Chemical and Functional Properties of Food Components Series

Methods of Analysis of Food Components and Additives

Second Edition

EDITED BY

Semih Ötleş

CRC Press
Taylor & Francis Group

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CRC Press
Taylor & Francis Group
Boca Raton London New York

CRC Press is an imprint of the
Taylor & Francis Group, an **informa** business

CRC Press
Taylor & Francis Group
6000 Broken Sound Parkway NW, Suite 300
Boca Raton, FL 33487-2742

Printed in the United States of America on acid-free paper
Version Date: 20110801

International Standard Book Number: 978-1-4398-1552-6 (Hardback)

Library of Congress Cataloging-in-Publication Data

Methods of analysis of food components and additives / editor, Semih Otles. -- 2nd ed.
p. cm. -- (Chemical & functional properties of food components)
Includes bibliographical references and index.
ISBN 978-1-4398-1552-6 (alk. paper)
1. Food additives--Analysis--Methodology. 2. Food additives--Analysis--Standards. 3. Food--Analysis--Methodology. I. Ötles, Semih.

TP455.M48 2012
664'.06--dc23 2011027105

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Preface

The first edition of this book *Methods of Analysis of Food Components and Additives* was published by CRC Press/Taylor & Francis Group in 2005. The book quickly became a highly successful and popular educational and scientific tool among academicians, students, and staff working in private companies and governmental institutions worldwide. The readers valued this book as good teaching material and a useful reference. Thus, it is of no surprise that the book enjoyed a high ranking on the CRC Press' book list. It has turned out that, as methodological and instrumental progress occurs, new applications materialize, which, in turn, provide a driving force for further methodological and instrumental developments. Advances in instrumentation and applied instrumental analysis methods have allowed scientists concerned with food and beverage quality, labeling, compliance, and safety to meet ever-increasing analytical demands.

The second edition of the book is an extended and updated version of the original. The new edition follows the same format as the first edition and covers topics such as selection of techniques, statistical assessments, rapid microbiological techniques, and demonstration of the applications of chemical, physical, microbiological, sensorial, and instrumental novel analysis to food components and additives such as proteins, peptides and amino acids, lipids, trace elements, vitamins, carotenoids, chlorophylls, polyphenols, drinking water, food allergens, genetically modified components, pesticide residues, pollutants, chemical preservatives, and radioactive components in foods. New additions include three chapters on analytical quality assurance, carbohydrates, and natural toxins. The second edition includes important developments in analytical quality assurance and the analysis of carbohydrates and natural toxins in foods. The chapter on the analysis of natural toxins is an especially welcome addition, a welcomed addition as it covers various methods of analysis of natural toxins, which is very important for food quality control related to toxicology. Information provided in the chapter "Analysis of Carbohydrates in Foods" would be equally valuable for scientists, students, and staff working in the field of food quality control. The text in the remaining chapters has been updated and is supported by numerous examples.

As the editor of this book, I would like to express my sincere thanks to all the authors for their excellent contributions to this book.

Semih Ötleş

Acknowledgments

I would like to thank the contributors for all the hard work they have put in and the valuable contributions they have made to the various chapters of the book. I want to thank the Taylor & Francis Group/CRC Press team for their help in production of this book and to express my sincere gratitude to Stephen M. Zollo, Taylor & Francis Group, for his help in publishing the book. Finally, special thanks to my wife, Sema Ötleş, for her patience during the preparation and publication steps of this book.

Semih Ötleş

Editor

A native of Izmir, Turkey, Professor Ötleş obtained his B.Sc. degree from the Department of Food Engineering (Ege University) in 1980. During his assistantship at Ege University, in 1985, he received an M.S. in Food Chemistry, and in 1989, after completing his research thesis on the instrumental analysis and chemistry of vitamins in foods, he received a Ph.D. in Food Chemistry from Ege University. In 1991–1992, he completed postdoctoral training including OECD—Postdoctoral Fellowship—in the Research Center Melle at Gent University, Belgium. Later, he joined the Department of Food Engineering at Ege University as a scientist of Food Chemistry and was promoted as Associate Professor in 1993 and as Professor in 2000. From 1996–1998, he was Deputy Director at the Ege Vocational School of Higher Studies. He was also Vice Dean at Engineering Faculty from 2003–2009 and head of the Department of Nutrition and Dietetics from 2008–2011.

The research activities of Professor Ötleş have been focused on instrumental analysis of foods. Ötleş began a series of projects on separation and analysis using HPLC techniques, first for analysis of vitamins in foods, then proteins, carbohydrates, and, most recently, carotenoids. Other activities span the fields of GC, GC/MS analysis, soy chemistry, aromatics, medical and functional foods, and nutraceutical chemistry. This includes multiresidue analysis of various foods, n-3 fatty acids in fish oils, and medical and functional foods.

Professor Ötleş is the author or coauthor of more than 250 publications (technical papers, book chapters, and books) and has presented some seminars in these areas. He is a member of several scientific societies, associations, and organizations, including the Asian Pacific Organization for Cancer Prevention and the International Society of Food Physicists. He is a member of the steering committee of APOCP local scientific bureau and a Turkish representative of ISFP and has organized international congresses on diet/cancer and food physics.

Professor Ötleş is a member of the Editorial Advisory Board of *Asian Pacific Journal of Cancer Prevention* (APJCP), *Food Science and Technology Abstracts* (FSTA) of International Food Information Service (IFIS), *Current Topics in Nutraceutical Research*, *Electronic Journal of Environmental, Agricultural and Food Chemistry* (EJEAFChe), *Newsline* (IUFoST, Corr.), *Journal of Oil, Soap, Cosmetics*, *Tr. World Food*, *Tr. Food Science and Technology*, *Pakistan Journal of Nutrition*, *Journal of Food Technology*, *Academic Food*, *Australian Journal of Science and Technology*, *Journal of Oil Research Institute*, *Journal of Food Safety*, *Key Food Magazine*, *Advances in Food Sciences* and is a referee/reviewer for *AOAC International*, *Journal of Experimental Marine Biology and Ecology*, *Journal of Medical Foods*, *die Nahrung*, *Journal of Alternative and Complementary Medicine*, *the Analyst*, *Journal of Scientific and Industrial Research*, *the Science of Total Environment*, *Current Microbiology*, *Journal of Essential Oil Bearing Plants*, and *Journal of Agricultural and Food Chemistry*.

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Contents

1 Choosing Techniques for Analysis of Food

Michael H. Tunick

CONTENTS

1.1 CLASSIFICATION OF TECHNIQUES

Any technique selected for food analysis depends on what the scientist is looking for, and there is a host of food properties from which to choose. A lengthy list of the physical properties of food was assembled by Jowitt.[1] Using that list as a starting point, a classification of food properties was created by Rahman[2] and revised by Rahman and McCarthy.[3] In reorganized form, it is as follows:

- Mechanical properties (mass–volume–area related, morphometric and structural, rheological, and surface)
- Other physical and physicochemical properties (electromagnetic, mass transfer, physicochemical constants, thermal, and thermodynamic)
- Kinetic properties (quality kinetic constants and microbial growth–decline–death kinetic constants)
- Sensory properties (color and appearance, odor, sound, tactile, taste, and texture)
- Health properties (functional properties, medical properties, nutritional composition, toxicity, and unbalanced intake)

Health properties are beyond the scope of this book, and sensory analysis will be addressed in Chapter 12. Compositional analyses, which were not included among physical properties in the classifications above, are covered in many of the remaining chapters.

1.2 CONSIDERATIONS

Scientists decide which characteristics are important for a particular food and which techniques should be used to examine them. The principles for method selection include the following criteria:

- Ability to conduct analysis (sample size, reagents, instruments, cost, and possible destruction of sample)
- Fundamental characteristics (precision, accuracy, sensitivity, specificity, detection limit, and reproducibility)
- Personnel concerns (safety, simplicity, and speed)
- Method status (official or in-house method)

Several techniques may fit these criteria. The remainder of this chapter will deal with different properties of food and the choices commonly available for analysis.

1.3 MECHANICAL PROPERTIES

The mechanical properties of a food deal with changes in its structure and behavior when force is applied. The Rahman and McCarthy classification[3] separated morphometric and structural properties, but these will be combined here since the same techniques are used for analyzing both.

1.3.1 Acoustic

Acoustic measurements are used to evaluate crispness, which has been claimed to be the most important food attribute affecting acceptability by consumers.[4] According to a review by Duizer,[5] the two acoustical measurements used for examining crispness and crunchiness are measurement of the perception of sounds by a trained panel and recording of the sounds produced during application of a force to a food. Perception is evaluated by playing prerecorded biting and chewing sounds to panelists or by having the panelists masticate the food themselves. Recording of sounds produces an amplitude–time plot in which the force required to displace molecules is measured with a microphone. Combinations of number, height, and duration of peaks, sound pressure and energy, and other factors (Table 1.1) are used to formulate regression equations that characterize crispness and crunchiness.[5] When used in combination with fracture, compression, and puncture tests, acoustic measurements can accurately describe food sounds.

1.3.2 Mass–Volume–Area Related

This class of properties includes density, shrinkage, and porosity and is important for material handling and process design and characterization. Shrinkage is the volume change upon thermal or other treatment, and porosity is the void volume per total volume. True density is defined as mass per unit volume, and other types of density used in food analysis are apparent density (includes all pores in material), particle density (includes closed pores but not externally connected ones), material or substance density (when no closed pores exist), and bulk density (when packed or stacked, as in a shipment). Rahman reviewed the techniques for mass–volume–area determinations,[6] which are summarized in Table 1.2.

1.3.3 Morphology and Structure

In food analysis, morphology is the study of the size and shape of particles and their relation with material properties. Morphometric and structural characteristics affect the mechanical and sensory properties of food, which are responsible for overall quality. Microscopy, x-ray microtomography, magnetic resonance imaging, and

TABLE 1.1
Acoustic Measurement Parameters

Parameter	Definition
Amplitude	Height of sound wave
Mean height × number of sound bursts	Mean height of peaks multiplied by number of peaks in amplitude–time plot
Acoustic intensity	Energy per second over 1-m^2 area
Sound pressure	Force per unit area of sound wave proportional to square root of acoustic intensity
Equivalent sound level	Average of energy of sound produced
Fast Fourier analysis	Determines predominant sound frequencies

Source: Duizer, L., *Trends Food Sci. Technol.*, 12, 17–24, 2001.

TABLE 1.2
Mass–Volume–Area Measurement Techniques

Technique	Description	Application
	Density and Shrinkage	
Bulk density	Measures mass and volume of container	Bulk density of stacked or packed materials
Buoyant force	Weighs in air and in water	Apparent density of irregularly shaped coated solids
Gas pycnometer	Volume of gas displaced by sample	Apparent shrinkage, apparent density, and particle density of irregularly shaped solids, material density of ground samples
Geometric dimension	Measures height, width, depth	Apparent shrinkage and apparent density of regularly shaped solids
Liquid displacement	Volume of nonaqueous liquid displaced by sample	Apparent shrinkage and apparent material and particle density of irregularly shaped solids
Mercury porosimetry	Pressure at which mercury intrudes into pores	Pore volume and distribution
Solid displacement	Volume of solid (sand and glass beads) displaced by sample	Apparent shrinkage and apparent density of irregularly shaped solids
	Porosity	
Direct	Compares bulk and compacted volumes	Porosity of soft materials
Optical microscopy	Views random section under microscope	Porosity of solids
X-ray microtomography	Attenuation of x-rays provides three-dimensional image	Visualize pores in solids, estimates void volume

Source: Rahman, M.S., Mass-volume-area-related properties of foods, Pages 1–39 in *Engineering Properties of Foods*, 3rd edn., M.A. Rao, S.S.H. Rizvi, A.K. Datta, eds, CRC Press, Boca Raton, FL, 2005.

ultrasound measurements are available for examining structure and morphology. Falcone et al.[7] compared these techniques, which are shown in Table 1.3.

1.3.4 Rheology

Food rheology may be defined as the study of flow and deformation of ingredients, intermediate products, and final products of food processing. The three classes of rheological instruments are empirical, imitative, and fundamental. The first two correlate well with sensory methods but are arbitrary and cannot be converted to physical equivalents. Fundamental tests give well-defined results that can be treated mathematically but require continuous, homogeneous, and isotropic

TABLE 1.3
Morphology and Structure Analysis Techniques

Technique	Description	Application
Light microscopy	Visible light illuminates stained sample	View components such as fat droplets and protein
Scanning electron microscopy	Electrons illuminate sample surface	Visualize surface
Transmission electron microscopy	Electrons pass through thin sample layer	Two-dimensional picture of internal structure
Confocal laser scanning microscopy	Laser spot induces fluorescence	Three-dimensional topographic map
Atomic force microscopy	Deflection of cantilevered probe by repulsive forces on sample surface	Surface roughness
X-ray microtomography	Attenuation of x-rays provides three-dimensional image	Three-dimensional picture of internal structure
Magnetic resonance imaging	Detects proton spin alignment	Spatial distribution of water, salt, fat, etc.
Ultrasound	Attenuation of ultrasonic pulses	Two- and three-dimensional images of structure and physical state

Source: Falcone, P.M., et al., *Adv. Food Nutr. Res.*, 51, 205–263, 2006.

(same physical properties in all directions) samples and do not correlate well with sensory methods.

1.3.4.1 Empirical and Imitative Tests

There are dozens of empirical instruments for measuring rheological characteristics of food, and these are required for evaluating samples that are not homogeneous or isotropic. Imitative tests include texture profile analysis (TPA), which mimics chewing, and instruments that simulate handling of bread dough. The quantities obtained in TPA include hardness (maximum force during first compression), springiness (height specimen recovers between end of first compression and start of second), cohesiveness (ratio of area of second force–distance peak to first peak), and chewiness (product of these three parameters).

1.3.4.2 Fundamental Tests

The two major categories of fundamental tests for liquid and semisolid samples are rotational type and tube type; for solid samples, compression, dynamic mechanical analysis, tension, and torsion instruments are used. These types have been discussed by Steffe[8] and Bourne[9] and are shown in Table 1.4. Dynamic mechanical analysis is often performed using oscillatory shear analyses in the linear viscoelastic range. All foods exhibit viscoelastic properties, and the equation governing this behavior is

$$G^* = G' + iG'' \tag{1.1}$$