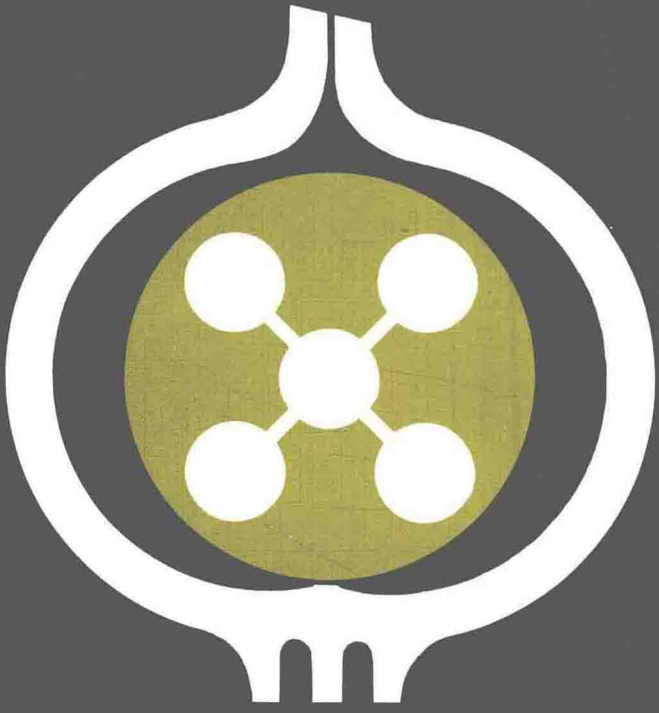
Developments in Food Science

35

TRENDS IN FLAVOUR RESEARCH

Edited by
H. MAARSE and D.G. VAN DER HEIJ



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DEVELOPMENTS IN FOOD SCIENCE 35

TRENDS IN FLAVOUR RESEARCH

Proceedings of the 7th Weurman Flavour Research Symposium,
Noordwijkerhout, The Netherlands, 15-18 June 1993

Edited by

H. MAARSE and D. G. VAN DER HEIJ
TNO - Nutrition and Food Research, P.O. Box 360, 3700 AJ Zeist,
The Netherlands

ELSEVIER
Amsterdam – London – New York – Tokyo 1994

ELSEVIER SCIENCE B.V.
Sara Burgerhartstraat 25
P.O. Box 211, 1000 AE Amsterdam, The Netherlands

Library of Congress Cataloging-in-Publication Data

Weurman Flavour Research Symposium (7th : 1993 : Noordwijkerhout, Netherlands)
Trends in flavour research : proceedings of the 7th Weurman Flavour Research Symposium, Noordwijkerhout, The Netherlands, 15-18 June, 1993 / edited by H. Maarse and D.G. Van der Heij.
p. cm. -- (Developments in food science ; 35)
Includes bibliographical references and index.
ISBN 0-444-81587-2
1. Flavoring essences--Congresses. I. Maarse, H. II. Heij, D. G. van der. III. Title. IV. Series.
TP418.W48 1993
664'.07--dc20 93-50139
CIP

ISBN: 0-444-81587-2

This book is printed on acid-free paper.

Printed in The Netherlands

DEVELOPMENTS IN FOOD SCIENCE 35

TRENDS IN FLAVOUR RESEARCH

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Trends in Flavour Research. Proceedings of the 7th Weurman Flavour Research Symposium, Noordwijkerhout, The Netherlands, 15-18 June 1993

Preface

This book reflects the lectures, posters and workshops of the 7th Weurman Flavour Research Symposium held 15–18 June 1993 at Noordwijkerhout, Netherlands. The symposium was the seventh of its kind. The first one was organized in 1975 on the initiative of the late Dr C. Weurman, then head of the Aroma Department of the Central Institute for Nutrition and Food Research (CIVO), now named TNO Nutrition and Food Research. Dr Weurman died in January 1975, some months before the symposium. In recognition of his great contribution to flavour research it was decided unanimously during the symposium to name future symposia after him. Subsequent symposia have been held at Norwich, England (1978), Munich, Germany (1981), Dourdan, France (1984), Oslo, Norway (1987) and Geneva, Switzerland (1990). At the end of the 1993 symposium it was decided to have the 8th symposium organized in the United Kingdom in 1996.

The Weurman symposia differ from most other ones in that attendance is only by invitation based on proposals for active participation. The number of participants is limited to 100 to facilitate informal communication. At least one tenth of them should be young scientists; the 7th Symposium was attended by 85 scientists including 10 Ph.D. students.

Manuscripts for these proceedings have been submitted both as paper copy and on diskette to enable careful editing. This procedure made it possible to obtain a uniform style and format and to use IUPAC chemical nomenclature throughout the book. The contributions are grouped under the main topics of the symposium. Under each topic the following items can be found: full papers and short contributions based on lectures read at the symposium, contributions based on the posters presented in the poster sessions, and (in some cases) a workshop report. The book is concluded with author and subject indexes aimed at improving the accessibility of these proceedings.

We are grateful to all authors whose kind co-operation and active support have smoothed our editorial task and helped us complete the job in four months' time.

Zeist, November 1993
Henk Maarse and Dirk G. van der Heij

Seventh Weurman Flavour Research Symposium
Noordwijkerhout, Netherlands, 15–18 June 1993

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Flavour release

H. Maarse and D.G. van der Heij (Eds.), Trends in Flavour Research

Methodology for measuring volatile profiles in the mouth and nose during eating

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Abstract

This paper describes the development of techniques to measure the volatile profile as it is sensed by the human nose during eating. The advantages of this method are discussed with reference to other methods of aroma analysis and with reference to the idea that sensory and instrumental data need to be considered together if we are to gain meaningful data on aromas from foods. The various approaches to sampling aroma from the nose (nosespace) are considered and data on the sensitivity and reproducibility of a system measuring the flavour release from mint-flavoured sweets are presented. Work on applying this technique to whole foods with lower levels of aroma, where some of the aroma components are generated during eating, is presented along with potential applications of the technique in the food industry.

Introduction

Many methods have been developed for analysing the aroma from foods over the past twenty years. Some methods have been designed on the basis of quantitative extraction of volatiles, others have tried to link instrumental analysis to sensory properties of foods. Currently most attention is focused on the combined instrumental/sensory approach. The analytical techniques are designed to measure two types of volatile profile, viz. the 'base' profile and the headspace profile.

The base profile represents the total volatile composition in a food while the headspace profile measures which volatiles are present in the air above a food and their concentration in that air. The next logical profile to analyse is the volatile profile that is sensed by the nose. This may be very different from the headspace profile in the same way that headspace sometimes differs from the base profile. The term 'nosespace' has been used to define the volatile profile existing in the nose and techniques to measure this profile have been developed (1). In the development process, it was important to consider whether the technique itself would alter or affect the profile. If the technique was to be used in an attempt to correlate sensory and instrumental data, it was also important to establish the reproducibility of the method, an aspect that has received little attention thus far. Before describing the potential methods for measuring

nosespace, it is useful to set the scene by discussing the limitations of methods for measuring base profile and headspace profile and the dynamic nature of some volatile profiles.

Limitations of volatile analyses

The base profile is usually analysed by extracting the food material with a solvent. To ensure that volatiles are extracted into the organic phase, a combined steam and organic solvent distillation is normally carried out using simultaneous distillation extraction (SDE) in a specialised apparatus like Likens-Nickersen. The disadvantages of the method as a general extraction technique are well known. Since samples must be steam-distilled, cooking takes place and the technique has limited applications for fresh samples. In addition, artefacts can be formed due to the high temperature and long times needed for distillation. Various modifications to the original process have been suggested using antioxidants or distilling under vacuum to improve the technique but care is still needed when interpreting the results obtained from analysis of these steam-distilled extracts. There does seem to be a suggestion that some workers judge the effectiveness of the extraction procedure on the number of peaks that appear on the gas chromatogram, with more peaks indicating a better extraction. It might equally indicate that more breakdown and artefact formation is taking place.

Extraction using solvents at very low temperatures may be more applicable to fresh food materials, and more work is needed to examine the merits of this approach. The combination of very low temperatures, exclusion of light and the use of antioxidants may minimise any changes and provide samples which truly represent base profile. Extraction with liquid carbon dioxide is another alternative but there are some reservations about the ability of this solvent to extract all compounds quantitatively.

While reliable analysis of base profile may be difficult, the analysis of headspace presents additional challenges. The low levels of volatiles in air require a concentration procedure and, if care is not taken, this too can distort the profile that is analysed. There are a number of ways in which concentration is achieved using, for example, purge-and-trap techniques and static or dynamic headspace collection. Trapping may be on adsorbents like charcoal or Tenax or by cryogenic means. The headspace methods give volatile profiles which are better related to the profiles experienced by human subjects when they smell food and are widely used because of this similarity. There are still some potential problems with the technique, particularly in deciding whether static or dynamic headspace is more relevant or when adsorbents are used for trapping volatiles. It is unlikely that human subjects experience a headspace that is in equilibrium with the food (which is the aim of static headspace) and the work of Wyllie and coworkers (2) showed that, in dynamic headspace, the profile varied according to the flow of purging gas used. It is also acknowledged that none of the adsorbents is perfect in trapping all volatile compounds with the same efficiency and they all exhibit some selectivity (3). The headspace techniques therefore do not provide absolute profiles but they are capable of providing a 'snapshot' of the profile under defined conditions.

Although many data on headspace profiles have been published, there are just few published data on the variability of headspace profiles as most workers use headspace qualitatively. If headspace profiles are to be compared with sensory data, then some measure of their reliability and reproducibility is needed. It is interesting that, in the proceedings of the previous Weurman Symposium, few authors indicated the number of replicates used to obtain their data points nor the standard deviations of these replicates. An exception was the paper of Larsen and Poll (4) who suggested that a CV of 15% was an acceptable value for variation in aroma analyses. In our laboratory, the best results we have obtained with headspace sampling show a mean coefficient of variation (CV = (standard deviation × 100)/mean) of 8 to 10% while 12–16% is normal. Values above this are considered unsatisfactory and sometimes indicate that the volatile compounds are labile and are undergoing chemical changes (degradation or irreversible adsorption) either during sampling or during analysis. Used carefully however, headspace analysis can provide reproducible 'snapshots' of the volatile profile above foods.

In the analysis of aromas described above, it is clear that extraction and concentration are usually prerequisites to the actual analysis of the volatiles themselves. Indeed, it is true to say that extraction and concentration are the difficult procedures compared to chromatographic analysis of the volatile extract which is normally considered to be straightforward. However, Block (5) recently reminded us that gas chromatography-mass spectrometry (GC-MS) of some samples (particularly sulphur compounds) can produce changes in the volatile profile and the potential for producing artefacts in high-temperature injection systems should not be forgotten. Ideally, cold on-column GC systems are preferred to hot injection techniques.

Changes in volatile profiles with time

The experimental design of procedures which are to analyse the sensory properties and volatile profile of foods must take into account the dynamic nature of many volatiles. The best known examples are fresh plant foods where rapid changes can take place as tissue is stressed or macerated. Fisher et al. (6), for example, showed that extraction of oils from the oil bodies of marjoram gave volatile profiles that were simple as compared to those obtained from homogenised tissue. These differences were due to the labile nature of the sabinene derivatives which reacted rapidly to give a range of compounds. Fruits like tomato and cucumber produce part of their flavour profile when the tissue is masticated in the mouth (7) and the volatile profile changes rapidly as the lipoxygenase enzyme degrades the fatty acid substrate. While these changes involve rapid changes in fresh foods (typically seconds to minutes), some processed foods also undergo changes in the volatile profile. With baked foods, there is a slow change in volatile profile with time (days to months) and the fresh baked aroma disappears and may be replaced after several months with a rancid odour. Because the changes in baked goods like biscuits are so slow, they are unlikely to cause problems with volatile sampling as people are aware of the different aromas and will not sample biscuits after three months and expect to obtain a volatile profile that is representative