ELSEVIER

Journal of Food Engineering xxx (2004) xxx–xxx

JOURNAL OF FOOD ENGINEERING

www.elsevier.com/locate/jfoodeng

Retention of aroma compounds in basil dried with low pressure superheated steam

S. Barbieri, M. Elustondo, M. Urbicain *

PLAPIQUI—UNS—CONICET, Camino La Carrindanga Km 7, 8000 Bahía Blanca, Argentina

Received 3 October 2003; accepted 4 January 2004

Abstract

Basil leaves were dried using either conventional hot air (50, 60 and 70 °C) and low pressure superheated steam (LPSS) dryers. The effect of the drying method on the retention of some volatile compounds was evaluated. The extraction from the fresh and dried products was performed by the simultaneous distillation–extraction technique. Identification and quantification was performed by capillary gas chromatography–mass spectrometry (GC–MS) and gas chromatography (GC) respectively.

The identified compounds were 23, with 61% of monoterpenes, 26% of aromatic and 13% of aliphatic compounds, out of which those characteristic of this spice were detected (1,8-cineole, methyl chavicol, methyl cinnamate and linalool).

Results show that in the LPSS dried product, the original aroma profile of the fresh vegetable is kept almost constant, while air-dried product shows a significant variation in the relative proportions of aroma compounds.

The conclusion is that the LPSS drying technique renders a better product in terms of the aroma compounds content, than the conventional air-drying, with the advantage of being cheaper.

1. Introduction

Dehydration is the most popular and cheap way of preserving foodstuffs. By reducing the water activity, micro-organisms growth is inhibited and adverse biochemical reactions are slowed down. The most ancient dehydration agent has been hot air, but it provokes undesirable changes in the physicochemical properties of the product, as a consequence of the thermal impact imposed on it and the presence of oxygen reacting with some components of the foodstuff.

Among these, volatile compounds are particular sensitive to those effects, and, being important as factors of quality and acceptance, the effect of drying on aromatic vegetables, spices and the like, has received attention by many authors (Huopalahti, Kesalahti, & Linko, 1985; Jerkovic, Mastelic, & Milos, 2001; Yousif, Durance, Scaman, & Girard, 2000; Yousif, Scaman, Durance, & Girard, 1999; Venskutonis, 1991, among many others).

Results have shown that changes in concentration of those compounds are always produced, the magnitude of which depend, for a given product, on the drying method and conditions.

An interesting alternative to the use of hot air is low pressure superheated steam (LPSS). It has been proved (Elustondo, 2001; Iwotech Limited, 1993; Kumar & Mujumdar, 1990; Mujumdar, 1980; Nomura & Hyodo, 1984; Svensson, 1980; Yoshida & Hyodo, 1960), that it results in significantly less damage as it works at lower temperatures and there is no oxygen present. The resulting product keeps the original colour and shape, and shows a highly porous structure which makes it easily re-hydrated. Since it has been proved that LPSS requires less energy than hot air-drying for the same duty, it makes the procedure attractive from the economical point of view also.

Thus far, there is no information about the impact of this procedure on the volatile compounds, both lost and retained in the dried product, hence this work is focused to study this point, i.e. the amount and quality of the original contents kept in the sample, as well as those captured from the withdrawn water vapor by means of cold traps. Also, a comparison is made between samples dried with hot air in a conventional drier and other dried with this technique.

* Corresponding author.
E-mail address: urbicain@criba.edu.ar (M. Urbicain).

0260-8774/$ - see front matter © 2004 Elsevier Ltd. All rights reserved.
doi:10.1016/j.jfoodeng.2004.01.003

Basil (*Ocimum basiculum*) is one of the preferred aromatic herbs for cooking and seasoning, and it is commercialised either fresh or dried. It belongs to the botany family Lamiacea, comprising many different species. Out of them the *O. basilicum* is the commonly used for cookery, pharmaceutical and cosmetic purposes (Simon, Morales, Phippen, Vieira, & Hao, 1999). The composition of volatiles depends on the species, origin, and culture conditions, but most authors have mentioned that 1,8-cineole, methyl cinnamate, methyl chavicol and linalool are the main responsible for the typical basil aroma (Backstroom-Sternberg, Stephen, Duke, & Wain, 1994; Díaz-Maroto, Pérez-Coello, & Cabezudo, 2002; Lachowicz et al., 1996, 1997; Simon et al., 1999; among others).

2. Materials and methods

2.1. Raw material

The experimented material was fresh basil (*O. basilicum*). Leaves were separated from the stems and stored at 4 °C until usage. Initial water content was determined on triple samples by using a laboratory vacuum oven at 50 °C where samples were dried to constant weight. The average of moisture and volatile matter contents was obtained to be 89% on wet basis.

2.2. LPSS drying equipment

A bench scale drying equipment was designed and built, its schematic is shown in Fig. 1. It consists of a cylindrical steel camera, (0.32 m in height, ID = 0.58 m, wall thickness = 0.1 m) externally covered by a 0.15 m thick isolation blanket, and containing an annular basket (0.06 m in height, OD = 0.33 m and ID = 0.21 m) made of wiremesh to contain the samples. A centrifugal fan, driven by a shaft connected to an electrical motor, enables the steam to flow through the sample as shown in Fig. 1.

The drying camera is provided with a pressure sensor Abs Sensyn, sensitivity = 1.5 mV/psia, span = 67.5 mV for a range of 0–1034 mbar, connected to a digital display. Temperature is measured and controlled by a LAE instrument, model MTW12T9RD/1, with an input range = −19.9 to 99.9 °C from a semiconductor sensor, and an output to a 5 A triac. This device controls the input power to an electrical resistance (1300 W) installed to heat steam. Accuracy is of ±0.1 °C from the set point.

The vapor–aroma mixture leaving the sample is extracted from the camera by the action of a vacuum system, Jet-1-automatic, Genser Scientific Instruments MFG. Co., D-8803 Rothenburg, OT, Germany (see Fig. 2). It goes through a condenser made of a 0.08 m in diameter and 0.30 m in height glass cylinder with a 0.008 m in diameter spiral tube built it, and fed with a countercurrent stream of tap water, where most of the water vapor is separated. The remaining exhaust gas is conducted to a cold trap made of a 0.03 m in diameter and 0.40 m in height glass cylinder, submerged in an ethyleneglycol–water mixture at −15 °C, where almost all compounds are also condensed.

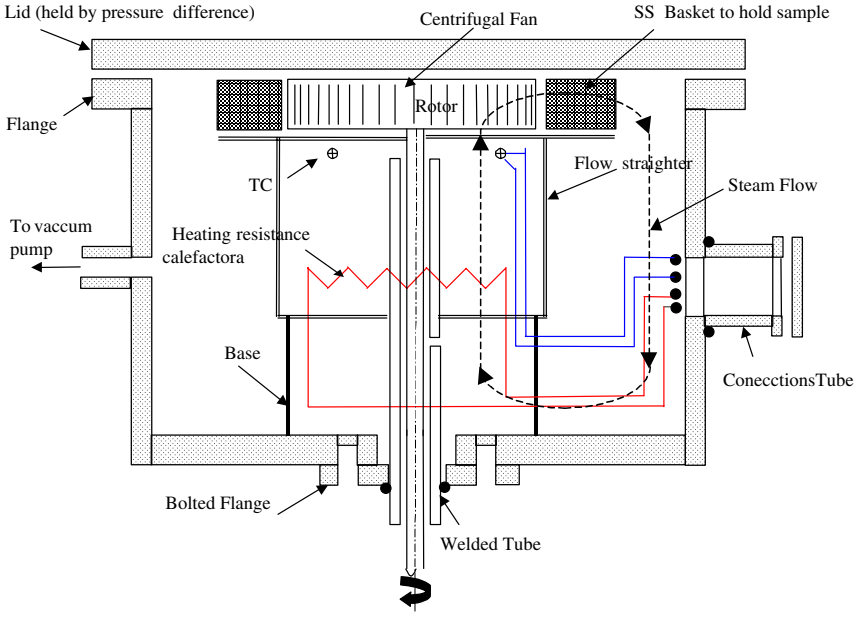

Fig. 1. Drying chamber cross-sectional view.

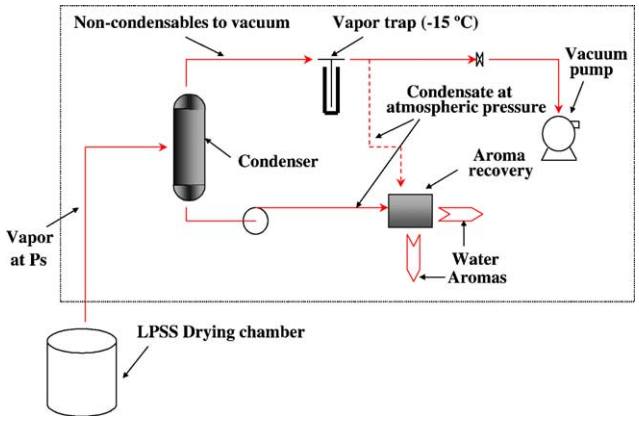

Fig. 2. Sketch of the experimental aroma recovery setup.

3. Experimental technique

3.1. LPSS drying

A sample of 300 g of fresh basil was dried by superheated steam at 50 °C, at a working pressure of 5066 Pa at which water equilibrium temperature is 33 °C. Steam was recirculated at a linear speed of 4.5 ms^{-1}. The operation was performed during 6 h. Once finished the volume of water and aromas trapped in the condenser and the cool trap was collected and measured. The liquid from the condenser as well as the dried product were stored at 4 °C for further extraction of volatile compounds.

3.2. Air-drying

Basil from the same source, though collected in the following season, was air-dried using a conventional convective drier, where air at 40, 50 and 60 °C was blown at an air velocity of 1.2 $m\,s^{-1}$. Temperature was automatically controlled at ±2 °C.

3.3. Extraction and concentration of volatiles

Samples of volatile compounds were obtained by the simultaneous distillation–extraction technique (SDE) (Likens & Nickerson, 1964; Shultz, Flath, Mon, Eggling, & Teranishi, 1977), with ethyl-ether as extraction agent. The same conditions were set for the extraction from fresh and dried samples and from the water solution captured in the condenser and the trap. Solid samples weighed 5 g and the liquid ones measured 100 ml. Samples of dehydrated and fresh basil were chopped previously. All samples were mixed with 200 ml of bidistilled water in a 1000 ml flask. The distillation–extraction process took 2 h at a pressure of 20,265 Pa.

The extract, once dried on $NaSO_4$, was concentrated by evaporating the solvent through a Vigreaux microcolumn to a volume of approximately 0.6 ml. Samples were finally stored in N_2 atmosphere at −18 °C.

3.4. GC conditions

A Hewlett Packard 4890D chromatograph (Agilent Technologies, USA) equipped with a fused silica capillary column CW 20M (50 m, ID 0.22 mm, film thickness 0.2 μm) was used for GC analysis. Conditions were as follows: sample size, 2 μl; initial column temperature, 46 °C, held for 2 min, followed by heating at a rate of 4 °C min^{-1} to 200 °C, held at this temperature for 20 min; injector temperature, 240 °C, split ratio, 1/50; FID detector temperature, 240 °C; carrier gas, H_2 (1.4 ml min^{-1}).

3.5. GC–MS conditions

For the identification of the volatiles compounds, some samples were submitted to GC–MS analysis on a Hewlett Packard 6890 gas-chromatograph (Agilent Technologies, USA), equipped with a 5972 A Series mass spectrometer, scan mode (ionization energy: 70 eV, mass range: 35–500 u). An amount of 5 μl of extract was analysed. The column was an HP5 fused silica (30 m, ID 0.25 mm, film thickness 0.25 μm); initial column temperature was 46 °C; held for 2 min, followed by heating at a rate of 4 °C min^{-1} to 200 °C, held at this temperature for 20 min. Injector temperature was 250 °C; detector temperature was 280 °C; carrier gas, He (1 ml min^{-1}).

3.6. Identification and quantitative analysis

The tentative identification of compounds was performed by comparison of mass spectra of each component with the corresponding standards in the data base, and using authentic standards when available, results were compared with those reported in literature (Backstroom-Sternberg et al., 1994; Díaz-Maroto et al., 2002; Lachowicz et al., 1996, 1997; Maarse, 1991; Simon et al., 1999; Yousif et al., 1999, among others).

The presence of the compounds selected for the study was confirmed by comparison of the retention times with those of pure reference standards. The quantitative determination was made by the internal standard method, with response factors calculated from pure reference compounds. The internal standard used was 2-heptanol.

All experiments were repeated three times and results were analysed statistically to obtain average values and sample standard deviations, as parameters of results reproducibility. Variance analysis (ANOVA, Origin v6) was used to evaluate the significance of the difference in the amounts of studied compounds. Values were considered significantly different when $p < 0.05$.

4. Results and discussion

4.1. Identification of volatile compounds

Fig. 3 shows a typical chromatogram obtained from a fresh basil sample, and Table 1 shows the percent composition of the identified compounds only, which turned out to be 23, as calculated from the respective chromatographic areas. They are mainly terpenes (61%), and in minor proportion aromatics (26%) and aliphatics (13%). Remaining peaks correspond to compounds that were separated but not identified.

The two major volatile compounds in our samples were linalool and 1,8-cineole, usually considered responsible for the basil typical aroma. Similar results were reported by Lachowicz et al. (1997) from a variety of species *O. basilicum* and by Díaz-Maroto et al. (2002). The area under the chromatographic peaks of most of the compounds is small and characteristic compounds like methyl cinnamate and methyl chavicol are present in very small quantities.

To study the impact of LPSS drying, eight compounds were selected: four of them are considered as character-impact of basil aroma, like 1,8-cineole, linalool, methyl chavicol and methyl cinnamate. The remaining four are also usually found in basil like α-pinene, limonene, *p*-cymene and hexadecane contribute to the aroma profile and standards were available. Camphene, though in larger proportion, was not selected because it has not been usually cited by other authors and it is felt that the selected compounds are enough for a proper analysis of the drying effects.

Table 1
Basil volatile components (Batch 1) (Percent by Gas Chromatography)

Peak[a]	Compound	Composition (%)
1	α-pinene[b]	0.42 ± 0.01
2	Camphene	3.97 ± 0.12
3	β-pinene	0.38 ± 0.01
4	α-terpinene	0.07 ± 0.00
5	Limonene[b]	0.16 ± 0.00
6	1,8-Cineole[b]	6.34 ± 0.08
7	γ-terpinene	0.41 ± 0.00
8	α-phellandrene	0.07 ± 0.00
9	*p*-Cymene[b]	0.05 ± 0.00
10	Terpinolene	0.09 ± 0.00
11	2-Heptanol (internal standard)	
12	Camphor	0.08 ± 0.00
13	Linalool[b]	28.89 ± 0.28
14	Bornyl acetate	0.19 ± 0.01
15	Linalyl acetate	1.00 ± 0.01
16	Hexadecane[b]	0.32 ± 0.02
17	α-caryophyllene	2.22 ± 0.03
18	β-caryophyllene	0.49 ± 0.00
19	Methyl chavicol[b]	0.56 ± 0.01
20	1-Octen-3-ol	0.58 ± 0.00
21	Octadecane	1.45 ± 0.01
22	Methyl cinnamate[b]	0.06 ± 0.00
23	Eugenol	0.31 ± 0.01
24	Methyl eugenol	0.13 ± 0.01

Values are given as mean ± standard deviation (*n*:3).
[a] The peak numbers correspond to the identification numbers in Fig. 3.
[b] Identification confirmed by injection of standard compound.

4.2. Effect of LPSS drying on volatile compounds

The Table 2 shows the composition of fresh basil, dried product and condensate from the condenser and the cold trap, in terms of the mentioned compounds with the results of the statistical analysis.

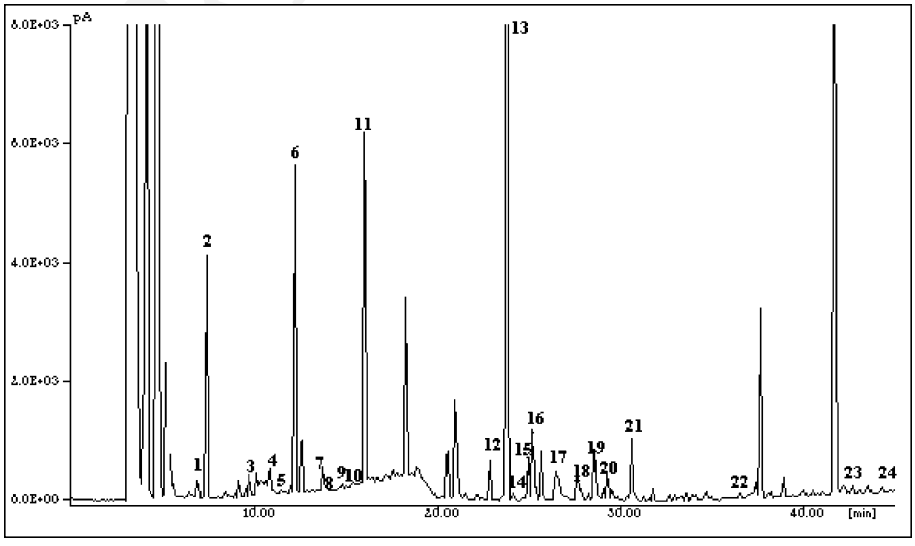

Fig. 3. Typical chromatogram of volatile compounds extracted from fresh basil from GC analysis (CW 20 M column).

Table 2
Volatile compounds contents in fresh product, LPSS dried product and extract from the condenser and the trap (Batch 1) [mg (kg of dry weight)$^{-1}$]

Compound	Fresh	LPSS dried material	In condensate
α-pinene	4.47 ± 0.304	3.13 ± 0.313	0.719 ± 0.033
Limonene	1.61 ± 0.111	1.37 ± 0.011	0.191 ± 0.001
1,8-Cineole	86.7 ± 3.33	79.6 ± 1.61	4.66 ± 0.048
p-Cymene	0.242 ± 0.026	0.179 ± 0.002	0.110 ± 0.002
Linalool	292.4 ± 29.7[a]	255.6 ± 11.3[a]	51.3 ± 0.428
Hexadecane	2.89 ± 0.066	1.06 ± 0.028	0.430 ± 0.007
Methyl chavicol	4.88 ± 0.051	3.98 ± 0.075	0.507 ± 0.016
Methyl cinnamate	0.530 ± 0.006[a]	0.443 ± 0.015[a]	0.095 ± 0.003

Values are given as mean ± standard deviation (n:3).
[a] Not significant differences ($p > 0.05$).

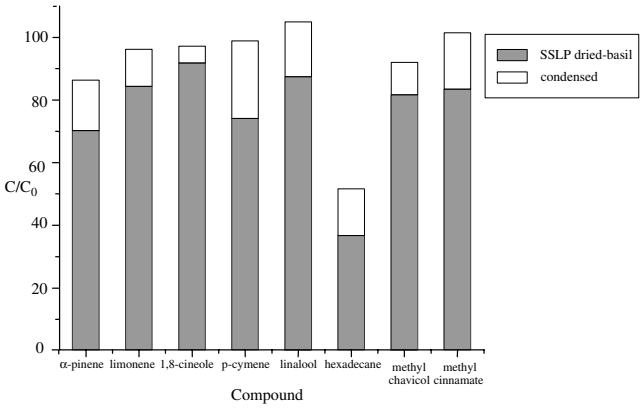

Fig. 4. Percent contents (relative to fresh product) of volatile compounds in LPSS dried basil and in condensed withdrawn vapor.

In all compounds, with the exception of linalool and methyl cinnamate, a statistically significant reduction has been observed as a consequence of the LPSS dehydration.

However, it is apparent that retention of all the compounds in the LPSS-dried product was higher than 70%, with the exception of hexadecane which was only 37%, which is a good result in terms of the product acceptance. The higher losses were those of not oxygenated terpenes, such as α-pinene (29.8%) and *p*-cymene (26%), while methyl cinnamate, 1,8-cineole, linalool and methyl chavicol, which are considered character-impact of basil aroma, were affected in lower proportion by drying process, keeping 83%, 80%, 88% and 82% of the original contents, respectively.

Fig. 4 shows the relative amounts of the mentioned compounds retained in the LPSS dried product and captured in the condenser and the trap, both expressed as percentage of the original contents in the basil sample.

The recovery of aroma compounds from the condensate show different efficiency for the different compounds. But considering that some of them are present in the original product at very low concentrations, handling and measuring errors are unavoidable.

It is worth to note that the total quantity of linalool and methyl cinnamate kept in the dried product plus that recovered from the condensate is higher than the original one (see Table 2). This result, tough unexpected, has been reported by other authors (Lachowicz et al., 1996; Yousif et al., 2000; Yousif et al., 1999; Venskutonis, 1997) and has been attributed to the hydrolysis of glycosides and the conversion of linalyl acetate into linalool during drying process.

On the other hand, compounds with losses higher than 10%, like hexadecane (48%) and α-pinene (13%), are not regarded as character-impact of basil aroma.

4.3. Effect of air-drying on volatile compounds

Table 3 shows the concentration (in mg kg^{-1} dry basis) of studied compounds in fresh basil and hot air-dried at different temperatures, as well as the results of the statistical analysis.

The difference in composition of the fresh vegetable used for air-drying and for LPSS drying is due to the fact that the samples, though from the same origin, are not the strictly the same. However, this is not a problem for the purpose of the work, as it is to compare the effect of each technique in terms of the final composition of the resulting product, that is, in what extent the original aroma profile is kept after undergoing different methods of drying. To identify the different samples, they are

Table 3
Volatile compounds contents in fresh basil and air-dried samples at 3 different temperatures (Batch 2) [mg (kg of dry weight)$^{-1}$]

Compound	Fresh	Air-dried 40 °C	Air-dried 50 °C	Air-dried 60 °C
α-pinene	11.0 ± 0.452[a]	6.96 ± 0.146[b]	5.20 ± 0.030[c]	5.76 ± 0.017[d]
Limonene	8.24 ± 0.337[a]	6.56 ± 0.05[b]	5.32 ± 0.004[b]	5.28 ± 0.006[b]
1,8-Cineole	998.5 ± 113.4[a]	396.9 ± 20.6[b]	283.4 ± 27.3[c]	347.9 ± 0.965[c]
p-Cymene	2.14 ± 0.105[a]	2.17 ± 0.053[a]	0.952 ± 0.021[b]	1.67 ± 0.226[c]
Linalool	2258.4 ± 141.5[a]	1162.0 ± 89.0[b]	1626.8 ± 133.0[c]	1583.2 ± 111.4[c]
Hexadecane	6.72 ± 0.679[a,b]	7.50 ± 0.131[a]	6.90 ± 0.117[b]	3.34 ± 0.061[c]
Methyl chavicol	71.3 ± 5.89[a]	16.4 ± 0.366[b]	18.4 ± 0.191[b]	110.6 ± 5.90[c]
Methyl cinnamate	1.56 ± 0.003[a]	0.851 ± 0.008[b]	0.639 ± 0.059[c]	0.123 ± 0.013[d]

Values are given as mean ± standard deviation (n:3).
Different letters (a, b, c, d) in the same row indicate statistical differences according to ANOVA analysis ($p < 0.05$).

named "batch 1" and "batch 2" respectively, keeping the denomination "sample" for those of the same batch submitted to different treatments.

Results show marked differences between the composition of the dried product as compared with the fresh one, and, exception made of the above mentioned methyl chavicol (dried at 60 °C), there is a decrease in the content of the studied compounds.

As the air temperature increases, the differences are greater, though the effect is not the same for all components and no trend can be established.

1,8-Cineole underwent a pronounced decrease, in the order of 65% for all of these air-drying temperatures. The amount of methyl cinnamate showed a significant decrease (45–60%) from original levels present in the fresh samples when basil was air-dried at 40–50 °C respectively, and it almost disappeared at 60 °C (8%). Linalool, other character-impact compound was also affected by air-dried.

The observed increase in the content of methyl chavicol upon air-drying (60 °C) was reported by Yousif et al. (1999).

The percentage of retention of most of the analysed compounds is higher for the LPSS dried samples than in those dried by hot air, at all temperatures. Moreover, and what is more relevant, the percent distribution of the retained compounds is similar to that of the fresh samples (see Fig. 5), while air-dried samples show a different percent composition (see Fig. 6). Since the hedonic appreciation of an aroma results from the simultaneous and combined perception of the individual components of the mixture, it is expected that the dry product exhibiting the same distribution profile than the fresh one, should have the same degree of acceptance by the consumer.

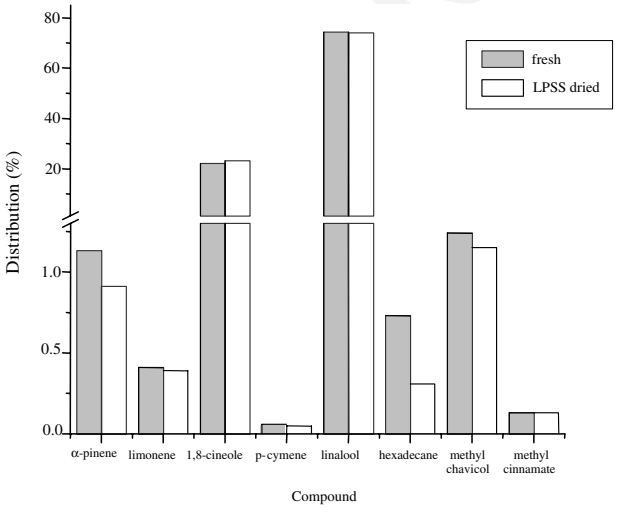

Fig. 5. Composition profile of fresh basil and LPSS dried sample.

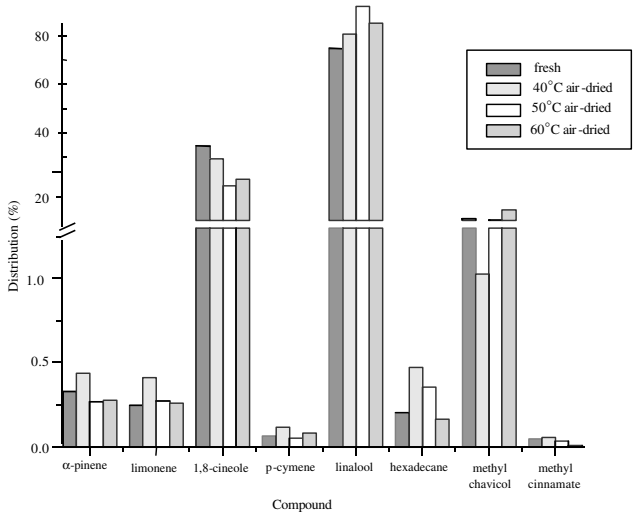

Fig. 6. Composition profile of fresh basil and air-dried samples at three different temperatures.

5. Conclusions

The technique for extraction of volatile compounds used in this work (SDE) proved to be efficient in separating components regarded as characteristics of basil aroma mentioned in literature. The extract obtained was constituted mainly by terpenes.

The use of low pressure superheated steam as drying agent rendered a product with a higher retention of the original volatiles compounds and a lower modification of the percent composition.

The LPSS drying technique allows the recovery of a substantial amount of the aroma compounds withdrawn from the product, increasing the economical advantages of the new method.

Regarding air-drying, it was observed a significant reduction in the content of most of the analysed compounds in the product. Also, changes in air temperature have different effect on different compounds: while concentration of some increase as temperature rise, other decrease.

From an overall analysis of the results obtained, it can be concluded that LPSS drying is a method that guarantees the preservation of main basil aroma compounds.

References

Backstroom-Sternberg, M., Stephen, J. A., Duke, & Wain, K. (1994). *The EthnobotanycalDatabase*. Available: http://probe.nalusda.gov:8300/cgin/browse/ethnobotdb. (ACEDB versión 4.3)—data version July 1994.

Díaz-Maroto, M. C., Pérez-Coello, M. S., & Cabezudo, M. D. (2002). Supercritical carbon dioxide extraction of volatiles from spices. Comparison with simultaneous distillation–extraction. *Journal of Chromatography A, 947*, 23–29.

Elustondo, D. (2001). *Secado de alimentos con vapor sobrecalentado a baja presión.* Ph.D. Thesis, PLAPIQUI (UNS–CONICET).

Iwotech Limited. (1993). Technical Bulletins on Moldrup Process, Iwotech Limited. Brande, Denmark.

Huopalahti, R., Kesalahti, E., & Linko, R. (1985). Effect of hot air and freeze drying on the volatiles compounds of dill (*Anethum graveolens* L.) herb. *Journal of Agricultural Science, 57*, 133.

Jerkovic, I., Mastelic, J., & Milos, M. (2001). The effect of air-drying on glycosidically boun volatiles from seasonally collected oregano (*Origanum vulgare* ssp. *hirtum*) from Croacia. *Nahrung, 45*(1), S47–49.

Kumar, P., & Mujumdar, A. S. (1990). *Superheated steam drying: A bibliography*. London: M. Dekker (p. 195, 205).

Lachowicz, K., Jones, G., Briggs, D., Bienvenu, F., Palmer, M., Mishra, V., & Hunter, M. (1996). Characteristics of essential oil from basil (*Ocimum basilicum* L.) grown in Australia. *Journal of Agricultural and Food Chemistry, 44*, 877–881.

Lachowicz, K., Jones, G., Briggs, D., Bienvenu, F., Palmer, M., Mishra, V., & Hunter, M. (1997). Characteristics of plants and plant extracts from five varieties of basil (*Ocimum basilicum* L.) grown in Australia. *Journal of Agricultural and Food Chemistry, 45*, 2660–2665.

Likens, S. T., & Nickerson, G. B. (1964). Detection of certain hop oil constituents in Brewing Products. *A.S.B.C. Proceedings,* 5–13.

H. Maarse (Ed.) (1991). *Volatile compounds in foods and beverages* (1st ed.). 1991.

Mujumdar, A. S. (1980). *CEA Report*. 817U 671, Montreal, Canada, 1980.

Nomura, T., & Hyodo, T. (1984). Behavior of inversion point temperature and new applications of superheated vapor drying. *Proceedings of the fourth international drying symposium* (Vol. 2). Kyoto, Japan, July 9–12, 1984.

Shultz, T., Flath, R., Mon, T., Eggling, S., & Teranishi, R. (1977). Isolation of volatile components from a model system. *Journal of Agricultural and Food Chemistry, 25*, 446–449.

Simon, J. E., Morales, M. R., Phippen, W. B., Vieira, R. F., & Hao, Z. (1999). Basil a source of aroma compounds and popular culinary and ornamental herb. In J. Janick (Ed.), *Perspectives on new crops and new uses* (pp. 499–505). Alexandria, VA: ASHS.

Svensson, C. (1980). Steam drying of pulp. *Drying '80* (Vol. 2). 1980. Section V: Drying of pulp and paper. In A. Mujumdar (Ed.), *Proc. of the second international symposium.* (p. 301, 307).

Yoshida, T., & Hyodo, T. (1960). Superheated vapor speeds drying of foods. *Food Engineering, 38*, 86–87.

Yousif, A. N., Durance, T. D., Scaman, C. H., & Girard, B. (2000). Headspace volatiles and physical characteristics of vacuum-microwave, air and freeze-dried Oregano (Lippia berlandieri Schauer). *Journal of Food Science, 65*(6), 926–930.

Yousif, A. N., Scaman, C. H., Durance, T. D., & Girard, B. (1999). Flavor volatiles and physical properties of vacuum-microwave and air-dried sweet basil (*Ocimum basilicum* L.). *Journal of Agricultural and Food Chemistry, 47*, 4777–4781.

Venskutonis, P. R. (1997). Effect of drying on the volatile constituents of thyme (*Thymus vulgaris* L.) and sage (*Salvia officinalis* L.). *Food Chemistry, 2*, 219–227.