386 % for spearmint, sage and sweet basil respectively. However, the percentage of distillate volatile oils was varied for the artificially dried plants and it was dramatically decreased as the drying air temperature increased over 40 ^{o}C presented in table (1). In general for the artificial drying method, the drying air temperature of 40 ^{o}C was found to be the optimum the getting the highest oil percentage. This means that, the volatile oil of the studied plants was affected by both longer drying time and higher drying temperature.

Table (1): Effect of drying methods and drying air temperature on percentage of volatile oils distilled at different kind of aromatic plants.

Plant condition	Drying methods	Temperature	Percentage of volatile oils, (%).		
			Spearmint	Sage	Sweet basil
Dry leaves	Natural	30°C ±1	0.329	1.351	0.386
	Artificial	35 ^{o}C	0.415	1.425	0.471
		40 ^{o}C	0.439	1.438	0.496
		45 ^{o}C	0.318	1.329	0.350

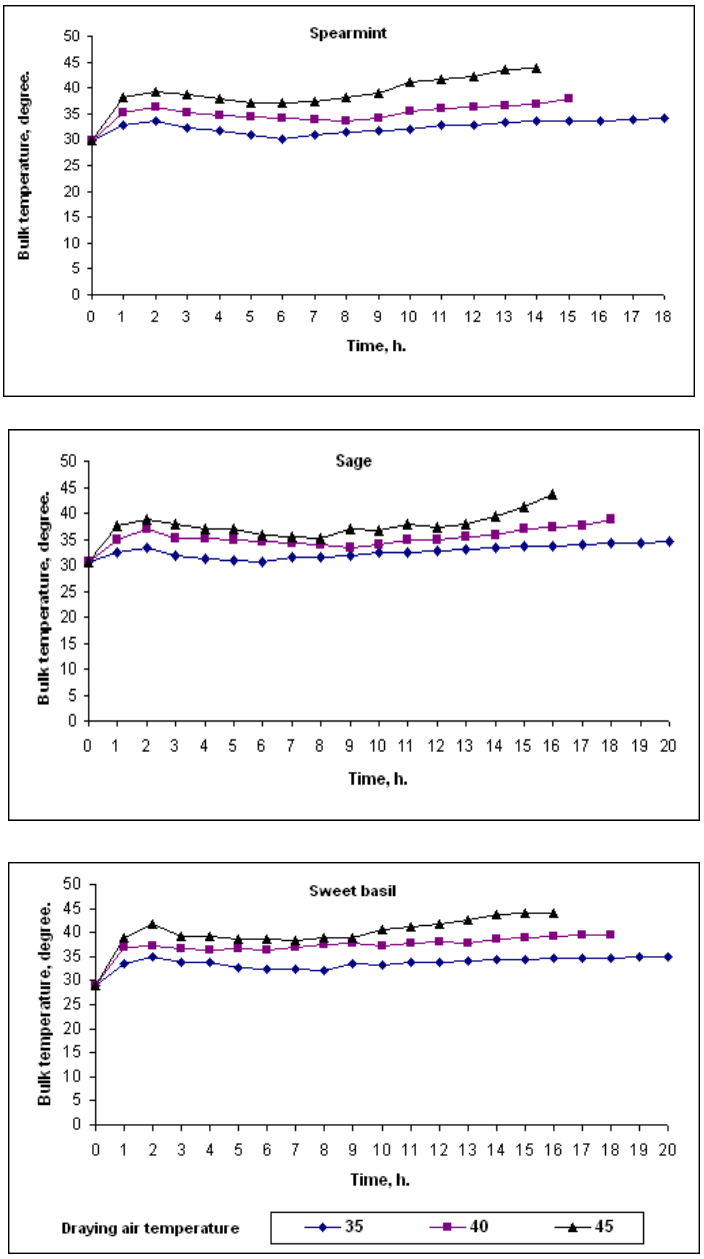

Fig. (3): Change in bulk temperature as a function of drying time for different studied plants.

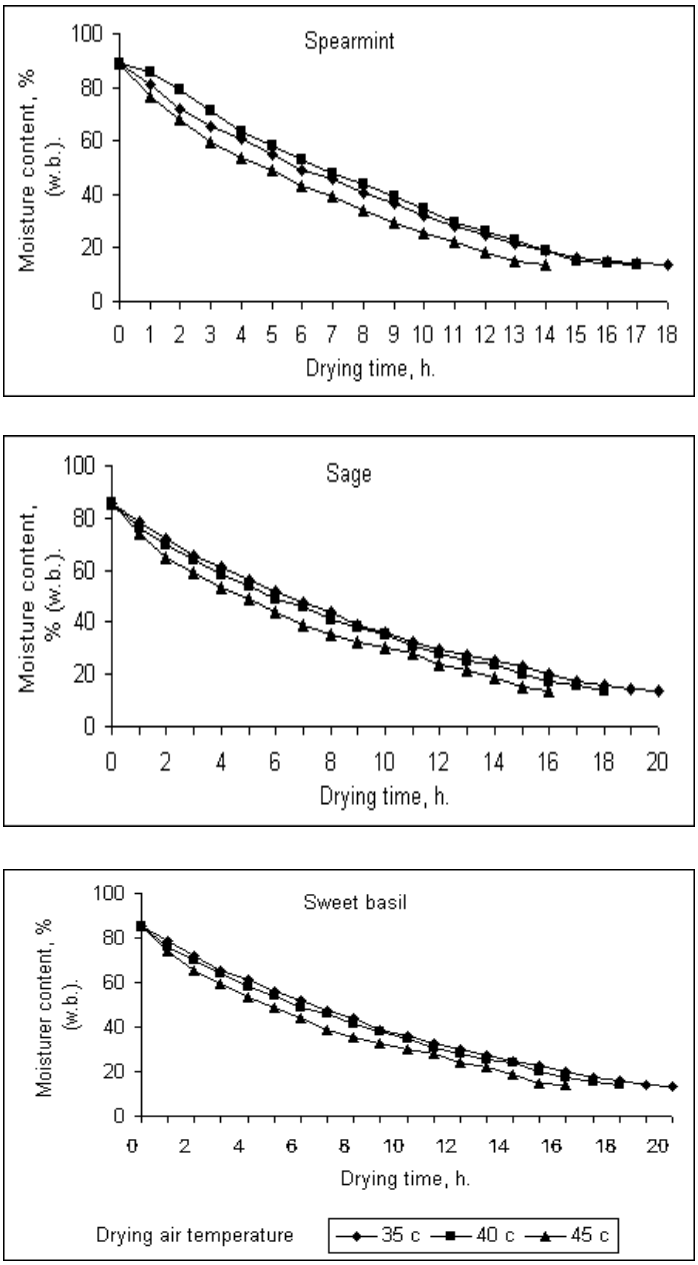

Fig.(4):Change in moisture content as a function of drying time for different studied plants.

4) Effect of plant conditions and drying method on the volatile oil components percentage :

Data in table (2) show the effect of plant conditions and drying method on the percentage of volatile oil components of spearmint, sage and sweet basil. As shown in the table for artificial drying method the air temperature of 40 ^{o}C recorded the highest percentages of the main components of volatile oil followed by the air temperature of 35 ^{o}C. Also, the temperature of 45 ^{o}C causes a noticeable reduction in volatile oil percentage as a result of the losses of the volatile comments of the oil. On the time, the longer drying time of the natural sun drying method causes considerable losses of the plants leaves which resulted in corresponding losses of the volatile oil percentages and components.

Table (2): Effect of plant conditions and drying method on the percentages of volatile oil components of spearmint, sage and sweet basil

P No	Components	Treatments				
		Fresh Herb	Dry herb			
			Natural Drying (30°C ±1)	Artificial drying		
				35°C	40°C	45°C
		Spearmint				
1	A-pinene	1.43	1.34	1.38	1.40	1.07
2	B-pinene	4.01	3.99	3.41	3.49	3.40
3	Limonene	16.35	16.01	16.24	16.30	16.29
4	1,8 cineole	1.02	0.99	0.41	0.90	0.40
5	Γ-terpinene	1.09	0.62	0.70	0.72	0.70
6	Linalool	5.15	5.01	5.07	5.02	4.90
7	Carvone	60.99	59.00	60.01	60.27	59.56
8	A –terpineol	2.41	2.38	2.39	2.40	2.06
9	B-carophyllene	3.35	3.17	3.31	2.25	2.86
10	Eugenol	1.61	1.42	1.56	1.61	1.45
*	Unidentified	2.39	4.53	5.07	4.13	6.59
		Sage				
1	A –pinene	4.88	4.37	4.83	7.33	5.46
2	Camphene	4.95	4.06	4.71	8.98	4.71
3	B-pinene	7.94	7.43	7.35	7.88	7.19
4	1,8Cineole	2.40	2.27	2.35	2.39	1.49
5	Thujone	44.25	35.25	42.43	43.66	40.62
6	Linalool	1.46	1.00	0.64	0.84	0.62
7	Methyl chavicol	2.26	1.05	0.95	0.97	0.90
8	Linalyl acetate	2.67	2.55	2.10	1.90	1.55
9	Camphor	9.01	7.81	8.44	9.05	7.29
10	Borneol	3.01	2.62	2.90	2.98	3.13
11	B-caryophyllene	2.18	1.57	2.15	2.16	1.48
		Sweet basil				
1	A –pinene	0.30	0.25	0.35	0.56	0.32
2	B-pinene	1.37	1.10	1.21	1.35	1.13
3	1,8Cineole	7.70	6.38	7.13	11.01	6.76
4	Linalool	43.65	30.76	33.04	34.48	31.89
5	Methyl chavicol	19.87	19.55	19.66	19.77	18.56
6	Methyl cinnamate	1.64	1.39	1.43	1.54	0.72
7	Eugenol	2.78	1.84	1.89	2.54	1.02
*	Unidentified					

5) Optimum distillation time and percentage of distillate volatile oils:

Fig. (5) shows the effect of plant conditions on distillation time and percentage of distillate volatile oils for different plants under study.

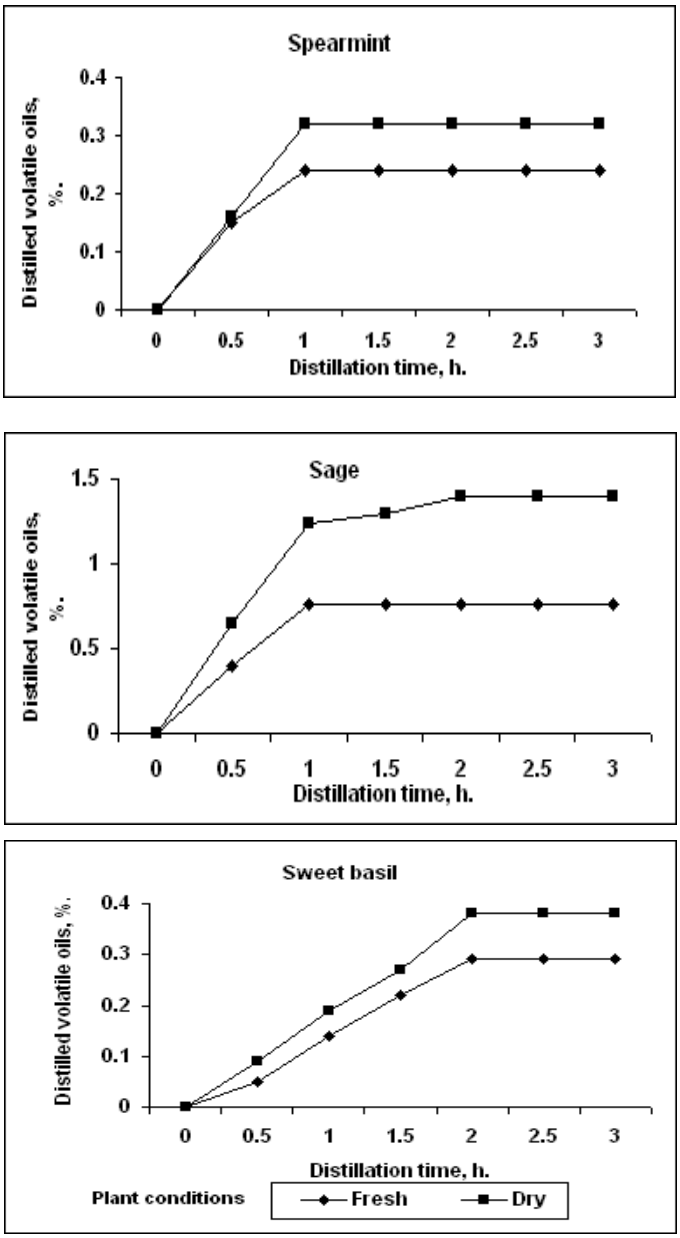

Fig. (5): Effect of distillation time on distillate volatile oil percentage of different studied plant.

From fig. (5) the optimum distillation time for fresh leaves was found to be one hour (first hour) for spearmint, and sage, which refers to the highest quantity of distilled volatile oils in leaves at the first hour. While, it was two hours (second hour) for sweet basil, in which the highest sweet basil percentage of distilled oil was obtained at the end of the second hour. However the optimum distillation time for the dry leaves was found to be one hour (first hour) for spearmint, and it was two hours (second hour) for sage and sweet basil.

A comparison between natural drying and artificial drying methods showed that, the artificial drying is faster than natural drying, and may keep the essential oil components content as high as possible. But the natural sun drying method can not protect the plants from dust, rain, rodents, birds and insets, and causes contamination with partly pathogenic micro organisms. Also, it was found that, the natural sun drying causes high losses or incomplete dehydration and a corresponding reduction in distilled oil percentage. In general artificial drying of the studied plants at drying air temperature of 40 ^{o}C is recommended for shorter drying time, acceptable percentage of volatile oil, and getting dried plants without contamination with pathogenic micro organisms, dust, insects,…… ect.

CONCLUSION

The results indicated that:

1- In natural drying, moisture content decreased from 88.7 to 13.7 %, 85.6 to 14.1 % and 89.2 to 13.9 % at drying time of 42, 46 and 44 hours for spearmint, sage and sweet basil, respectively.

2- In artificial drying the recorded drying times at drying air temperature of 35, 40 and 45 ^{o}C were 18, 16 and 14 hours for spearmint, 20, 18 and 16 hours for sage, and 20, 18 and 16 hours for sweet basil moisture content decreased from 88.7 to 13.7 %, 85.6 to 14.1 % and 89.2 to 13.9 % for spearmint, sage and sweet basil, respectively.

3- Percentage of volatile oils distilled from the leaves of the studied aromatic plants, varied and it decreased as the drying air temperature increased over 40 ^{o}C.

4- The optimum distillation time to distillate volatile oils for dry leaves from spearmint, was one hour, while it was two hours for sage and sweet basil.

5- The maximum percentage of volatile oil components were obtained from artificial drying at 40 ^{o}C.

6- Artificial drying of the studied plants at drying air temperature of 40 ^{o}C is recommended for shorter drying time, higher percentage of volatile oil, and getting dried plants without contamination.

REFERENCES

AOAC, Association of Official Analytical Chemists (1990) "Official Methods of Analysis" 15th ed. Association of Official Analytical Chemists, Washington D.C., U.S.A.

Arafa, G. K. (2001) "Factors affecting drying aromatic plants" P.hD. Th.., Fac. of Agric., Al-Azhar Univ., Egypt:99-105.

Arafa, G. K. (2007) "Optimum drying conditions for thin-layer drying of sweet basil" Misr J. Ag. Eng., 24(3):540-556.

British Pharmacopoeia (1963) "Determination of volatile oil in drugs" The Pharmaceutical Press, London.

CAGMC (Centre Authority for General Mobilization and Statistics (2002) "The annual stistical book for A.R.E.".

Hazra, P., A. P. Kahol and J. Ahmed, (1990) "Study of the effect of mode of drying on the yield, quality and steam consumption in distillation of the essential oil of Mettha arvensis" India-perfumer 34 (1) :47-55.

Kassem, A. M., I. E. El-Batawi and Mahassen M. S. (2006) "Effect of solar energy and other drying methods on quality of some medicinal plants" The 14th Annual Conference of the Misr Society of Ag. Eng., 22 November :766:782.

Mujumdar, A.S. (1995) "Handbook of industrial drying" 2 nd. Ed. revised and expanded. Vol.(2).:150-174.

Nedkov, N. K.; and G. V. Georgiev, (1991) "A study of different irriagation practices used for Mrntha piperita in Bulgaria" Journal of essential oil research. 3 (6) ;435-440.

Ozguven, M. and Tansi, S. (1999) "Determination of yield and quality in marjoram as influenced by development periods" Turkish-J of Agr and Forestry. (23)1: 11-17.

الطرق المختلفة لأستخلاص الزيوت الطيارة من اوراق النباتات الطبية المختلفة

محمد طه عبيد

معهد بحوث الهندسة الزراعية - الدقي - جيزة.

تزداد الاهمية الاقتصادية والعلمية للنباتات العطرية يوما بعد يوم، وذلك نظرا لما يترتب علية من استعمال المواد الكيماوية من أضرار وأثار جانبية تؤثر على مستعمليها بالسلب. مما جعل فى الاونة الاخيرة الاهتمام والاتجاة للنباتات الطبية والعطرية أى الرجوع الى الطبيعة. والفائدة من هذة النباتات تكمن فى الزيت التى تحتوية خلاياها والتى تدخل فى كثير من الصناعات ومنها صناعة الروائح والعطور، وصناعة المنتجات الزراعية والحيوانية، وصناعة المربى والحلويات، والصناعات الدوائية كمواد طبيعية بدلا من الصناعية.

لذا يتناول هذا البحث دراسة طرق التجفيف المختلفة والمؤثرة على نسبة ومكونات الزيوت المستخلصة من بعض اوراق النباتات العطرية، وقد تم تغيير درجة حرارة التجفيف الصناعى فى المدى 35 - 45 م ^{5}، عند سرعة هواء التجفيف 2 م/ث ، وتم تغيير زمن التقطير فى المدى 0.5 – 3 ساعة وذلك لـ 3 نباتات عطرية مختلفة وهى النعناع (صنف النعناع الفلفى)، والمريمية (صنف المريمية الشائعة)، والريحان (صنف الريحان الحلو الابيض).

وكانت النتائج المتحصل عليها كالتالى:

- وجد انة فى حالة التجفيف الطبيعى ينخفض المحتوى الرطوبى لاوراق النباتات من 88.7 – 13.7، 85.6 – 14.1، 89.2 – 13.9 % للنعناع الفلفى، والمريمية الشائعة، والريحان الحلو الابيض عند زمن تجفيف 42، 46، 44 ساعة على الترتيب.
- فى حالة التجفيف الصناعى يقل المحتوى الرطوبى وزمن التجفيف من 18-14 ساعة للنعناع الفلفى، ومن 20-16 لكلا من المريمية الشائعة والريحان الحلو الابيض بزيادة كلا من درجات الحرارة من 35 الى 45 م ^{5} .
- وجد ان نسبة الزيوت المستخلصة من اوراق النباتات محل الدراسة متغيرة. وان نسبة الزيوت المستخلصة تقل بزيادة درجة حرارة هواء التجفيف عن 40 م ^{5}.
- وجد أن انسب زمن لتقطير الاوراق الجافة هو ساعة واحدة لنبات النعناع الفلفلى، وساعتان لكلاً من المريمية الشائعة، والريحان الحلو الابيض.
- أدت المعاملة بالتجفيف الصناعى عند درجة حرارة تجفيف 40 م ^{5} الى اعلى محتوى من مكونات الزيت الطيارللنباتات محل الدراسة.
- من النتائج السابقة يوصى باستخدام التجفيف الصناعى عند درجة حرارة التجفيف 40 م ^{5} وسرعة هواء 2 م/ث للنباتات محل الدراسة وذلك لتقليل زمن التجفيف وللحصول على اعلى نسبة للزيوت ومكوناتها حيث تقل نسبة الزيت الطيار ومكوناتها بارتفاع درجة الحرارة عن 40 م ^{5}.

قام بتحكيم البحث

أ.د / عماد الدين امين عبدالله — كلية الزراعة – جامعة المنصورة

أ.د / محمد سعد الدين الشال — كلية الزراعة – جامعة الزقازيق