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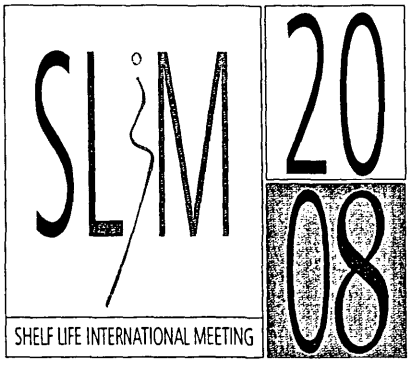

SLIM 2008

Shelf-life International Meeting

Ischia, June 25-27th 2008

Edited by

GIOVANNA G. BUONOCORE & ELENA TORRIERI

Special Issue

ITALIAN JOURNAL
OF
FOOD SCIENCE

CHIRIOTTI EDITORI

This Special Issue of the Italian Journal of Food Science collects the presentations given at the "SLIM 2008, Shelf Life International Meeting" organized by GSICA, National Research Council – IMCB, University of Naples – DSA and DIMP, held at Ischia on June 25-27th 2008.
These papers were reviewed by the Scientific Committee of the congress before their presentation but they did not undergo the conventional reviewing system of the Italian Journal of Food Science.

Chiriotti Editori S.A.S. - Pinerolo - Italy

ISSN 1120-1770

ITALIAN JOURNAL OF FOOD SCIENCE

(RIVISTA ITALIANA DI SCIENZA DEGLI ALIMENTI)

Property of the University of Perugia
Official Journal of the Italian Society of Food Science and Technology
Società Italiana di Scienze e Tecnologie Alimentari (S.I.S.T.Al)
Initially supported in part by the Italian Research Council (CNR) - Rome - Italy
Recognised as a "Journal of High Cultural Level"
by the Ministry of Cultural Heritage - Rome - Italy

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E-mail: info@chiriottieditori.it - URL: www.chiriottieditori.it

Aim: The Italian Journal of Food Science is an international journal publishing original, basic and applied papers, reviews, short communications, surveys and opinions in food science (chemistry, analysis, microbiology), food technology (engineering, processing) and related areas (nutrition, safety, toxicity, physiology, dietetics, economics, etc.). Upon request and free of charge, announcements of congresses, presentations of research institutes, books and proceedings may also be published in a special "News" section.

Review Policy:
The Advisory Board with the Editor-in-Chief will select submitted manuscripts in relationship to their innovative and original content. Referees will be selected from the Advisory Board and/or qualified Italian or foreign scientists. Acceptance of a paper rests with the referees.

Frequency: Quarterly - One volume in four issues. Guide for Authors is published in each number and annual indices are published in number 4 of each volume.

Impact Factor: 0.518 published in the 2007 Journal of Citation Reports, Institute for Scientific Information

Subscription Rate: 2009: Volume XXI

PDF version	€ 40.00
Ordinary	€ 150.00
Supporting	€ 1,000.00

IJFS is abstracted/indexed in: Chemical Abstracts Service (USA); Foods Adlibra Publ. (USA); Gialine - Ensia (F); Institut Information Sci. Acad. Sciences (Russia); Institute for Scientific Information; CurrentContents®/AB&ES; SciSearch® (USA-GB); Int. Food Information Service - IFIS (D); Int. Food Information Service - IFIS (UK); EBSCO Publishing; Index Copernicus Journal Master List (PL).

IJFS has a page charge of € 20.00 up to 5 pages; extra pages are € 30.00.
Reprints (100) will be sent free of charge.

INFLUENCE OF STORAGE TIME ON THE COMPOSITION OF VOLATILE COMPOUNDS OF AIR DRIED, FROZEN AND FREEZE DRIED THYME AND ROSEMARY CULTIVATED IN SARDINIA

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ABSTRACT

This paper deals with the effect of different stabilizing techniques on the evolution of the volatiles in rosemary (*Rosmarinus officinalis* L) and thyme (*Thymus officinalis* L.) cultivated in Sardinia during nine months of storage. Fresh leaves were collected and soon divided in four batches, which were subjected to hydro distillation and GC-MS analysis, the first batch as fresh, the second one after drying in a laboratory pilot dryer, the third after freezing in a forced air freezer and the fourth after freeze drying in a laboratory freeze dryer.

All the samples were adequately packaged and stored. Samples for analysis were taken at 3 months intervals. The fresh, stabilised and stored plant material were hydro distilled for 4 hours using a Clevenger-type. The oils were analysed in duplicate by gas chromatography, using a flame ionization detector. Qualitative analysis was done by GC/Mass and mass units were monitored from 10 to 450 at 70 eV.

Results of the evolution of volatile compounds of the differently samples seem to evidence that the best way to stabilize the herbs is freezing.

Key words: *Rosmarinus officinalis* L.; *Thymus officinalis* L.; essential oils; stabilisation; storage.

INTRODUCTION

The shelf life of spices is traditionally extended by drying. Fresh herbs, due to their high water content, undergo micro-organism growth and adverse biochemical reactions. On the other hand drying may result in a lot of physical and chemical alterations. Air and oven-dehydration are the main methods used to stabilize spices. During oven drying, in general, losses of volatile compounds are directly dependent on the temperature and time used. Anyway, other unit operations that may be proposed are freezing and freeze-drying.

Thyme and rosemary grow both wild in the Mediterranean basin and they are very much appreciated for their aromatic, antimicrobial and antioxidant properties (Dorman and Deans, 2000; Nguyen *et al.*, 2000). The effect of mechanical air drying on the volatiles of *T. officinalis* and *R. officinalis* have been extensively reported (Blanco *et al.*, 2002; Fadel and El-Massry, 2000; Raghavan *et al.*, 1995; Venskutonis, 1997). However, we have no knowledge about changes during storage on volatile composition of air dried, freeze dried or frozen samples of these two species cultivated in Sardinia.

The aim of this paper is to verify the effects of the above cited stabilizing techniques on the evolution of the volatiles in rosemary (*Rosmarinus officinalis* L) and thyme (*Thymus officinalis* L.) cultivated in Sardinia during nine months in storage.

MATERIALS AND METHODS

Fresh leaves were collected and soon divided in four batches, which were subjected to hydro distillation and GC-MS analysis, the first batch as fresh, the second one after drying in a laboratory pilot dryer, the third after freezing in a forced air freezer and the fourth after freeze drying in a laboratory freeze dryer. Drying was done at 45°C and 1250 m^3/h for thyme and 38°C and 300 m^3/h for rosemary. Freeze drying was done with an Edwards Modulyo freeze dryer at a condenser temperature of -52°C, shelf temperature of 20°C and pressure below 10 Pa for 24 hours. The oven dried samples were packaged both under vacuum inside a polypropylene film and at atmospheric pressure inside polyethylene bags, while freeze dried herbs were packaged in an aluminate film under vacuum; both samples were stored in the dark at 20°C. Frozen herbs were packaged in polyethylene bags and stored in the dark at -18°C. Samples for analysis were taken at 3 months intervals up to nine months. The fresh, stabilised and stored plant material were hydro distilled for 4 hours using a Clevenger-type and the oils were stored at –20°C before analyses, which were carried out on two replicates of each sample by gas chromatography, using a flame ionization detector. Qualitative analysis was done by GC/Mass and mass units were monitored from 10 to 450 at 70 eV, as reported in a previous paper (Piga *et al.*, 2007).

RESULTS AND DISCUSSION

Rosemary essential oil yield showed the most significant reduction in freeze dried samples, while frozen herbs evidenced the least reduction. Results of GC/MS analyses of frozen and freeze dried samples showed that borneol, γ-terpinene and mirtenol had the major losses during storage, (myrtenol disappeared after 9 months in storage on freeze-dried samples). Dried samples evidenced a major decrease also in β-pinene, terpinolene, and methyl charvacrol.

Thyme essential oil yield showed the most significant reduction in air dried samples, while frozen herbs evidenced the least reduction. Results of GC/MS analyses of frozen and freeze dried samples showed that terpinel-4-ol, thymol methyl ether, carvacrol methyl ether and caryophyllene ether had the major losses during storage, (carvacrol methyl ether and caryophyllene ether disappeared after 9 months in storage). Dried samples evidenced a major decrease also in α-thuyene, octen-3-ol, myrcene, γ-terpinene and carvacrol.

Packaging material of dried herbs resulted in contrasting results.

From these preliminary results we can state that the best stabilising unit operation is freezing for both herbs.

ACKNOWLEDGEMENTS:

The authors thank the Regione Autonoma della Sardegna for financial support (LR 14.11.2000 n. 21 Art. 11 "Ricerca e sviluppo", (attività di studio e ricerca aventi la finalità generale dello sviluppo del settore agricolo), project title "Effetto di diversi interventi tecnologici di stabilizzazione di piante officinali tipiche della Sardegna, per l'ottenimento di principi attivi e la produzione di estratti di alta qualità".

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