Food and dairy powders are created by dehydrating perishable produce, such as milk, eggs, fruit and meat, in order to extend their shelf life and stabilise them for storage or transport. These powders are in high demand for use as ingredients and as food products in their own right, and are of great economic importance to the food and dairy industry worldwide. Today, the ability to control food and dairy powder quality is a source of key competitive advantage. By varying the dehydration process design, and by controlling the technological and thermodynamic parameters during dehydration, it is possible for manufacturers to engineer the biochemical, microbiological and physical characteristics of the food powder to meet their specific product requirements.

This book provides an overview of the existing, adapted or new techniques used to analyse safety and quality in modern food and dairy powders. Based on original research by the authors, the book uses 25 commercial dairy and non-dairy powders to illustrate a range of biochemical and physical methods used to evaluate and characterise powdered food products. Written from a practical perspective, each chapter focuses on a particular analytical technique, outlining the purpose, definition and principle of that method. The authors guide the reader through all of the instruments needed, the safety measures required, and the correct procedures to follow to ensure successful analysis. Instructions on accurate measurement and expression of results are included, and each chapter is richly illustrated with original data and worked examples.

Analytical Methods for Food and Dairy Powders is a unique step-by-step handbook, which will be required reading for anyone involved in the development and manufacture of powdered food products. Food and dairy scientists based in industry will find it essential for new product development and improved quality control, while researchers in the laboratory will especially value the new techniques it comprises.

About the authors
Pierre Schuck, Anne Dolivet and Romain Jeantet are all based at INRA (the French National Institute for Agricultural Research), Agrocampus Ouest, Rennes, France. Pierre Schuck is Research Engineer, Anne Dolivet is Research Technician and Romain Jeantet is Professor in Food Science and Process Engineering.

Also available
Dairy Powders and Concentrated Products
Edited by A.Y. Tamime

Drying Technologies in Food Processing
Edited by Xiao Dong Chen and Arun S. Mujumdar
ISBN 978-1-4051-5765-0

Statistical Methods for Food Science: Introductory procedures for the food practitioner
John Bowler

Cover design by www.hsandhersdesign.co.uk
Cover image credit: © iStockphoto.com/hlphoto
www.wiley.com/go/food
Analytical Methods for Food and Dairy Powders
Analytical Methods for Food and Dairy Powders

Pierre Schuck\textsuperscript{1,2}, Anne Dolivet\textsuperscript{1,2} and Romain Jeantet\textsuperscript{2,1}
\textsuperscript{1}INRA, UMR 1253, F-35000 Rennes, France
\textsuperscript{2}Agrocampus Ouest, UMR 1253, F-35000 Rennes, France
# CONTENTS

## Foreword

### Chapter 1 Dehydration Processes and their Influence on Powder Properties

- **1.1. Overview of operations**
  - 1.1.1. Concentration by evaporation
  - 1.1.2. Drying
- **1.2. Properties of dehydrated products**
  - 1.2.1. Biochemical and physicochemical properties
  - 1.2.2. Microbiological properties
  - 1.2.3. Properties of use
- **1.3. Bibliography**

### Chapter 2 Determination of Dry Matter and Total Dry Matter

- **2.1. Determination of free moisture or dry matter**
  - 2.1.1. Purpose and range of application
  - 2.1.2. Definition
  - 2.1.3. Principle
  - 2.1.4. Reagents and other products
  - 2.1.5. Instruments and glassware
  - 2.1.6. Safety
  - 2.1.7. Procedure
  - 2.1.8. Expression of results
  - 2.1.9. Remarks
  - 2.1.10. Precision values
2.1.11. Examples 49

2.2. Determination of total moisture or total dry matter 50
  2.2.1. Purpose and range of application 50
  2.2.2. Definition 51
  2.2.3. Principle 51
  2.2.4. Reagents and other products 51
  2.2.5. Instruments and glassware 51
  2.2.6. Safety 53
  2.2.7. Procedure 53
  2.2.8. Expression of results 54
  2.2.9. Remarks 55
  2.2.10. Precision values 56
  2.2.11. Analysis report 57
  2.2.12. Examples 57

2.3. Bibliography 57

Chapter 3 Determination of Nitrogen Fractions 59
  3.1. Determination of the total nitrogen content (Kjeldahl method) 60
    3.1.1. Purpose and range of application 60
    3.1.2. Definition 60
    3.1.3. Principle 60
    3.1.4. Reagents and other products 61
    3.1.5. Instruments and glassware 61
    3.1.6. Safety 62
    3.1.7. Procedure 62
    3.1.8. Expression of results 65
    3.1.9. Precision values 66
    3.1.10. Examples 66
    3.1.11. Annex 67

  3.2. Determination of the nitrogen content soluble at pH 4.60 69
    3.2.1. Purpose and range of application 69
    3.2.2. Definition 69

Contents
3.2.3. Principle 69
3.2.4. Reagents and other products 69
3.2.5. Instruments and glassware 70
3.2.6. Safety 70
3.2.7. Procedure 70
3.2.8. Expression of results 72
3.2.9. Precision values 73
3.2.10. Examples 73
3.2.11. Annex 73

3.3. Determination of the non-protein nitrogen content 76
3.3.1. Purpose and range of application 76
3.3.2. Definition 76
3.3.3. Principle 76
3.3.4. Reagents and other products 76
3.3.5. Instruments and glassware 77
3.3.6. Safety 77
3.3.7. Procedure 77
3.3.8. Expression of results 78
3.3.9. Precision values 79
3.3.10. Examples 80
3.3.11. Annex 80

3.4. Determination of non-denatured whey protein nitrogen in skimmed milk powder 82
3.4.1. Purpose and range of application 82
3.4.2. Definition 82
3.4.3. Principle 82
3.4.4. Expression of results 83
3.4.5. Remarks 83
3.4.6. Examples 84

3.5. Protein nitrogen conversion factors based on amino acid composition in the case of milk and soy 85
3.5.1. Methods for the determination of the conversion factor 85
3.5.2. Conversion factors for milk, specific milk proteins, certain milk products and infant formulas 86
3.5.3. Conversion factors for soy and its derivatives 88
3.5.4. Conclusion 90
3.6. Bibliography 90

Chapter 4 Determination of the Rate of Lactose Crystallisation 93
4.1. Definitions 94
4.2. Principle 95
  4.2.1. Determination of the moisture content 95
  4.2.2. Determination of the total moisture content 95
4.3. Expression of results 95
4.4. Remarks 95
4.5. Examples 96
4.6. Bibliography 96

Chapter 5 Determination of Total Fat and Free Fat Content 99
5.1. Determination of total fat content 100
  5.1.1. Purpose and range of application 100
  5.1.2. Definition 101
  5.1.3. Principle 101
  5.1.4. Reagents and other products 101
  5.1.5. Instruments and glassware 101
  5.1.6. Safety 102
  5.1.7. Procedure 102
  5.1.8. Expression of results 105
  5.1.9. Remarks 106
  5.1.10. Precision values 106
  5.1.11. Examples 106
5.2. Determination of free fat content 107
Chapter 5 Determination of the Ash Content

5.2.1. Purpose and range of application

5.2.2. Definition

5.2.3. Principle

5.2.4. Reagents and other products

5.2.5. Instruments and glassware

5.2.6. Safety

5.2.7. Procedure

5.2.8. Expression of results

5.2.9. Remarks

5.2.10. Precision values

5.2.11. Analysis report

5.2.12. Examples

5.3. Bibliography

Chapter 6 Determination of the Ash Content

6.1. Definitions

6.2. Principle

6.3. Instruments and glassware

6.4. Personal protection

6.5. Procedure

6.5.1. Preparation of the sample

6.5.2. Preparation of the crucible

6.5.3. Sample

6.5.4. Measurement

6.6. Expression of results

6.7. Precision values

6.7.1. Repeatability

6.8. Examples

6.9. Bibliography

Chapter 7 Determination of Particle Size and Friability

7.1. Definition

7.2. Principle

7.3. Methods

7.3.1. Sieve particle size analysis

7.3.2. Laser particle size analysis

7.4. Reagents and other products

Contents
## Contents

### Chapter 7

<table>
<thead>
<tr>
<th>Section</th>
<th>Page</th>
</tr>
</thead>
<tbody>
<tr>
<td>7.5. Instruments and glassware</td>
<td>120</td>
</tr>
<tr>
<td>7.5.1. Sieve particle size analysis</td>
<td>120</td>
</tr>
<tr>
<td>7.5.2. Laser particle size analysis</td>
<td>121</td>
</tr>
<tr>
<td>7.6. Personal protection</td>
<td>121</td>
</tr>
<tr>
<td>7.7. Procedure</td>
<td>121</td>
</tr>
<tr>
<td>7.7.1. Sieve particle size analysis</td>
<td>121</td>
</tr>
<tr>
<td>7.7.2. Laser particle size analysis</td>
<td>121</td>
</tr>
<tr>
<td>7.8. Expression of results</td>
<td>121</td>
</tr>
<tr>
<td>7.8.1. Sieve particle size analysis</td>
<td>121</td>
</tr>
<tr>
<td>7.8.2. Laser particle size analysis</td>
<td>122</td>
</tr>
<tr>
<td>7.8.3. Friability</td>
<td>123</td>
</tr>
<tr>
<td>7.9. Remarks</td>
<td>123</td>
</tr>
<tr>
<td>7.9.1. Particle size analysis</td>
<td>123</td>
</tr>
<tr>
<td>7.9.2. Sieve particle size analysis</td>
<td>124</td>
</tr>
<tr>
<td>7.9.3. Mesh size less than 120μm</td>
<td>124</td>
</tr>
<tr>
<td>7.10. Precision values</td>
<td>124</td>
</tr>
<tr>
<td>7.10.1. Repeatability</td>
<td>124</td>
</tr>
<tr>
<td>7.11. Examples</td>
<td>125</td>
</tr>
<tr>
<td>7.12. Bibliography</td>
<td>127</td>
</tr>
</tbody>
</table>

### Chapter 8

Chapter 8  Determination of Flowability and Floodability Indices  129

<table>
<thead>
<tr>
<th>Section</th>
<th>Page</th>
</tr>
</thead>
<tbody>
<tr>
<td>8.1. Definition</td>
<td>129</td>
</tr>
<tr>
<td>8.1.1. Flowability–fluidity</td>
<td>129</td>
</tr>
<tr>
<td>8.1.2. Floodability</td>
<td>130</td>
</tr>
<tr>
<td>8.2. Principle</td>
<td>130</td>
</tr>
<tr>
<td>8.2.1. Flowability–fluidity</td>
<td>130</td>
</tr>
<tr>
<td>8.2.2. Floodability</td>
<td>130</td>
</tr>
<tr>
<td>8.3. Reagents and other products</td>
<td>130</td>
</tr>
<tr>
<td>8.4. Instruments and glassware</td>
<td>130</td>
</tr>
<tr>
<td>8.4.1. The main unit</td>
<td>131</td>
</tr>
<tr>
<td>8.4.2. Accessories</td>
<td>131</td>
</tr>
<tr>
<td>8.5. Procedure</td>
<td>132</td>
</tr>
<tr>
<td>8.5.1. Flowability–fluidity</td>
<td>132</td>
</tr>
<tr>
<td>8.5.2. Floodability</td>
<td>136</td>
</tr>
<tr>
<td>8.6. Expression of results</td>
<td>137</td>
</tr>
<tr>
<td>8.6.1. Flowability–fluidity</td>
<td>137</td>
</tr>
<tr>
<td>8.6.2. Floodability</td>
<td>137</td>
</tr>
</tbody>
</table>
Chapter 9  Determination of Density, Interstitial Air Content and Occluded Air

9.1. Definition 146
9.2. Principle 146
9.3. Methods 146
  9.3.1. Bulk density, $\rho_B$ and tapped density, $\rho_T$ 146
  9.3.2. True density, $\rho_{TR}$ 147
9.4. Equipment and glassware 147
  9.4.1. Bulk density, $\rho_B$ and tapped density, $\rho_T$ 147
  9.4.2. True density, $\rho_{TR}$ 147
9.5. Safety 147
  9.5.1. Personal protection 147
9.6. Procedure 147
  9.6.1. Bulk density, $\rho_B$ and tapped density, $\rho_T$ 147
  9.6.2. True density, $\rho_{TR}$ 148
9.7. Expression of results 148
  9.7.1. Bulk density ($\rho_B$) 148
  9.7.2. Tapped density ($\rho_T$) 149
  9.7.3. True density ($\rho_{TR}$) 149
  9.7.4. Interstitial air (IA) 149
  9.7.5. Occluded air (OA) 149
9.8. Remarks 149
  9.8.1. True density 149
  9.8.2. True volume 150
9.9. Precision values 151
  9.9.1. Repeatability 151
9.10. Examples 151
9.11. Bibliography 154
12.7. Expression of results 195
   12.7.1. Differential calorimetry 195
   12.7.2. Rheological method 196
12.8. Remarks 196
   12.8.1. Adapt methods depending on powders being analysed 196
   12.8.2. Conventional or modulated temperature differential scanning calorimetry 197
   12.8.3. $T_g$ values determined by differential scanning calorimetry and rheological analysis 197
12.9. Precision values 198
   12.9.1. Repeatability 198
12.10. Examples 198
12.11. Bibliography 201

Chapter 13 Determination of Rehydration Ability 203
13.1. Determination of wettability 204
   13.1.1. Definition 204
   13.1.2. Principle 204
   13.1.3. Instruments and glassware 204
   13.1.4. Procedure 204
   13.1.5. Expression of results 205
   13.1.6. Remarks 205
   13.1.7. Precision values 205
   13.1.8. Examples 206
13.2. Determination of dispersibility 207
   13.2.1. Definition 207
   13.2.2. Principle 207
   13.2.3. Instruments and glassware 207
   13.2.4. Procedure 207
   13.2.5. Expression of results 208
   13.2.6. Remarks 208
   13.2.7. Precision values 209
   13.2.8. Examples 209
13.3. Determination of solubility 209
   13.3.1. Definition 209
## Contents

13.3.2. Principle 210  
13.3.3. Reagents and other products 210  
13.3.4. Instruments and glassware 211  
13.3.5. Procedure 211  
13.3.6. Expression of results 212  
13.3.7. Remarks 212  
13.3.8. Precision values 213  
13.3.9. Examples 213  

13.4. Bibliography 215

Chapter 14 Summary and General Conclusion 217

Index 227

A colour plate section falls between pages 156 and 157
The main purpose of drying dairy and food products is to stabilise them in order to facilitate storage and extend shelf life. Since the 1970s, the most common dehydration technique for liquid food has been spray drying. This technique was developed based on industrial know-how through empirical reasoning given the lack of scientific and technical papers on the subject, and, in particular, the impact of spray drying and the physicochemical and microbiological properties of the concentrate on the quality of the powder. Today, the diversity and complexity of the concentrates make it necessary to develop a more rigorous approach taking into account physicochemical and thermodynamic factors. This approach is based on a better understanding of the biochemical properties of the concentrate before drying, water transfers during drying, powder properties and the factors that influence them.

Food powders are generally characterised by their dry matter content. However, other characteristics need to be taken into account such as, for example, biochemical properties (protein, carbohydrate, lipid and mineral composition), as well as microbiological and physical properties (e.g. density, interstitial and occluded air, particle size, solubility, dispersibility, wettability, flowability, floodability, hygroscopicity, etc.), all of which will be discussed in this book. These characteristics depend on the manufacturing process (premixing, co-drying, dry-mixing, etc.), technological and thermodynamic parameters during the different stages of dehydration (drying chamber design, type of spray drying, fines recycling and thermodynamic properties of the air: temperature, relative humidity and velocity) and the characteristics of the concentrate before spraying (composition,
physicochemistry and rheology, water availability, etc.). All these properties determine:

- the preservation/storage conditions of powders, in relation to their sensitivity to different types of alteration (non-enzymatic browning or Maillard reactions, oxidation, microbial growth, etc.)
- the use properties of powders (flowability, solubility, rehydration, etc.)
- and, more generally, powder quality, from a functional, nutritional and sensory perspective.

Today, the quality control of powders based on these elements is a key competitive advantage. However, this involves controlling all the influencing factors, whether they are related to the composition of the products or the technologies used. The main barriers to understanding the impact of composition and process parameters on spray-dried products were methodology and analytical protocol. The physicochemical analyses described in the literature mainly result from methods applied to basic dairy powders, often yielding poor or inadequate results for the characterisation of new functionalities or complex non-dairy powders.

The development of methods and techniques suitable for powder analysis meets these requirements. This book contributes to this overall strategy by outlining all the relevant measures to effectively carry out the drying and quality control of powders.

Chapter 1 deals with dehydration processes in general and their influence on powder properties. Chapters 2 to 6 (Part 1) outline a set of tools and biochemical methods while Chapters 7 to 13 (Part 2) outline the physicochemical methods with the aim of providing a more accurate characterisation of powders as well as a more precise definition of their specifications. These tools and methods have been described in detail by providing comments, remarks and suggestions for improvement with regard to their implementation and the formulation of analytical results. For illustrative purposes, these methods were tested in triplicate on ten commercial dairy powders obtained by spray drying, including:
skimmed milk
milk with 26% fat in dry matter (milk 26% fat)
micellar casein obtained by microfiltration of skimmed milk (micellar casein)
calcium and sodium caseinates obtained by neutralising an acid curd with calcium and sodium hydroxide, respectively
sweet whey obtained from the manufacture of hard pressed cheese (whey) and fat-filled whey with 40% fat using coconut oil (whey 40% fat)
ultrafiltration permeate of milk (UF permeate)
whey protein concentrate with 35% protein in dry matter obtained by ultrafiltration of whey (WPC 35)
whey protein isolate with 90% protein in dry matter obtained by ultrafiltration of milk microfiltrate (WPI 90)
and 15 commercial non-dairy food powders obtained by spray drying, including:
maltodextrins with different dextrose equivalents (DE): two powders with DE 12 (MD DE 12 (1) and (2)), one with 19 (MD DE 19) and a glucose syrup with DE 39 (GS DE 39)
polyols, as sorbitol and maltitol
apple extracts (apple (1) and (2))
chicory extracts (chicory (1) and (2))
egg products: whole egg, egg yolk and white egg
pet food
gelatin.

The analytical results obtained are individually discussed at the end of each chapter. Finally, a comprehensive analysis was conducted to identify the correlations between the analysed properties (Chapter 14).

Dr Pierre Schuck
Pr Romain Jeantet
Pr Gérard Brulé
Dr Jean-Louis Maubois
Chapter 1

DEHYDRATION PROCESSES AND THEIR INFLUENCE ON POWDER PROPERTIES

Most microbial and biochemical changes that alter the quality of food occur in the aqueous phase. Water plays a dual role:

- As a solvent, it ensures the transfer of substrates, growth promoters, biological agents and reaction products, which allows reactions to take place in optimal conditions.
- As a reaction substrate, it is involved in hydrolysis reactions (proteolysis, lipolysis).

This dual action requires that water is available, which can be characterised by its water activity ($a_w$; cf. 1.2.1.2), i.e. the ratio between the partial pressure of the water vapour of the product and the partial pressure of pure water vapour at the same temperature. Any process that reduces this availability also slows down reaction times.

Water activity can be lowered by the crystallisation of solvent water (freezing) or by the addition of highly hydrophilic solutes that bind water molecules through hydrogen or dipolar interactions (salting, sugaring). It can also be lowered by eliminating the available water (concentration, evaporation and drying); in this case the inhibition generated is removed by dilution or rehydration.
This book deals with the properties of food powders obtained through drying. This preservation method only slightly alters the nutritional and organoleptic qualities during dehydration and any pre-treatments are well controlled with regard to heat and mass transfer.

Given the high latent heat of the vaporisation of water (2258 kJ kg\(^{-1}\) at 100°C), the drying process is often preceded by a concentration of the dry matter within the product to reduce the energy cost of processing. This pre-concentration can be achieved by cross-flow filtration (reverse osmosis for example) or by vacuum evaporation. In reverse osmosis, water is removed without phase change by passing the product through a membrane under the action of a pressure gradient, which reduces the energy cost of water elimination (10–40 kJ kg\(^{-1}\) water). However, the efficiency of the process decreases with increasing viscosity and osmotic pressure resulting from a concentration of dry matter (proteins and molecules of low molecular weight, respectively). Thus, it is generally not possible to concentrate the product beyond 25% (w/w) of dry matter.

Therefore, in this chapter we only deal with concentration by vacuum evaporation and drying, which are the two main unit operations used in the manufacture of dried products.

1.1. Overview of operations

1.1.1. Concentration by evaporation

Concentration by evaporation involves exposing a liquid to temperature and pressure conditions that allow vaporisation of the solvent. This process therefore facilitates a concentration of non-volatile elements in the treated product. In the food industry, it is mainly used to remove water from true solutions, emulsions and/or colloidal solutions.

A key aspect of this technique is the energy cost involved, since water is removed by a phase change (liquid–vapour), contrary to separation techniques. Therefore, the concentration at an atmospheric pressure of 1 kg of a 10% sucrose solution, initially at 20°C, to 20% sucrose (elimination of 0.5 kg water) requires a total of 1439 kJ. This energy breaks down as follows: the addition of
sensible heat allowing an increase from 20 to 100°C (311 kJ) and the addition of latent heat to vaporise 0.5 kg of water at 100°C (1128 kJ). However, the energy difference between the initial and final systems at 20°C is only 0.5 kJ, which corresponds to an isothermal compression of sucrose molecules. Concentration by evaporation therefore has a very low efficiency, without any energy recovery. Most of the technical developments made were aimed at improving efficiency.

Furthermore, processed food liquids are often heat sensitive. To minimise the biochemical alteration of components, concentration by evaporation is generally carried out under a partial vacuum to reduce the processing temperature by 45–80°C. While the qualitative advantage of this practice may be obvious, the energy gain is in fact low. The alteration of components, according to a time–temperature relationship, can also be reduced by decreasing the residence time in the facility. The physicochemical characteristics of the concentrate (non-denatured protein nitrogen \([WPNi]\); cf. 1.2.1.3], viscosity, insoluble mineral) depend on the length and temperature of the process and the ionic force; these characteristics largely determine the properties and qualities of the final powder.

First the principle of vacuum evaporation is discussed. Then the different techniques to reduce energy consumption are explored.

1.1.1.1. Principle of vacuum evaporation

Single-stage vacuum evaporation consists of placing the liquid to be concentrated, which has been brought to its boiling temperature beforehand, into a vacuum chamber (evaporation body; Figure 1.1). The vacuum, obtained by the condensation of spray in contact with a cold source, corresponds to the saturation vapour pressure at the boiling point of the product.

In this context, any heat applied to the product will result in vaporisation of some liquid. The evaporation body is thus a heat exchanger for providing the product with the latent heat of vaporisation. In practice, the energy supplied to the heat exchanger (tube bundle in general) comes from vapour at a temperature of 5–10°C higher than that of the product.

The liquid–vapour mixture is separated in a separation container attached to the evaporation body. In this way the secondary vapour
(still called vapour spray) as well as the concentrated liquid is collected. The energy contained in the vapour mist is usually recovered either to reheat the incoming product or to heat a second evaporation body. This principle of multiple-stage evaporation will be explained in greater detail later.

Each evaporation unit must meet three industry requirements: high evaporation capacity, low specific energy consumption and ability to maintain quality of the concentrate. The types of evaporator differ depending on the liquid flow or the geometry of the heating surfaces, and are more or less adapted to the different food liquids:

1. Climbing film evaporators, used in the sugar industry in particular. The concept of ‘climbing film’ means that the liquid, introduced at the base of the unit, rises while concentrating inside the tubes, thereby ensuring complete wetting of the exchange surfaces.
2. Falling film evaporators (Figure 1.1), used mainly in the dairy industry. The heat transfer is improved compared with climbing film evaporators due to better liquid flow conditions: the liquid,