- Food

Integrating Safety and Environment Knowledge
Into Food Studies
towards European Sustainable Development

Margarida Vieira
Peter Ho
Editors

Experiments in Unit Operations and Processing of Foods

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Margarida Vieira
Universidade do Algarve, Instituto Superior de Engenharia
Campus da Penha, 8000 Faro, Portugal

Peter Ho
Instituto Politécnico de Viana do Castelo, Avenida do Atlântico-Apartado 574
4900-348 Viana do Castelo, Portugal

Springer

Editors
Margarida Vieira
Universidade do Algarve
Instituto Superior de Engenharia
Campus da Penha
8000 Faro, Portugal
mviera@ualg.pt

Peter Ho
Instituto Politécnico de Viana do Castelo
Avenida do Atlântico-Apartado 574
4900-348 Viana do Castelo
Portugal
peter@estg.ipvc.pt

Series Editor
Kristberg Kristbergsson
Department of Food Science
Faculty of Food Science and Nutrition
University of Iceland
Iceland
kk@hi.is

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To all our students because they deserve our effort

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SERIES PREFACE

The single most important task of food scientists and the food industry as a whole is to ensure the safety of foods supplied to consumers. Recent trends in global food production, distribution and preparation call for increased emphasis on hygienic practices at all levels and for increased research in food safety in order to ensure a safer global food supply. The ISEKI-Food book series is a collection of books where various aspects of food safety and environmental issues are introduced and reviewed by scientists specializing in the field. In all of the books a special emphasis was placed on including case studies applicable to each specific topic. The books are intended for graduate students and senior level undergraduate students as well as professionals and researchers interested in food safety and environmental issues applicable to food safety.

The idea and planning of the books originates from two working groups in the European thematic network "ISEKI-Food" an acronym for "Integrating Safety and Environmental Knowledge In to Food Studies". Participants in the ISEKI-Food network come from 29 countries in Europe and most of the institutes and universities involved with Food Science education at the university level are represented. Some international companies and nonteaching institutions have also participated in the program. The ISEKI-Food network is coordinated by Professor Cristina Silva at The Catholic University of Portugal, College of Biotechnology (Escola) in Porto. The program has a web site at: http://www.esb.ucp.pt/iseki/. The main objectives of ISEKI-Food have been to improve the harmonization of studies in food science and engineering in Europe and to develop and adapt food science curricula emphasizing the inclusion of safety and environmental topics. The ISEKI-Food network started on October 1st in 2002, and has recently been approved for funding by the EU for renewal as ISEKI-Food 2 for another 3 years. ISEKI has its roots in an EU funded network formed in 1998 called Food Net where the emphasis was on casting a light on the different Food Science programs available at the various universities and technical institutions throughout Europe. The work of the ISEKI-Food network was organized into five different working groups with specific task all aiming to fulfill the main objectives of the network.

The first four volumes in the ISEKI-Food book series come from WG2 coordinated by Gerhard Schleining at Boku University in Austria and the undersigned. The main task of the WG2 was to develop and collect materials and methods for teaching of safety and environmental topics in the food science and engineering curricula. The first volume is devoted to Food Safety in general with a practical and a case study approach. The book is composed of 14 chapters which were organized into three sections on preservation and protection; benefits and risk of microorganisms and process safety. All of these issues have received high public interest in recent years and will continue to be in the focus of consumers and

regulatory personnel for years to come. The second volume in the series is devoted to the control of air pollution and treatment of odors in the food industry. The book is divided into eight chapters devoted to defining the problem, recent advances in analysis and methods for prevention and treatment of odors. The topic should be of special interest to industry personnel and researchers due to recent and upcoming regulations by the European Union on air pollution from food processes. Other countries will likely follow suit with more strict regulations on the level of odors permitted to enter the environment from food processing operations. The third volume in the series is devoted to utilization and treatment of waste in the food industry. Emphasis is placed on sustainability of food sources and how waste can be turned into by products rather than pollution or land fills. The Book is composed of 15 chapters starting off with an introduction of problems related to the treatment of waste, and an introduction to the ISO 14001 standard used for improving and maintaining environmental management systems. The book then continues to describe the treatment and utilization of both liquid and solid waste with case studies from many different food processes. The last book from WG2 is on predictive modeling and risk assessment in food products and processes. Mathematical modeling of heat and mass transfer as well as reaction kinetics is introduced. This is followed by a discussion of the stoichiometry of migration in food packaging, as well as the fate of antibiotics and environmental pollutants in the food chain using mathematical modeling and case study samples for clarification.

Volumes five and six come from work in WG5 coordinated by Margarida Vieira at the University of Algarve in Portugal and Roland Verhé at Gent University in Belgium. The main objective of the group was to collect and develop materials for teaching food safety-related topics at the laboratory and pilot plant level using practical experimentation. Volume five is a practical guide to experiments in unit operations and processing of foods. It is composed of 20 concise chapters each describing different food processing experiments outlining theory, equipment, procedures, applicable calculations and questions for the students or trainee followed by references. The book is intended to be a practical guide for the teaching of food processing and engineering principles. The final volume in the ISEKI-Food book series is a collection of case studies in food safety and environmental health. It is intended to be a reference for introducing case studies into traditional lecture-based safety courses as well as being a basis for problem-based learning. The book consists of 13 chapters containing case studies that may be used, individually or in a series, to discuss a range of food safety issues. For convenience the book was divided into three main sections with the first devoted to case studies, in a more general framework with a number of specific issues in safety and health ranging from acrylamide and nitrates to Botulism and Listeriosis. The second section is devoted to some well known outbreaks related to food intake in different countries. The final section of the book takes on food safety from the perspective of the researcher. Cases are based around experimental data and examine the importance of experimental planning, design and analysis.

The ISEKI-Food books series draws on expertise form close to a hundred universities and research institutions all over Europe. It is the hope of the authors,

editors, coordinators and participants in the ISEKI network that the books will be useful to students and colleagues to further their understanding of food safety and environmental issues.

March, 2008 Kristberg Kristbergsson

PREFACE

Understanding basic principles and concepts in food engineering and their application in the processing of food should be an integral part of any Food Studies Curricula. Practical knowledge of engineering principles and processing is essential for anyone intending to work in the food industry or otherwise involved in food research, whether being trained as a microbiologist, food chemist, nutritionist, sensory scientist or a food engineer. However, the degree of importance and depth of information taught in courses on unit operations and food processing of undergraduate degree programs in Food Engineering, Food Technology and Food Science in Europe varies tremendously. Practical and laboratory training at the pilot plant level is one particular area that was lacking in some courses of academic institutions participating in the ISEKI-Food thematic network project (http://www.esb.ucp.pt/isekipast), due to inadequate pilot plant facilities.

This book contains experimental protocols in unit operations and food processing, adapted from practical courses currently running in those institutions involved in ISEKI-Food. It is based on information from a database, developed by ISEKI-Food, on equipment found in laboratories and pilot plants in European academic institutions teaching Food Studies programs. The book does not try to cover all unit operations used in the food industry, nor does it intend to give a representative view of food processes, as it is based solely from information presently available from the pilot plant database. Instead, it is seen as a resource meant to complement existing practical courses, when equipment needed to perform certain unit operations may be unavailable. The book may also be useful for institutions considering modifying or implementing new practical courses and may not have the resources available to invest in the acquisition of new equipment. Basic theory, discussion questions and references to relevant sources are provided for each experimental protocol. In most cases, they are also accompanied with an example questionnaire and a dataset from a typical practical experiment that was run by the authors of the experimental protocol.

This book is divided into two main parts on unit operations and a third on Food processing. The first part is on conversion unit operations while the second part concentrates on preservation unit operations. **Chapter 1** examines different equipment used for the mixing of solid particles, while **Chapter 2** looks at filterability of suspensions, and **Chapter 3** on the use of a Plate-and-frame filtration apparatus. Separation of cream from milk by centrifugation is handled in **Chapter 4**, and direct osmotic concentration of liquids in **Chapter 5**. Two other techniques are considered in the final two chapters of this section, vacuum impregnation of fruit in **Chapter 6** and the extraction of pumpkin oil in **Chapter 7**.

In part II, the drying of solid foodstuffs are covered by the first two chapters. **Chapter 8** looks at tray drying whereas **Chapter 9** examines the use of a combined air-

microwave drying process. The operation of a spray dryer and a fluidized bed dryer are subsequently considered under **Chapter 10** and **Chapter 11**. **Chapter 12** covers freeze drying, **Chapter 13** the use of a plate freezer, whereas **Chapter 14** compares air blast freezing with fluidized bed freezing. Pasteurization with a plate heat exchanger (**Chapter 15**) and sterilization in a retort (**Chapter 16**) are covered in the last two chapters of this section.

The final section of this book gives four examples of food processing. **Chapter 17** looks at the production of pre-gelatinized amaranth flour, **Chapter 18** on wheat crisps, **Chapter 19** on Semolina and **Chapter 20** on Cheese making.

We hope that ***Experiments in Unit Operations and Processing of Foods*** will eventually become an essential addition to any course curricula in Food Studies, aimed at practical training of food engineering concepts and principles.

Portugal in February 2008
Peter Ho
Maria Margarida Cortez Vieira

CONTENTS

CONTRIBUTORS

Paulina Aleknavičienė
Department of Horticulture, Faculty of Agronomy, University of Agriculture, Studentų 11 Akademija – 53361 Kaunas, Lithuania, ns@lzuu.lt

Mustafa Bayram
Department of Food Engineering, Faculty of Engineering, University of Gaziantep 3, 27310-Gaziantep-Turkey 3, mbayram@gantep.edu.tr

Rui Costa
Escola Superior Agrária de Coimbra, Instituto Politécnico de Coimbra, College of Agriculture, 3040-316 Coimbra, Portugal, ruicosta@esac.pt

Honorata Danilčenko
Department of Horticulture, Faculty of Agronomy, University of Agriculture, Studentų 11 Akademija – 53361 Kaunas, Lithuania, ns@lzuu.lt

Albert Duquenoy
UMR Génie Industriel Alimentaire, ENSIA, Ensia, 1 Avenue des Olympiades – 91744 Massy Cedex, France, albert.duquenoy@agroparistech.fr

Victoria Ferragut
Centre Especial de Recerca Planta de Tecnologia Dels Aliments (cerpta), Departament de Ciència Animal i Dels Aliments, Facultat de Veterinària, Universitat Autònoma de Barcelona, 08193 Bellaterra, Spain, victoria.ferragut@uab.es

Pedro Fito
Food Technology Department, Universidad Politécnica de Valencia, Camino de Vera s/n 46022 Valencia, Spain, pfito@tal.upv.es

Fahrettin Göğüş
Department of Food Engineering, Faculty of Engineering, University of Gaziantep 3, 27310-Gaziantep-Turkey 3, fahret@gantep.edu.tr

David Gomes
Escola Superior Agrária de Coimbra, Instituto Politécnico de Coimbra, College of Agriculture, 3040-316 Coimbra, Portugal

Chelo Gonzalez-Martinez
Food Technology Department, Universidad Politécnica de Valencia, Camino de Vera s/n 46022 Valencia, Spain, cgonza@tal.upv.es

Cecilia Hodúr
Department of Unit Operation and Environmental Technique, Faculty of Food Engineering, University of Szeged, 6724 Szeged-Hungary, hodur@mk.u-szeged.hu

Lea Hyvönen
Department of Food Technology, University of Helsinki, P.o. Box 66, fi-00014, Finland, lea.hyvonen@helsinki.fi

Elvyra Jarienė
Department of Horticulture, Faculty of Agronomy, University of Agriculture, Studentų 11 Akademija – 53361 Kaunas, Lithuania, ns@lzuu.lt

Kirsi Jouppila
Department of Food Technology, University of Helsinki, P.o. Box 66, FI-00014, Finland, Finland, kirsi.jouppila@helsinki.fi

Jurgita Kulaitienė
Department of Horticulture, Faculty of Agronomy, University of Agriculture, Studentų 11 Akademija – 53361 Kaunas, Lithuania, ns@lzuu.lt

Marie-Laure Lameloise
Departement de Genie Industriel Alimentaire, Ecole Nationale Superieure des Industries Agricoles et Alimentaires, 1 Avenue des Olympiades, 91744 Massy Cedex – France, marie-laure.lameloise@agroparistech.fr

Harris n. Lazarides
Laboratory of Food Processing and Engineering, Department of Food science and Technology, School of Agriculture, Aristotle University of Thessaloniki, Box 255, Thessaloniki 540 06, Hellas, lazaride@agro.auth.gr

Cornelius Lucher
Department of Food Biotechnology and Food Process Engineering, Technische Universität Berlin, Berlin, cornelius.luscher@tu-berlin.de

Eugenia Martin-Esparza
Department of Food Technology, Universidad Politécnica de Valencia, Camino de Vera s/n, 46022 Valencia, Spain, eesparza@tal.upv.es

Alexandra Morim
Escola Superior de Biotecnologia, Universidade Católica Portuguesa, Rua dr. António Bernardino de Almeida, 4200-072 Porto, Portugal, amorin@esb.ucp.pt

Semih Otles
Ege University, Department of Food Engineering, Tr-35100 Izmir, Turkey, Semih.otles@ege.edu.tr

Maria Papageorgiou
National Agricultural Research Foundation, Cereal institute, Laboratory of Cereal Chemistry and Technology, P.o.b 60411 gr-57001 Thermi Greece, tech.ci@nagref.gr

Jorge Pereira
Escola Superior de Tecnologia, Universidade do Algarve, Campus da Penha 8000-225 Faro, Portugal, aper@ualg.pt

Cristina Luisa Silva
Escola Superior de Biotecnologia, Universidade Católica Portuguesa, Rua dr. António Bernardino de Almeida, 4200-072 Porto, Portugal, clsilva@esb.ucp.pt

Toni Trujillo
Centre Especial de Recerca Planta de Tecnologia Dels Aliments (cerpta), Departament de Ciència Animal i Dels Aliments, Facultat de Veterinària, Universitat Autònoma de Barcelona, 08193 Bellaterra, Spain, toni.trujillo@uab.es

Margarida Vieira
Escola Superior de Tecnologia, Universidade do Algarve, Oampus da Penha 8000-225 Faro, Portugal, mvieira@ualg.pt

Gernot Zweytick
Department of Food Science and Technology, University of Natural Resources and applied Life Sciences, 1190 Vienna – Austria, gernot.zweytick@boku.ac.at

Part I
Conversion Operations

1

Mixing – Determining Mixing Parameters

Mustafa Bayram and Fahrettin Göğüş

OBJECTIVE AND LEARNING OUTCOMES

1. Examine mixing mechanisms and operations.
2. Review the principles of mixing.
3. Calculate the mixing index.
4. Calculate the optimum mixing time.
5. Compare different mixers based on their mixing indexes and times.
6. Evaluate energy consumptions of the mixers.

1.1. INTRODUCTION

The mixing of solids is an important unit operation in many industries in which a relatively uniform mixture is obtained from two or more components. Mixing is the dispersion of components, one throughout the other. It occurs in innumerable instances in the food industry and is probably the most common of all process operations.

A mixing process begins with the components, grouped together in some container, but still separated as pure components. Thus, if small samples are taken throughout the container, almost all samples will consist of one pure component. The frequency of occurrence of the components is proportional to the fractions of these components in the whole container. As mixing proceeds, samples will increasingly contain more of the components, in proportions approximating to

MUSTAFA BAYRAM, AND FAHRETTIN GÖĞÜŞ • Department of Food Engineering, Faculty of Engineering, University of Gaziantep,

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the overall proportions of the components in the whole container. Complete mixing could then be defined as that state in which all samples are found to contain the components in the same proportions as in the whole mixture (Figure 1.1).

Three mechanism types are often used to describe mixing performance: *diffusion (diffusive mixing),* but *not* molecular diffusion – an expanded bed of

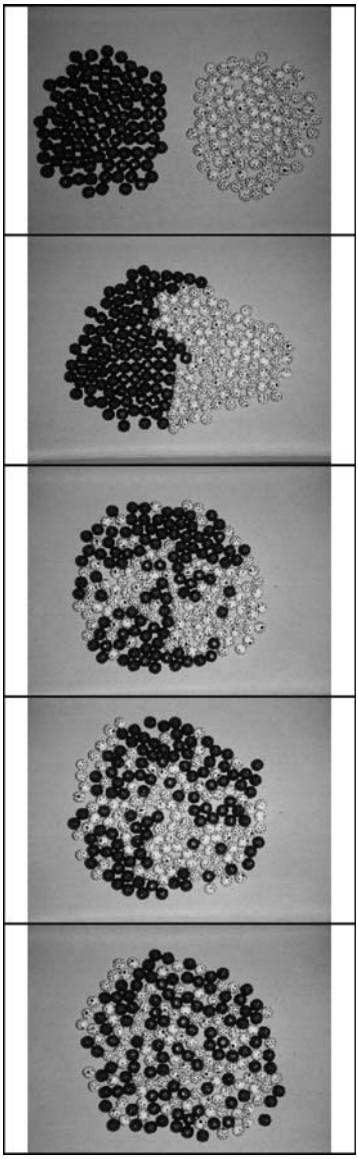

Figure 1.1. Stages in dark and light coloured beads to give a complete random mix.

free flowing material occurs with particles in random movements, *convection (convective mixing)* – when volumes, or regions, of the mix are moved en masse to different areas and *shear (shear mixing)*– mixing occurs along the slip planes between the regions of the particles. All the mechanisms may exist in a single mixer, but one or two may predominate. They depend on the type of mixer and on the properties of the particles. The mixer type needs to be right for the material mixed, e.g., cohesive powders are more likely to require shear (and convection) hence blades and ploughs are more appropriate than tumbling.

In general, for particle mixing the following *physical particle properties* should be considered. Monosize particles are easy to mix, provided they are free flowing, but segregation by size, density and rotational inertia are possible with free flowing powders possessing differences in these properties. Fine particles with high surface forces (diameters <100 μm and very high forces with diameters <10 μm), may need agglomerate breakage requiring high power, but can give good mixing of *cohesive* powders. *Aeration:* e.g., catalyst particles in gas fluidization, may undergo diffusion-like type mixing, which is a low-energy process, but with a risk of powder flooding. *Friability:* for delicate particles mixing by shear mechanisms would be inappropriate. *Explosion hazard:* an inert gas blanket is needed and low specific power input (low shear) is required. *Physiological hazard:* need to avoid airborne dust formation. *Adherence to surfaces:* easy to clean surfaces are needed and if a liquid-cleaning fluid is used then a new pollution problem may result.

It is not possible to achieve a completely uniform mixture of dry powders or particulate solids. The degree of mixing achieved depends on:

- The relative particle size, shape and density.
- The efficiency of the particle mixer for the components being mixed.
- The tendency of the materials to aggregate.
- The moisture content, surface characteristics and flow characteristics of each component.

Generally, materials similar in size, shape, and density are able to form the most uniform mixtures. Differences in these properties can also cause segregation during mixing or mechanical jiggling of the mixture. To provide good solid mixing the phenomenon to avoid, or overcome, is the tendency for particles to *segregate*. Segregation occurs in competition with mixing and prevents a perfect homogeneous powder blend to be obtained. Hence, the quality of a powder mixture depends upon the dynamic equilibrium between mixing and segregation, which in turn depends on the physical and chemical properties of the particles (Figure 1.2). Segregation occurs, for example, when a system contains particles with different sizes or densities. and motion can cause particles to preferentially accumulate into one area over another, e.g. when large particles work their way to the top of breakfast cereal, fines are found at the bottom of the packet. In contrast, motion of gases and miscible liquids due to flow (convection) provides mixing on a large scale and molecular diffusion is

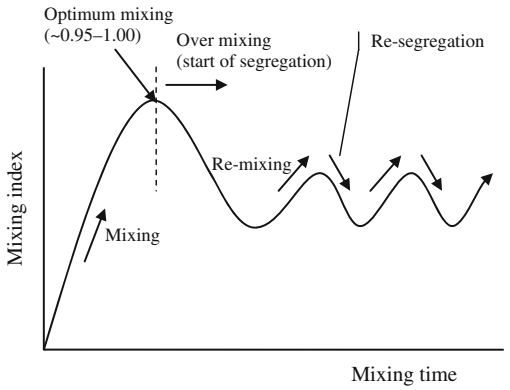

Figure 1.2. Ideal profile for a blending process (Brennan et al., 1990).

important for completing the process at the micro-scale level. A mixture of particles will never be as homogeneous as that of a fluid as particles tend to segregate, whereas fluid molecules tend to mix.

The duration of powder mixing in a process is frequently determined by experimental trials or by operator experience. The quality of a blended powder product can be expressed in terms of composition variance, which will decrease over time as mixing takes place (Figure 1.2). The most commonly used method to determine blend homogeneity is analysis of "grab" samples.

Experience shows that materials with a size >75 μm will segregate readily during mechanical jiggling of the mixture, but those below 10 μm will not segregate appreciably.

Means of overcoming segregation and poor mixing include:

- Comminution to smaller sizes.
- Use of powders with a narrow size distribution.
- Use of the same volume-average diameter for all components.
- Granulation.
- Coating processes.
- Controlled continuous mixing.

1.1.1. Mixing of Widely Different Quantities

The mixing of particles that vary substantially in size or in density presents special problems, as there will be gravitational forces acting in the mixer which will tend to segregate the particles into size and density ranges. In such a case, initial mixing in a mixer may then be followed by a measure of (slow gravitational) un-mixing and so the time of mixing may be quite critical.

Mixing is simpler when the quantities to be mixed are roughly in the same proportions. In cases where very small quantities of one component have to be

blended uniformly into much larger quantities of other components, it is best to split the mixing into stages, keeping the proportions not too far different in each stage.

1.1.2. Cohesive Powder Mixing

When mixing bread dough a lot of effort is required to stir the cohesive mixture but it does not segregate. However, only small pockets of mixture receive shear, and mixing, and the energy input is high. In commercial systems this means a powerful motor is necessary. Cohesive mixtures tend to be formed by fine particles and systems with binders present. Smaller particles lead to a larger number of particles for a given sample mass. As the number of particles goes up the variance between samples will go down. This is intuitive, as the limit would be to go to molecular mixing (very small particles) where the variance is zero between samples.

1.2. CALCULATIONS

1.2.1. What Sample Size Should Be Taken?

To take extreme cases, if the sample is so large that it includes the whole mixture, then the sample composition is at once average composition and there remains no mixing to be done. At the other end of the scale, if it were possible to take samples of molecular size, then every sample would contain only one or other of the components in the pure state and no amount of mixing would make any difference. Between these lie all of the practical sample sizes, but the important point is that the results will depend upon the sample size.

In many practical mixing applications, process conditions or product requirements prescribe suitable sample sizes. For example, if table salt is to contain 1% magnesium carbonate, the addition of 10 kg of magnesium carbonate to 990 kg of salt ensures, overall, that this requirement has been met. However, if the salt is to be sold in 2 kg packets, the practical requirement might well be that each packet contains 20 g of magnesium carbonate with some specified tolerance and adequate mixing would have to be provided to achieve it.

A realistic sample size to take from this mixture, containing 1000 kg of mixture, would be 2 kg. As mixing proceeds, the greater numbers of samples containing both components appear and their composition tends towards 99% salt and 1% magnesium carbonate.

It should be large enough so that the required amount of the smallest ingredient is easily measured, yet small enough so that you are confident that the quantities normally used will contain the ingredients in the required concentration (for example, pack size or less).

It can be seen from this discussion that the deviation of the sample compositions from the mean composition of the overall mixture represents a measure

of the mixing process. This deviation decreases as mixing progresses. A satisfactory way of measuring the deviation is to use the statistical estimator called the standard deviation. This is the mean of the sum of the squares of the deviations from the mean and thus it gives equal value to negative and positive deviation and increasingly greater weight to larger deviations because of the squaring. The "standard deviation" provides a satisfactory way of quantifying the extent to which the fractional concentration of a component scatters about its mean value in the various samples (Figure 1.3). It is given by Equation (1.1):

$$S = \sqrt{\frac{1}{N-1} \cdot \sum_{n=1}^{N} (X_n - \bar{X}^2)} \tag{1.1}$$

where $\bar{x}$ is given by Equation (1.2):

$$\bar{x} = \frac{1}{N} \sum_{i=1}^{N} x_i \tag{1.2}$$

where "σ" is the standard deviation, N is the number of samples taken (i.e. number of spot), x_1, x_2, ... x_n, are the fractional compositions of component "x" in the 1, 2, ... N samples and "$\bar{x}$" is the mean fractional composition of component "x" in the whole mixture. Equation 1.1 can be used for max. 30 sample ($N <$ 30), for the bigger sample number, it should be modified as ($1/N$).

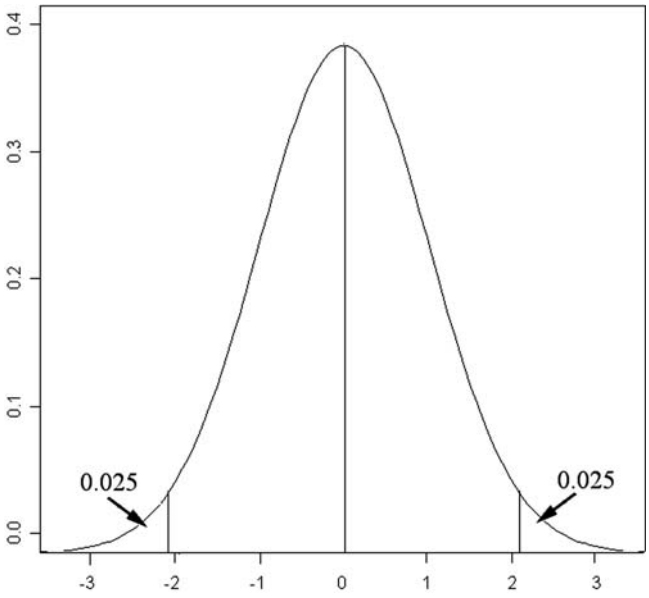

Figure 1.3. The normal probability distribution-symmetrical around the mean value.

Using Equation (1.1) values of σ can be calculated from the measured sample compositions, taking the "N" samples (number of spot) at some stage of the mixing operation. Often, instead of σ it is convenient to use σ^2, known as the variance of the fractional sample compositions from the mean composition (i.e. the *variance* of the mixture). For a perfect mixing σ (or σ^2) should be equal to zero. Lower standard deviations are found as the uniformity of the mixture increase ($\sigma_\infty = 0$). But $\sigma_\infty = 0$ cannot be achieved, but in efficient mixers the value becomes very low after a reasonable period.

1.2.2. How Long Should Ingredients Mix ?

1.2.2.1. Mixing Index

If particles are to be mixed, starting out from segregated groups and ending up with the *components randomly distributed*, the expected variances (σ^2) of the sample compositions from the mean sample composition can be calculated.

Consider a two-component mixture consisting of a fraction p of component P and a fraction q of component Q. In the unmixed state virtually all small samples taken will consist either of pure P or of pure Q. From the overall proportions, if a large number of samples are taken, it would be expected that a proportion p of the samples would contain pure component P. That is their deviation from the mean composition would be $(1 - p)$, as the sample containing pure P has a fractional composition 1 of component P. Similarly, a proportion q of the samples would contain pure Q, that is, a fractional composition 0 in terms of component P and a deviation $(0 - p)$ from the mean. Summing these up in terms of fractional composition of component P and remembering that $p + q = 1$ (Earle, 1983) gives Equation (1.3):

$$\sigma_0{}^2 = 1/N[pN(1-p)^2 + (1-p)N(0-p)^2] = p(1-p) \text{ (for } N \text{ samples)} \tag{1.3}$$

When the mixture has been thoroughly dispersed, it is assumed that the components are distributed throughout the volume in accordance with their overall proportions. The probability that any particle picked at random will be component Q will be q, and $(1 - q)$ that it is not Q. Extending this to samples containing "n" particles, it can be shown, using probability theory (Earle, 1983):

$$\sigma_r^2 = p(1-p)/n = \sigma_0^2/n \tag{1.4}$$

This assumes that all the particles are equally sized and that each particle is either pure P or pure Q. For example, this might be the mixing of equal-sized particles of sugar and milk powder. The subscripts o and r have been used to denote the initial and the random values of σ^2, and inspection of the formulae, Equation (1.3) and (1.4), shows that in the mixing process the value of σ^2 has decreased from $p(1 - p)$ to $1/n$ th of this value. It has been suggested that intermediate values between $\sigma_0{}^2$ and $\sigma_r{}^2$ could be used to show the progress of

mixing (Earle, 1983). Suggestions have been made for a mixing index (I_M) based on this assumption, for example:

$$I_M = (\sigma_0^2 - \sigma^2)/(\sigma_0^2 - \sigma_r^2) \tag{1.5}$$

During the course of the mixing process, I_M varies from 0 to 1. This measure can be used for mixtures of particles and also for the mixing of heavy pastes.

1.2.2.2. Rate of Mixing and Mixing Efficiency

The mixing index ought to be such that the rate of mixing at any time, under constant working conditions such as in a well-designed mixer working at constant speed, ought to be proportional to the extent of mixing remaining to be done at that time.

In mixing as in other rate processes, the rate is proportional to the driving force. The mixing index I_M is a measure of how far mixing has proceeded towards equilibrium. It has been found that for short mixing times the rate of change of I_M is directly proportional to $1 - I_M$ as:

$$\mathrm{d}I_M/\mathrm{d}t = k\,[1 - I_M] \tag{1.6}$$

where k is the mixing rate constant, which varies with the type of mixer, conditions e.g., rpm and the nature of the components and t (s) mixing time, and it can also be used to predict, for example, the times required to attain a given degree of mixing. By integrating from $t = 0$ to $t = t$ during which I_M varies from 0 to I_M, giving

$$[1 - I_M] = e^{-kt} \tag{1.7}$$

or

$$(I_M) = 1 - e^{-kt} \tag{1.8}$$

The equilibrium value of I_M is 1 (for the practical application I_M is taken as 0.95) therefore the driving force for mixing at any time can be considered to be $1 - I_M$ with rearranging and integrating between limits (from Equation (1.6).

$$t = (1/t)\,[\ln(1 - I_{M,0}) / (1 - I_{M,0})] \tag{1.9}$$

or from

$$t = \ln(I_M)/k \tag{1.10}$$

This exponential relationship, using (I_M) as the mixing index, has been found to apply in many experimental investigations at least over two or three orders of magnitude of (I_M). In such cases, the constant I_M can be related to the mixing machine and to the conditions and it can be used to predict, for example, the times required to attain a given degree of mixing.

It would be expected that small samples taken from various locations in the container at the start of mixing should contain close to 100% of either one or another of the components, so a plot of composition as a function of sample location would be very uneven. As mixing proceeds, successive plots

of the composition of samples taken from these same locations should become more uniform. Ideally, when mixing is complete all samples should contain the same percentage of each ingredient that added to the vessel at the start.

The mixing index at zero mixing is given by:

$$I_{M,0} = (n/(n-1)) \tag{1.11}$$

The efficiency of a mixer or blender (η_M) has been proposed as:

$$\eta_M = (\sigma/\sigma_0) \tag{1.12}$$

1.2.2.3. Mixed Particle Sizes and Size Analysis

In a sample of uniform particle diameter D_p the total volume of the particles is (m/ρ_p), where m and ρ_p are the total mass of the sample and the density of the particles, respectively. Since the volume of one particle is V_p, the number of particles in the sample n is;

$$N = [m/(\rho_p V_p)] \tag{1.13}$$

1.2.3. Energy Input in Mixing

Quite substantial quantities of energy can be consumed in some types of mixing, such as in the mixing of plastic solids. There is no necessary connection between energy consumed and the progress of mixing: to take an extreme example there could be shearing along one plane in a sticky material, then recombining to restore the original arrangement, then repeating which would consume energy but accomplish no mixing at all. However, in well-designed mixers, the energy input does relate to mixing progress, though the actual relationship has normally to be determined experimentally. In the mixing of flour dough using high-speed mixers, the energy consumed, or the power input at any particular time, can be used to determine the necessary mixing time. This is a combination of mixing with chemical reaction as flour components oxidize during mixing in air which leads to increasing resistance to shearing and so to increased power being required to operate the mixer.

1.2.4. Selection of Mixers

Selection of mixers must take into account any tendency towards segregation. This may be evaluated with a "heap test", in which a well-mixed material is poured through a funnel to form a heap. If the composition of samples taken from the outside varies significantly from compositions of samples from the center of the heap, the material is likely to segregate during mixing or later processing.

It is possible to obtain a good mixer quality, but a poor product quality, as there are other operations between mixing and use, such as emptying,

Table 1.1. Comparison of continuous and batch of free flowing powders (*italicised description indicates preferred option*)

Property	Batch	Continous
Capital cost:		
Mixer	Higher	*Lower*
Ancillary	*Lower*	Higher
Operating cost	Automation reduce labour costs but increase capital costs	*Fewer operators in general*
Flexibility	*More flexible*	One operation only
Proportion ingredients	*More confidence*	Can be OK but needs analysis
Handling and storage		*Not necessary useful for segregating mixes*
Capacity		*Much greater*
Quality control	*Batch identifiable*	Not so
Design knowledge	High	OK
Mixing efficiency	Similar if operated ok	

transportation and use, all of which may induce segregation. In a process analysis the permissible variation in the product quality must be known and its relation to quality from the mixer deduced. Batch mixing large quantities (up to 2 tonnes) reduces labour costs but as size goes up, so does time to reach desired quality, filling and emptying times per batch. Continuous mixing depends on metering rates, capacity of mixer, axial and radial dispersion performance. Loading two, or more, components together should reduce batch mixing time, but requires metering or practice. Table 1.1 compares batch and continuous mixing strategies. Mixers can be classified into two groups regarding segregation:

(a) **Segregating mixers** have mainly diffusive mechanisms, encouraging the movement of individual particles, making segregation more significant. Non-impeller type mixers tend to be of this type.
(b) **Less segregating mixers** have mainly convective mixing mechanisms. These are typically impeller types in which blades, screws, screws, ploughs, etc., sweep groups of particles through the mixing zone.

1.3. EXPERIMENTAL PROCEDURE

1.3.1. Apparatus

- Micrometer
- Timer

Figure 1.4. Tyler Sieves' Series.

- Graduated cylinder (100 ml)
- Graduated cylinder (25 ml)
- Analytical balance (+ /– 0.01 g)
- Sieve (1 mm hole diameter) (Frame diameter is approx. 20 cm, for bulk sieving) and (1 mm hole diameter) (Frame diameter is approx. 5 cm, for sample sieving) (Figure 1.4)

- Cone type (Figure 1.5) and V-shape tumbling mixer (Figure 1.6)

Tumbler Mixers

The tumbling action induces particles to roll and fall. The material is elevated beyond its angle of repose falling to the free surface giving a good mixing to free flowing materials e.g., tea, seeds. Easy cleaning, emptying, power consumption and wear are considered to range from low to moderate.

- The equipment operates by tumbling the solids inside a revolving vessel.
- It may be fitted with baffles, to assist mixing, or with internal rotating devices to break up agglomerates.

Figure 1.5. Cone type tumbling mixer; on the left, cone view; on the right, inside view of cone.

- It operates at speeds up to about 100 rpm (about half the critical speed at which the centrifugal force on the particles exceeds the pull of gravity).
- It has a working capacity of about 50–60% of its volume.
- It is best suited for gentle blending of particles with similar physical characteristics however segregation can be a problem.
- The equilibrium is generally reached in about 10–15 min.

- Ribbon type (screw conveyor) mixer

Horizontal Trough (Screw Conveyor) Mixers

An agitator mixes material in a trough with gentle mixing but with shear and impact. Not suitable for very cohesive materials, unless a dough is required.

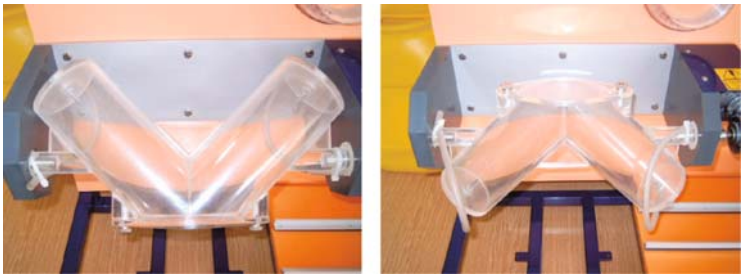

Figure 1.6. V-shape tumbling mixer on the left, mixer up view, on the right, mixer down view.

Figure 1.7. Ribbon type mixer.

Used for addition of small amounts to larger components, but can be difficult to clean.

- Consist of semi-cylindrical horizontal vessels in which one or more rotating devices (e.g., screw conveyors or a ribbon mixer) are located.
- In a typical ribbon mixer one ribbon moves the material slowly in one direction, while the other moves it quickly in the opposite direction, so there is a net movement of material and the system can be used as a continuous mixer.
- Particle damage can occur due to the small clearance between the ribbon and the vessel wall, and the mixer has a high power requirement.
- Segregation is less of a problem.

- Angle of repose instrument (Figure 1.8)

Figure 1.8. Apparatus for angle of repose.

Figure 1.9. Voltmeter and Amperometer.

- Voltmeter and amperometer (Figure 1.9)

1.3.2. Materials

- 1 kg semolina.
- 1 kg black fine bulgur wheat.

1.3.3. Procedure

Semolina and bulgur wheat both come from wheat differing only on their particle sizes.

Please follow the following steps:

1. Measure the hectolitre-weight (bulk density; weight the 100 ml graduated cylinder filled with semolina and bulgur) and angle of repose (flowing angles of the solids; fill the half of the angle of repose container, and then rotate it slowly until flowing of samples) for both cereals.
2. Then, standardize the particle size of the semolina and fine bulgur wheat using a 1 mm sieve. For the semolina and bulgur wheat, use the lower and upper parts of the 1 mm of sieve, respectively.
3. Determine the hectolitre-weights and the angles of repose of the sieved semolina and bulgur wheat.

4. Adjust the rpm of the mixer and record it.
5. Load the semolina and bulgur wheat into the Cone type tumbling mixer (500 g semolina + 500 g bulgur wheat).
6. Start the mixing operation at constant rpm. At the same time, start the timer ($t = 0$).
7. Measure the current intensity (A) and difference in electrical potencial ΔV (V).
8. After 2 min ($t = 2$ min), stop the mixing.
9. Collect five nearly equal amounts of spots (approx. 30 g of sample) from the different places of the cone mixer.
10. Measure and record their hectolitre-weights.
11. Sieve them using a sieve having 5 cm frame diameter (1 mm hole diameter). During 1 mm of sieving of the spots, the upper and lower will be bulgur and semolina, respectively.
12. Weigh and record the weight of the bulgur wheat and semolina individually.
13. Continue the mixing operation for the next 2 min ($t = 4$ min). Stop the mixing. Repeat Step 10.
14. Continue the mixing operation for the next 2 min ($t = 6$ min). Stop the mixing. Repeat Step 10.
15. Continue this procedure untill $t = 10$ min.
16. Repeat Steps 5–14 for the V-Shape tumbling and Ribbon type (screw conveyor) mixers.

(Note: Same experiments can be made at different rpm and sample quantities to determine the effect of mixer speed and sample quantities.)

1.4. DATA ANALYSIS AND QUESTIONS

1.4.1. Data Analysis

Calculate at $t = 0$, 2, 4 and 6 min:

1. Standard deviation
2. Coefficient of variation
3. Hectolitre-weight.
4. k-value.
5. Find mixing indexes (I_M) and plot I_M vs. time.
6. Calculate mixing time to reach the mixing index to 0.95.
7. Calculate mixing efficiencies.
8. Calculate the energy consumptions and power requirements of the mixers at $t = 0$, 2, 4, 6 and optimum mixing time.

Given:

For bulgur particle (assume sphere); $\rho_p = 1.24 \times 10^{-3}$ g/mm^3, $D_p = 1.2$ mm

For semolina particle (assume sphere);

$\rho_p = 1.11 \times 10^{-3}$ g/mm^3, $D_p = 0.4$ mm

(1.14) $$V_p = (4/3\,\pi\, r^3)$$

(1.15) $$P = IV$$

(1.16) $$E = Pt$$

where

V is voltage in volts, P is power in kWh, I is current intensity in ampere, t is time in hour.

1.4.2. Questions

1. What are the practical applications of the hectolitre-weight and angle of repose in the food industry ?
2. What was hectolitre-weight with the particle size, ash and protein contents of the wheat (unprocessed, raw wheat) ?
3. Before and after the sieving, you measured the hectolitre-weight and angle of repose of the semolina and bulgur wheat, which one was higher before and after sieving, why ?
4. Compare the hectolitre-weight changes during each operation.
5. Compare the ampere and voltage values for each operation.
6. What is the importance of ampere and voltage for the mixing ?
7. Which mixer is more efficient ?
8. In mixing, if the initial amounts of the samples are different, what will happen ?
9. In mixing, if the amount of one sample (Sample A) (e.g., 1 t) is higher than another (Sample B) (e.g., 2 g), how can you mix them ?
10. In a batch mixer, blending starch and dried-powdered vegetables for a soup mixture, the initial proportions of dried vegetable to starch were 40:60. If the variance of the sample compositions measured in terms of fractional compositions of starch was found to be 0.0823 after 300s of mixing, how much longer should the mixing continue to reach the specified maximum sample composition variance of 0.02 ? Assume that the starch and the vegetable particles are of approximately the same physical size and that a sample contains 24 particles.

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Some Web Sites of Interest

The Experimental Nonlinear Physics Group at the Univ. of Toronto describes experiments of unmixing sand and provides illustrations at http://mobydick.physics.utoronto.ca/sand.html.

Julio Ottino (Dept. of Chem. Engg., Northwestern Univ.) provides links to many of his papers on granular mixing. See http://pg.chem-eng.nwu.edu/mixing/.

The Hayes & Stolz Industrial Mfg. Co., Inc. uses a ribbon blenders in its Continuous Blender (see http://www.hayes-stolz.com/continumxr.htm) and in its Counterpoise Mixer (seehttp://www.hayes-stolz.com/cntrpoimxr.htm).

Continuous mixers – for example Littleford Day Inc., see http://www.littleford.com/km.html

Dispersing solids in gas to aid drying, extraction, etc. – Littleford Day Inc., see http://www.littleford.com/dvt.html

Cooling and mixing – Littleford Day Inc., see http://www.littleford.com/k.html

High shear equipment for granular and fibrous materials, etc. – Littleford Day Inc., see http://www.littleford.com/cb.html

Littleford Day – http://www.littleford.com

Eastern-Cleveland Mixers – http://www.emimixers.com

The Hayes & Stolz Industrial Mfg. Co. – http://www.hayes-stolz.com/

Food Processing Equipment Company – http://www.fpec.com

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2

Filtration I – Determining Filterability of Suspensions

Cecilia Hodúr

OBJECTIVES AND LEARNING OUTCOMES

1. Examine and determine the filterability of different suspensions (e.g., waster water, sludge).
2. Discuss and evaluate the effectiveness of preliminary treatments (e.g., flocculation, adsorption, polyelectrolytes, sedimentation).

2.1. INTRODUCTION

Waste water which has suspended materials could be removed from water by simple filtration, which is an especially effective unit operation in the removal of very small particles, like microorganisms (Earle, 1983). These filters could be used as a bio-reactor in waste treatment. The waste filtration process is usually performed using a medium such as sand or anthracite coal (Brennan et al., 1990).

Filterability is not a specific property of a suspension, but it is an interactive property between a suspension and some filter media. If the properties of one of these are kept constant (i.e., as a standard filter medium) the changes in filterability only reflect changes in the suspension (Brennan et al., 1990).

2.1.1. Filterability Index

A suspension is considered to be filterable if it can pass rapidly through a porous medium, giving a clear filtrate, with little clogging of the filter medium. The clogging is usually reflected in loss of permeability, which is shown as an increase in pressure drop or head loss.

CECILIA HODÚR • Department of Unit Operation and Environmental Technique, Faculty of Food Engineering, University of Szeged

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The Filterability Index (F) takes these factors into account and can be represented by the following equation:

$$F = \frac{HC}{vC_o\tau} \tag{2.1}$$

where H is the head loss (water column pressure in mm liquid gauge), C is the average filtrate quality, C_o is the inlet suspension quality, v is the approach velocity (volumetric flow rate per unit face area), and t is the filtration run time

As C/C_o is a ratio, any unit of quality can be used (e.g., turbidity, COD, total coliforms) provided they are used consistently for both the feed suspension and the filtrate.

A filtration process is considered to be adequate when the filterability index (F) is low. This is achieved with a low head loss (from clogging), a low permeate concentration and a high approach velocity (i.e., a high inlet concentration under a long operation time).

However, no particular significance can be attached to the actual numerical value of F, since values of F indicate relative filterability. For example, a pretreatment of a suspension which decreases F results in better filterability. A minimum value of F from a number of tests would indicate an optimum filterability, even though nothing could be inferred from the numerical value attached to this minimum F.

As the F number has not been derived rigorously from filtration theory, it cannot be used at extreme values or under boundary conditions. For example, although extremely high values of v (e.g., 2 $m \cdot min^{-1}$) might give low F values, they would probably lead to C/C_o values of 1, thus preventing filtration and clogging. However, negligible value of H would result with very coarse media (e.g., 5 mm in diameter), leading to C/C_o approaching 1 and again preventing filtration. Consequently the interpretation of Filterability Index values has to be made with the awareness of its limitations to practical conditions where filtration is expected to occur (Cleasby, 1969 and Matterson and Orr, 1987).

2.2. EXPERIMENTAL PROCEDURE

2.2.1. Filterability Apparatus

The apparatus for measuring the Filterability Index (Figure 2.1) consist of a Perspex column with a diameter of 38 mm and a length of 65 mm between the inlet and outlet connections so that liquid flows downwards through the column. At the base of the column, a 0.5 mm brass gauze mesh (i.e., B.S. 30 sieve mesh) retains the granular media. The cap on top of the column can be removed easily by unscrewing the knurled screw. It has a small air release screw at the top.

When a liquid suspension is introduced into the column from a 1.5 L capacity glass conical funnel, it flows from the base unit of the Perspex column

Figure 2.1. Filterability index unit.

through a needle-type flow control valve and a G. A. Platon Gapmeter flowmeter to drain it. The flow rate is indicated at the top of the float in the flowmeter (range 20–300cc·min^{-1}). The cap above the column and at the base unit below the column are made of rigid PVC and both are connected to glass tube liquid manometers. The difference in levels gives the head loss (liquid gauge) directly, although the manometer tubes are connected at the top to keep the air pressure above atmospheric pressure. The valve and tubing connectors are made of chrome-plated brass. All tubing are 9 mm diameter translucent plastic, except the manometer connections which are 7 mm in diameter.

2.2.2. Additional Apparatus and Materials

The following additional apparatus are required for the experiment (Figure 2.2):

- Nephelometer (Hach 2100A).
- One-liter glass beaker (to collect filtrate).

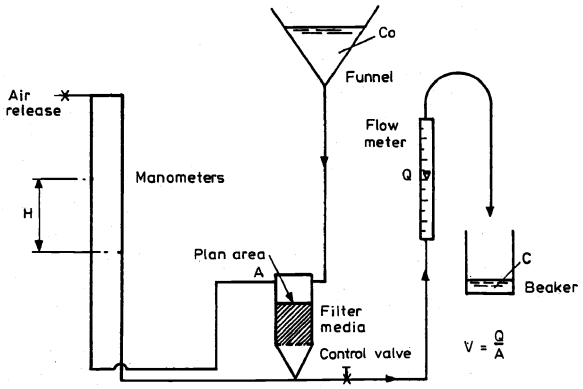

Figure 2.2. Schematic illustration of the filterability apparatus.

- Thermometer (–10 to 110°C).
- Stop Clock (reads seconds to minutes).
- 100-mm diameter Funnel (for reverse flow filling).
- Leighton Buzzard sand.
- Kaolin.
- Alum.

2.2.3. Procedure

1. Pre-sieve the filter media (Leighton Buzzard sand) to a uniform size fraction (the sand should pass through a 0.600 mm sieve (B.S. 25 mesh) and should be retained on a 0.500 mm sieve (B.S. 30 mesh), having therefore a mean particle size (D_p) of 0.55 mm.
2. Fill the apparatus with 40 mm depth of granular media of known porosity in the Perspex column (A).
3. Pour clear water up to the base of the inlet funnel.
4. Prepare a 100 $mg \cdot L^{-1}$ suspension of kaolin in tap water.
5. Add 5 $mg \cdot L^{-1}$ of alum to flocculate the suspension.
6. Mix vigorously the suspension for 15 s and then slowly stir it for 10 min at approximately 50 rpm.
7. Carefully pour the test suspension into the inlet funnel.
8. Adjust the flow control valve to give the required flow rate on the flow meter. A standard flow rate in water treatment would be 93 $cm^3 \cdot min^{-1}$ which corresponds to an approach velocity (v) of 8.2 $cm \cdot min^{-1}$. (Note: you can calculate it with the plan area of the filter column.) However, other flow rates can be used if different aspects of filterability are being investigated.

9. Time must be allowed for the displacement of the clean liquid above the inlet surface of the bed of porous media. At 93 $cm^3 \cdot min^{-1}$, this displacement time is approximately 40 s.
10. Start the clock to obtain the filterability test run time (t). Additional time must be allowed for displacement of the clean liquid in the filter pores, in the base unit of the column and the outlet tubing including the flow meter (for 93 $cm^3 \cdot min^{-1}$ this time is approximately 75 s).
11. Insert the outlet tube into the 1L beaker to collect the filtrate for quality analysis after the liquid has been displaced.
12. Adjust the displacement times for other flow rates. For example, at a flow rate of 120 $cm^3 \cdot min^{-1}$, the inlet displacement time (before starting the timing clock) should be $(93/120) \times 40 = 31$ s; the outlet displacement time (before collecting filtrate) should be $(93/120) \times 75 = 58$ s.
13. Take a sample from the inlet funnel for analysis while the suspension is filtering through the porous media. For analysis, a nephelometer should be used to measure the difference between the haze of original suspensions and the filtered liquid.
14. Insert a thermometer in the beaker collecting the filtrate, and record the temperature. This is needed for future comparisons, as marked differences in temperature (more than 5°C) between different tests would produce different results. All other conditions being equal, higher temperatures produce better filterability (i.e., lower F number).
15. Check the flow meter and adjust the control valve during the filtration operation to maintain a constant flow rate. Due to the clogging of the filter media and the falling inlet level, the flow rate will tend to decrease, requiring a gradual opening of the control valve.
16. Check the difference in the manometer levels. This difference is due to the head loss across the porous media and the support gauze mesh. However, the head loss due to the support gauze mesh is negligible compared with that of the porous media when it becomes clogged.
17. Read the two manometer levels when the inlet suspension level has fallen to the base of the inlet funnel (the difference is H) and stop the stop clock, noting the time (t), and then close the flow control valve. At the standard flow rate of 93 $cm^3 \cdot min^{-1}$ the filterability test run time should be about 10 min.

2.2.4. Calculations

$$\text{Solid volume of sand} = (1 - \varepsilon)V$$

where

V – suspension volume. F depends on the volume. At first the solution is clear and F is very low, but with time it starts increasing leading to an

Table 2.1. Data for *F* evaluation (Cleasby, 1969)

Description	Characteristics
Column	
Length (L)	40 mm
Diameter (d)	38 mm
Leighton Buzzard sand	
Density (ρ)	2650 $kg \cdot m^{-3}$
Porosity (ε)	0.4

increase of F because leading to an increase of F the volume of particulates (suspension) will be separated by the sand (filter media) Causing an increase in the head loss (H) . ε – is the porosity, it is a volume ratio between the unloaded/free volume and the whole volume of filter media and it is a given parameter.

2.3. DATA ANALYSIS AND QUESTIONS

1. Calculate the Filterability Index from data obtained from experiments A and B presented in Appendix A.2. What do these results illustrate?
2. Can alum be used for improving filterability? Why?
3. How would you determine the optimum quantity of filter media?
4. Explain how you would measure the effect of temperature?
5. Discuss the effect of temperature and velocity of filtration.

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3

Filtration II – Using a Plate-and-Frame Filter

Marie-Laure Lameloise

OBJECTIVES AND LEARNING OUTCOMES

1. Understand the operation of a plate-and-frame-filter press.
2. Verify cake filtration laws at constant flow rate, at constant pressure.
3. Learn how to size a filter through measurement of cake specific resistance on a laboratory filtration cells.

3.1. INTRODUCTION

Cake filtration is adapted to filtration of solid–liquid suspension containing more than 0.1% particles (w/w). The particles accumulating on the surface of a cloth or sheet, form a porous layer or "cake" which becomes the main filtering medium.

When a fluid passes through a porous medium, the relationship between pressure drop, ΔP (Pa), and flow rate, dV/dt ($m^3 s^{-1}$), can be described by Darcy–Carman's law (Perry and Green, 1997, Orr, 1979 and Lameloise, 2002):

$$\Delta P = \frac{1}{S}\left(\frac{dV}{dt}\right) \cdot \eta \cdot R \tag{3.1}$$

where S is the cross-sectional area (m^2), η is the dynamic viscosity of the fluid (Pa·s), R is the flow resistance (m^{-1}). In cake filtration, the total pressure ΔP can be decomposed into the pressure drop across the cloth (Equation 3.2), and across the cake (Equation 3.3):

$$\Delta P_1 = \frac{1}{S}\left(\frac{dV}{dt}\right) \cdot \eta \cdot R_S \tag{3.2}$$

$$\Delta P_2 = \frac{1}{S}\left(\frac{dV}{dt}\right) \cdot \eta \cdot R_G \tag{3.3}$$

MARIE-LAURE LAMELOISE • Ecole Nationale Supérieure des Industries Agricoles et Alimentaires, Département de Génie Industriel Alimentaire, mlam@ensia.fr

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where R_S is the cloth resistance (m^{-1}), R_G is the cake resistance (m^{-1}), whereas R_S is constant (and generally negligible compared to R_G), and R_G increases as the cake gets thicker according to (Equation 3.4):

$$R_G = \frac{r \cdot c \cdot V}{S} \tag{3.4}$$

where c is the concentration of the suspension (kg solid/m^3 of filtrate), r is the specific cake resistance (mkg^{-1}) and is a key parameter for project design and scale-up, and V is the volume of suspension (m^3).

The total pressure drop across the filter can then be described by:

$$\Delta P = \left(\frac{\eta c r}{S^2} V + \frac{\eta R_S}{S}\right) \frac{\mathrm{d}V}{\mathrm{d}t} \tag{3.5}$$

At a constant flow rate (for example if the filter is fed with a volumetric pump), $\mathrm{d}V/\mathrm{d}t = Q$. Eq. 3.5 can be modified to:

$$\Delta P = \frac{\eta c r Q^2}{S^2} t + \frac{\eta R_S Q}{S} \tag{3.6}$$

The total pressure drop across the filter increases linearly with time. At constant pressure drop:

$$\Delta P \mathrm{d}t = \frac{\eta c r V \mathrm{d}V}{S^2} t + \frac{\eta R_S \mathrm{d}V}{S} \tag{3.7}$$

$$\frac{\eta c r V^2}{2S^2} + \frac{\eta R_S V}{S} - t\Delta P = 0 \tag{3.8}$$

The filtration curve $V = f(t)$ is parabolic, as the flow rate decreases against time. A linear relationship can be obtained by expressing t/V against V, which can be represented as:

$$\frac{t}{V} = \frac{\eta c r}{2S^2 \Delta P} V + \frac{\eta R_S}{S \Delta P} \tag{3.9}$$

3.2. EXPERIMENTAL PROCEDURE

3.2.1. Laboratory Filtration Cell

A filtering funnel (Büchner type, 8 cm diameter, 1 L capacity) equipped with a stirrer is connected to a 1 L graduated test glass tube, which is in turn connected to a vacuum pump equipped with a vacuum gauge. This device allows filtration at constant ΔP, however, this is limited to pressures between 0.8 and 0.9 bar. The funnel may be insulated from the test glass tube by a tap. In order to protect the sintered glass bottom of the funnel, disks are punched in cellulose sheets through an adequate hollow device. For each 100 ml, the time of filtration is measured with a stop clock.

3.2.2. Plate-and-Frame COFRAM-SEITZ Filter Press

The plate-and-frame sheet filter COFRAM-SEITZ (Figure 3.1, Table 3.1) is similar to the standard filter press, with the textile cloth replaced by a thick disposable filter medium of (SEITZ cellulose).

It is commonly used in clarification, where the quantity of solids to be retained is very small (e.g., filtration of beer, some wines, vegetable oil) or in microbial stabilization (e.g., sugar syrups) (Rivet, 1981, Lameloise, 2002). However, due to the size of the kieselguhr particles and the concentration of the slurry, the application proposed here relates to the *cake filtration mechanism* (and not to the in-depth filtration). This kind of filter is composed of alternating vertical square plates and frames. Hollow frames, covered on both sides by sheets of cellulose, provide space for cake accumulation during filtration. Perforated plates allow filtrate to be drained toward outlet.

Two manometers that are located at the inlet and outlet of the filter show relative pressure. As the filtrate is drained, the outlet manometer should

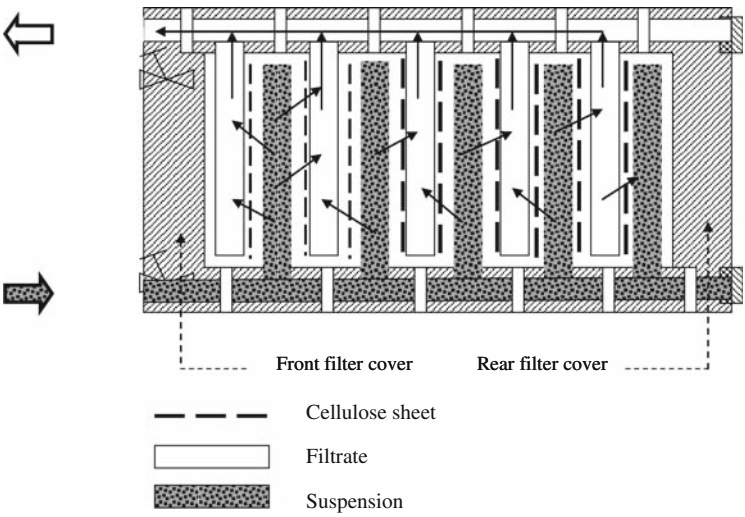

Figure 3.1. Schematic illustration of a Plate-and-frame filter press.

Table 3.1. Main characteristics of the COFRAM-SEITZ filter used

Description	Characteristics
Sheet size	20 × 20 cm
Effective filtration area	0.03 m^2
Maximum numbers of sheets in the filter	25
Maximum inlet pressure	10 bar
Maximum ΔP between inlet and outlet	3 bar

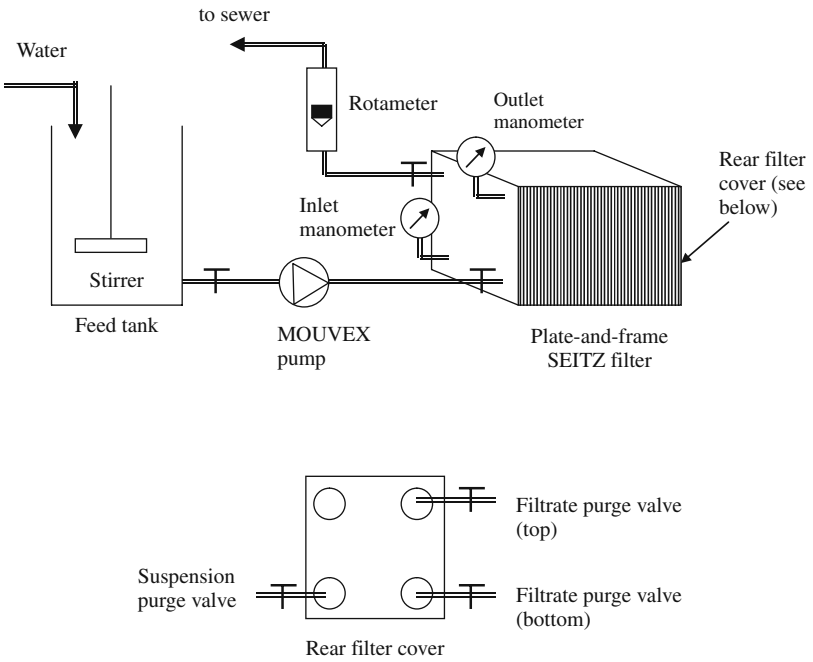

Figure 3.2. Pilot implementation.

normally indicate "zero" whereas the inlet manometer gives a straightforward measurement of ΔP.

The rear filter cover is equipped with three valves used for purging the filter: one on the suspension circuit, two on the filtrate circuit.

The kieselguhr suspension is prepared in a feed tank (200 L capacity) and continuously stirred. It is sent to the filter through a volumetric pump (Mouvex type) with adjustable speed. The filtrate goes through a rotameter which ensures instantaneous flow rate $\frac{dV}{dt}$ to be measured (Figure 3.2).

Continuous stirring is necessary to avoid sedimentation in the feed tank as the feed suspension would no more be controlled.

The use of a volumetric pump allows the filter to be fed at constant flow rate in a first stage. When pressure drop in the filter has reached a predetermined value, it is kept constant in a second stage by continuously decreasing the flow rate through manual action on the pump speed variator.

3.2.3. Procedure

This protocol is composed of two sequential parts determination of cake specific resistance: (i) with a laboratory filtration cell; and (ii) a pilot plant plate and frame filter.

3.2.3.1. Evaluation of Specific Cake Resistance with a Laboratory Filtration Cell

Considering the limited sample volume (1 L) that will be collected for the experiment, laboratory tests should be carried out with a 10 g L^{-1} suspension in order to build a cake, thick enough to be used as a filter. The experiment is carried out twice using sheets of different permeability: for example K-250 and K-1000 from COFRAM-SEITZ.

1. Prepare a 3 L suspension of kieselguhr.
2. Switched on the vacuum pump.
3. Fill the funnel with the suspension and then, open the valve when constant vacuum is achieved in the test glass ($\Delta P \approx 0.85$ bar).
4. Begin measuring time (t) when 100 ml filtrate have been collected (after opening the valve, a slight temporary decrease in vacuum is observed which will stabilize when the first 100 ml of the filtrate passes through the system).
5. Register the time at intervals of 100 ml. When 1 L filtrate has been collected, the experiment is stopped.

3.2.3.2. Filtration of Kieselguhr Solutions with the Plate and Frame Filter

(1) Fill the feed tank with water.

(a) *filter implementation*

Cellulose sheets are put on each side of the frames. With three sheets to place, this gives: one blind plate (special extremity plate), one sheet, one frame, one sheet, one blind frame (special extremity frame). The sheets are placed in such a way that the reference is against the plate. The rear filter cover is pushed with all the elements against the front filter cover and the whole is pressed.

(b) *purging the filter*

Caution: The pump should never turn without feed.

All purge valves are closed. The inlet valve (suspension) should be opened and the outlet valve (filtrate) closed. Feed pump is switched on. Open and close successively the three purge valves (in the following order: suspension, bottom filtrate, top filtrate) and the outlet valve, until the liquid flows without any air bubble. Press again the whole filter to account for sheet expansion.

Set the flow rate at 400 L h^{-1} and ensure that outlet manometer indicates zero. Straightforward ΔP measurement is obtained from inlet manometer. A zero value is obtained, proving that there is no pressure drop in the filter until the cake begins to build.

(2) Switch off the pump and turn off the vessel valve.

(c) *experiment*

The feed tank is filled with 200 L water. Four-hundred grams of kieselguhr is dispersed through stirring, giving a 2 $g \cdot L^{-1}$ suspension.

Open the outlet tap of the tank. Switch on simultaneously pump and chronometer.

Adjust flow rate at 400 $L \cdot h^{-1}$ and maintain constant by actuating the pump speed variator if necessary. Register ΔP increase with time.

When $\Delta P = 1$ bar, pressure drop is maintained by actuating the pump speed variator. Measure the flow rate that decrease with time.

The experiment is stopped when the flow rate has decreased below 220–230 $L \cdot h^{-1}$.

(d) *cake drying*

Water in the cake is displaced with compressed air blown co-currently. All the valves are closed except suspension purge and filter bottom purge. Compressed air is sent by suspension purge and water is evacuated by filtrate bottom purge. Open the filter and verify whether the cake layers are regular and homogeneous on each sheet. Sheets and cakes may then be discharged.

(e) *filter washing*

Wash all the installation, filter, pump and tank with water. Periodically ensure that manometers are not clogged.

3.2.4. Calculations

1. Determining cake specific resistance on laboratory filtration cell

For each experiment, $\frac{t}{V} = f(V)$ is drawn. Parallel lines should be observed. Specific resistance r is calculated from the slope through Equation (3.2). The average value is used for the following calculations.

2. Calculation of S for the pilot experiment

At pilot scale, a 2 $g \cdot L^{-1}$ kieselguhr suspension is to be filtered, at first at constant flow rate (400 $L \cdot h^{-1}$) and then at constant pressure. The filtering surface is calculated so that total pressure drop ΔP should reach 1 bar after 10 min filtration at a constant flow rate of 400 $L \cdot h^{-1}$.

S is obtained through equation (3.1), assuming that sheet resistance is negligible (this will be verified at the beginning of the experiment). This filtering surface is to be implemented with sheets of 0.03 m^2 effective surface.

3.3. DATA ANALYSIS AND QUESTIONS

1. Plot $\Delta P = f(t)$ for the first stage at constant flow rate. Verify if the relationship is linear (cake filtration law at constant flow rate) and determine r from the slope.
2. Plot $\frac{dV}{dt} = f(t)$ for the second stage at constant ΔP.

3. Calculate $\frac{t}{V} = f(V)$ through numerical integration for the second stage. Verify if the relationship is linear (cake filtration law at constant ΔP) and determine r from the slope.
4. Confirm the validity of the cake filtration law.
5. Compare the different values obtained for r (laboratory filtration cell and plate and frame filter). Conclude on the validity of lab cell experiments for scaling-up (Purchas, 1977).

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4

Centrifugation – Separating Cream from Milk

Rui Costa and David Gomes

OBJECTIVES AND LEARNING OUTCOMES

1. Recognize the fundamentals of centrifugation and review the importance of milk composition on centrifugation.
2. Operate a centrifuge at a pilot scale that is used for the production of cream and skim milk.
3. Calculate the critical diameter for separation.
4. Compare the values obtained for critical diameter with given distributions of milk fat globules.

4.1. INTRODUCTION

Centrifugation is a separation operation used in the food industry for the treatment of milk, principally for the standardization of milk and milk products, and in the production of cream and/or skim milk. This operation is a separation process that uses centrifugal force to separate two mixed liquids or insoluble solids from liquids (suspension). In this case, the fat globules separate from the rest of the milk suspension. The original milk feed (3.7% fat) is separated into a cream portion (higher than 30% fat) and a skim milk portion (around 0.05% fat). The milk should be at a temperature around 40°C before entering the centrifuge.

RUI COSTA • Escola Superior Agrária de Coimbra, Instituto Politécnico de Coimbra, ruicosta @esac.pt. DAVID GOMES • Escola Superior Agrária de Coimbra, Instituto Politécnico de Coimbra

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4.2. EXPERIMENTAL PROCEDURE

4.2.1. Apparatus and Materials

The centrifuge used in this experiment is a disc centrifuge (Figure 4.1). A feed (of milk) is supplied from the bottom of the stack of discs and the fluid is separated into two phases: a light phase (cream) and a heavy phase (skim milk). The number of discs used in the experiment is greater than that represented in Figure 1. Each disc contains holes that are aligned to form flow channels for milk entering from the bottom of the centrifuge to be fed to the rest of a disc surface. The separation occurs when the fluid flows through the spaces between the discs.

The following equipment and materials are required for the experiment:

- Disc centrifuge.
- Milk analyzer (or a protocol to measure the fat content).
- Densitometer.
- Stop clock (reads seconds to minutes).
- Thermometer.
- Test tube.

4.2.2. Procedures

In a typical laboratory experiment demonstrating the centrifugation of milk, each group of students uses a different flow rate and measures the flow rates of milk (feed) and of cream and its fat contents. After running the experiment,

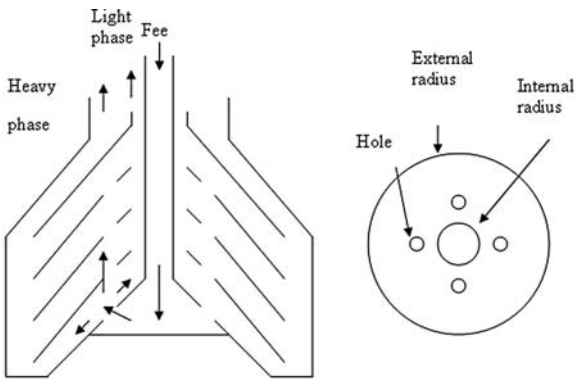

Figure 4.1. Simplified scheme of (a) a disc centrifuge and (b) a disc.

students use the measured flow rate values to calculate the critical diameter of fat globules and compare it to published results of fat globule distributions.

1. Before centrifugation.
   1. Keep a sample of milk for analysis.
   2. Preheat the milk to 30–35°C (heat exchanger).
2. Setting up of the centrifuge.
   1. Mount the discs on the centrifuge.
   2. Turn on the centrifuge and wait until the working rotation speed is achieved.
   3. Feed the centrifuge with preheated raw milk and adjust the milk flow rate entering the centrifuge and check the temperature.
3. Operation
   1. Measurements: flowrate, milk temperature, milk density, fat content.
   2. Measure the milk flow rate using a chronometer and a test tube.
   3. Collect the cream and skimmed milk streams and measure the cream flow rate.
4. Analysis of milk and centrifugation products
   1. Determine the raw milk density and fat content.
   2. Determine the density and fat content of the skimmed milk and the cream (using a chronometer and a test tube).

4.2.3. Calculations

The mechanical analysis of this operation (Foust et al., 1980) is similar to a sedimentation process that uses gravity force. The net force that results in the separation of two phases with different densities is a balance between the centrifugal force of the heavy liquid portion (or solid), the buoyancy force and the drag force:

$$\rho_s V_s R\varpi^2 - \rho_L V_s R\varpi^2 - \frac{C_A v^2 \rho_L S}{2\rho_s V_s} = \rho_s V_s \frac{dv}{d\theta} \tag{4.1}$$

where s is the solid (or heavy phase – skim milk, in this case), L is the liquid (or light phase – fat globules, in this case), ρ is the density, V is the volume, R is the radius at some point in the centrifuge, ω is the angular velocity of the centrifuge, C_A is the drag coefficient, S is the particle area normal to the flow direction, and θ is the time.

Equation (4.1) can be represented in terms of acceleration:

$$\frac{dv}{d\theta} = R\varpi^2\left(1 - \frac{\rho_L}{\rho_s}\right) - \frac{3C_A v^2 \rho_L}{4 D_s \rho_s} \tag{4.2}$$

When the milk enters the centrifuge, the heavy particles will be pushed to the exterior and light particles to the interior of the center. Their velocity will

increase due to the centrifugal force and the acceleration will decrease due to the increase of the drag force. When the acceleration is null the velocity will have the maximum value (terminal velocity). Equalizing the last equation to zero and assuming laminar flow will give the following equation for the terminal velocity of the particles (in milk centrifugation, the fat globules) being separated:

$$v_t = \frac{(\rho_s - \rho_L) D_s^2 R\varpi^2}{18\mu_L} \tag{4.3}$$

This is the general equation for calculating the radial velocity a particle (v_t). However, when light particles flow over the heavy fluid, the viscosity is not the light phase viscosity but the heavy phase viscosity. Solving this equation for D_s, and substituting v_t for $r/(V/Q)$ (r is the distance that the fat globule must dislocate in order to be separated from the heavy phase – in this case, the radius of the holes in the discs; V is the volume of the centrifuge and Q is the flow rate, and thus V/Q is the residence time), the critical diameter of particles for separation can be determined by:

$$D_{s,crit} = \sqrt{\frac{18\mu_L}{(\rho_s - \rho_L) R\varpi^2} \frac{r}{V/Q}} \tag{4.4}$$

The critical diameter signifies that half of the particles with this diameter will migrate to the heavy phase and the other half to the light phase. Thus, fat globules with higher diameters than $D_{s,crit}$ will be separated. Based on this value the proportion of fat to be separated from the milk can be predicted if data of the fat globules diameter distribution are also available.

The minimum diameter for non-homogenized milk is around 0.7 μm (Walstra et al., 1999). Values for critical diameter higher than this would indicate that not all the fat will be separated. It can be easily observed from Equation (4.4) that increasing the flow rate increases the critical diameter which results in less fat being separated from the milk.

4.3. DATA ANALYSIS AND QUESTIONS

(1) Calculate milk and cream flow rates using results obtained from the experiment in Appendix A.4.
(2) Plot these values against the fat content of the cream and explain any observed differences.
(3) Calculate the critical diameter of the fat globules for each milk flow rate used and compare these values with examples of fat globule distributions (see Walstra et al., 1999).

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5

Concentration – Direct Osmotic Concentration of Liquid Foods

Harris N. Lazarides

OBJECTIVES AND LEARNING OUTCOMES

1. Review the application of membrane technology in food process operations.
2. Assess the use of minimal (low temperature) processing in protecting quality characteristics (i.e., taste, aroma, color, nutrition) and minimize energy input during the concentration of liquid foods.
3. Evaluate the impact of crucial process parameters on process performance.
4. Use experimental data to model a membrane separation process.

5.1. INTRODUCTION

Evaporative concentration techniques, used in concentrating liquid foods (i.e., fruit and vegetable juices) are commonly linked to quality deterioration, due to high temperatures and phase change associated with evaporative concentration techniques. Alternative techniques, such as direct osmotic concentration (DOC), have been developed to reduce quality losses associated with more traditional food process operations.

Direct osmotic concentration uses hydrophilic, reverse osmosis type membranes to concentrate liquid foods without a phase change, at moderate (room) temperatures, under minimal pressures (i.e., pressure is not used

HARRIS N. LAZARIDES • Department of Food Science and Technology, School of Agriculture, Aristotle University of Thessaloniki, lazaride@agro.auth.gr

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as a driving force). Under a large osmotic pressure difference, water is removed in liquid form (i.e., no phase change) through the membrane into a concentrated (NaCl) brine solution, which is only subject to dilution, and therefore, reconstitution is an easy task that can take place with a simple evaporation step.

5.2. EXPERIMENTAL PROCEDURE

5.2.1. Apparatus and Materials

Direct osmotic concentration should be conducted using liquid foods, such as juices, that are preferably free of suspended solids (to avoid membrane fouling). Model fluids of varying sugar solutions can also be used to demonstrate the operation of this process. In case of a real food system with suspended solids, a filtration step (i.e., macro- or ultrafiltration) can precede the DOC process. The impact of increasing driving force (osmotic pressure difference) on water flux (water removal rate through the membrane) can be examined by using an osmotic medium, such as a concentrated solution of brine (i.e., NaCl solution) at varying concentrations. Typical membranes used are RO type membranes, with a high NaCl rejection index (>98%), preferably with a very thin supporting layer, to allow for minimal mass transfer resistance leading to increased flux rates.

The experimental set-up consists of two fluid circulation loops and the membrane module (Figure 5.1). The circulation loop on the left refers to the fluid food to be concentrated, while the right loop refers to the osmotic medium. The morphology of the two covers of the osmotic module ("cell") promotes turbulent flow and close contact conditions on both sides of the membrane.

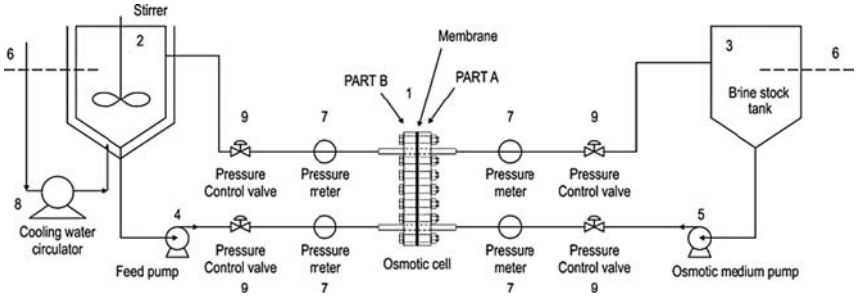

Figure 5.1. Schematic diagram of experimental set-up (Dova et al., 2006a).

Figure 5.2. Experimental direct osmotic rig (Dova et al., 2006a).

The experimental rig consists of the following parts (Figure 5.2):

- A cylindrical jacketed feed ("juice") tank, constructed from stainless steel (SS 316) with an internal diameter of 0.19 m and a height of 0.31 m that consists of a stirrer adjusted to the top of the tank to ensure raw materials are well mixed. Temperature can be controlled by circulating either a heating fluid or a coolant through the tank jacket.
- A cylindrical osmotic medium tank, constructed from stainless steel (SS 316) with an internal diameter 0.295 m and height 0.295 m. A round flat piece of SS 316 (diameter 0.26 m; height 0.02 m) is placed at the bottom of the tank to prevent swirling, which could create unstable flow conditions or harm the osmotic medium pump.
- A centrifugal feed pump (EBARA model CDM 70/7 Italy) with a 0.55 kW motor that can deliver between 20 and 80 $L \cdot min^{-1}$ of water.
- An osmotic medium pump with similar characteristics as that of the centrifugal feed pump.
- Two Pt-100 thermocouples with digital output, connected to the feed and osmotic tanks respectively.
- Four analog pressure gauges to measure the inlet and outlet fluid pressures before and after the osmotic module.
- A centrifugal pump, to circulate cooling water in the product tank jacket (RS 25/50r WILO – Germany).

- Four flow and pressure control ball-type valves; two for controlling pressure at the feed inlet/outlet and other two for the osmotic medium line.
- Plastic reinforced pipes with internal diameter 8 mm for the interconnections of the parts of the above mentioned experimental rig.

5.2.2. Procedures

Experimental conditions for comparative treatments are shown in Annex 5. The following additional experimental conditions were established in these experiments (Table A5.1 and Table A5.2). Experiments can be planned to study the impact of several process parameters (i.e., temperature, concentration, viscosity, flow rate, membrane characteristics) on process performance (i.e., flux, overall mass transfer coefficient) and membrane suitability for the specific concentration task. Flux values, expressed in $kg \cdot m^{-2}\ h^{-1}$, offer an objective basis for performance evaluation. They can be obtained from water removal rate (rate of mass increase of osmotic medium or mass decrease of raw material, $kg\ h^{-1}$) divided by total membrane area (m^2). Flux data for experimental treatments listed in Table A5.1 can be found in Table A5.2. This data can be correlated to process conditions to explore the impact of process conditions.

5.2.3. Calculations

Based on measured flux values, calculation of the *overall mass transfer coefficient* (U) is carried out by using the fundamental mass transfer equation:

$$U = \frac{\text{Flux}}{\Delta\Pi - \Delta P} \tag{5.1}$$

where Flux is the flow rate ($kg\ m^{-2}\ h^{-1}$), and $\Delta\Pi$ is the osmotic pressure difference across the membrane (bars).

In case of advanced courses, flux values can also be used to determine the constants of the physical model, describing the specific process. The following model has been proposed for Osmotic flux (F) in a DOC process (Dova et al, 2006b):

$$F = \frac{(\Delta\Pi - \Delta P)}{\left(C_1 \mu_1^{N_1} F_1^{S_1} + 1/A + C_2 \mu_2^{N_2} F_2^{S_2}\right)} \tag{5.2}$$

where Flux is the flow rate ($kg\ m^{-2}\ h^{-1}$), $\Delta\Pi$ is the osmotic pressure difference across the membrane (bars), $\Delta\Pi$ is the osmotic pressure difference across the membrane, ΔP is the hydraulic pressure differential across the membrane, μ_1, μ_2

are viscosities of osmotic medium and feed liquid, F_1, F_2 are mass flow-rates of osmotic medium and feed liquid, A is the water permeation coefficient, and C_1, C_2, N_1, N_2, S_1, S_2 are positive constants.

5.3. DATA ANALYSIS AND QUESTIONS

1. Calculate and determine overall mass transfer coefficients using flow rate data presented in Appendix A.5.
2. Discuss the effect of different treatments on calculated values.
3. Based on the data of Table A5.2 create appropriate correlations to delineate the impact of crucial process parameters on process performance (i.e., water flux).
4. Compare relative importance of process parameters and indicate proper interventions to improve process efficiency.
5. How would you plan an experiment to differentiate between the impact of temperature and that of viscosity?
6. Apply the proper optimization program to determine the constants of (Equation 5.2).

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6

Osmotic Dehydration – Vacuum Impregnation of Fruit

Eugenia Martin-Esparza and Chelo Gonzalez-Martinez

OBJECTIVES AND LEARNING OUTCOMES

1. Review the technique of vacuum impregnation.
2. Examine the optimization of the operation based on raw material characteristics and the final desired product.
3. Evaluate the influence of the impregnation solution on the product physicochemical changes.

6.1. INTRODUCTION

Vacuum impregnation of porous foods implies the partial substitution of the gas phase, in the intercellular spaces by an external liquid (i.e., impregnation solution), by imposing low pressures in the system (porous food immersed in a liquid phase) followed by the restoration of atmospheric pressure. Different solid–liquid operations, such as salting processes (González-Martínez et al., 2001; Barat et al., 2001a,b; Andrés et al., 2001a), fruit candying (Barat et al., 2001c), acidification, and the addition of preservatives, can be improved by reducing the processing time, depending on the product effective porosity and mechanical properties (Andrés et al., 2001b).

Compositional and structural changes that occur as a consequence of this operation have to be considered as they induce physico-chemical changes in the product, thus affecting the quality parameters. The desired food ingredients

EUGENIA MARTIN-ESPARZA AND CHELO GONZALEZ-MARTINEZ • Department of Food Technology, Universidad Politécnica de Valencia, • Department of Food Technology, Universidad Politécnica de Valencia, cgonza@tal.upv.es

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can be introduced directly into the product in a controlled way by using vacuum impregnation. The introduction of cryoprotectant solutes in a short time into porous fruits has been seen to make them more suitable for resisting damages caused by the frozen–thawing processes (Martínez-Monzó et al., 2001).

Mass transfer is governed by the hydrodynamic mechanism, which involves the inflow of external liquid throughout the capillary pores, controlled by the expansion/compression of internal gas. At the initial stage, internal water pressure inside the intercellular spaces (pores) is lower than atmospheric pressure, allowing external liquid to penetrate into the food by capillarity until pressure equilibrium is reached. When applying vacuum conditions (vacuum pressure $p_1 \sim$ 50–100 mbar), pressure outside the pores decreases and therefore the internal gas expands and partially flows out, while the system tends to equilibrium conditions. When restoring atmospheric pressure (p_2), the residual gas is compressed and the new established pressure gradient provokes the introduction of external liquid to the intercellular spaces where the gas (air) was placed before. In order to obtain the best results during this last step, atmospheric conditions are kept for a certain time, which must be optimized for each product. Pressure changes can also promote deformations of the product because of the viscoelastic properties of its solid matrix.

In order to study the coupling of hydrodynamic mechanism and the deformation–relaxation phenomena, it is necessary to control the mass and volume changes of the product along the process. For this purpose, a previously described equipment (Fito et al., 1996; Salvatori et al., 1997, 1998) has to be used, and the experimental data allow us to determine the characteristic parameters of vacuum impregnation process (impregnated sample volume fraction X, effective porosity (ε_e) of the sample to the hydrodynamic mechanism action, and the relative deformations (γ)). This coupling has been described and modeled using Equations (6.1) and (6.2) (Andrés, 1996; Fito et al., 1996), showing that the volume changes at the end of the vacuum and the atmospheric steps, as well as the effective porosity affect the volume fraction of the product impregnated by the external liquid. In practical terms, sample deformations in vacuum impregnation are seen to be negligible for a great number of fruits (Fito et al., 2001) such that

$$r = \frac{p_2 + p_c}{p_1} \tag{6.1}$$

$$\varepsilon_e(r-1) = (X-\gamma)r + \gamma_1 \tag{6.2}$$

where r is the compression ratio, and γ_1 is the sample volume deformation at the end of the vacuum step. If $\gamma = \gamma_1 = 0$, Equation (6.1) gives the relationship for vacuum impregnation of non-deformed products (Fito, 1994).

Nevertheless, it is important, on a first approach, to measure the mass gained and the new physicochemical characteristics of the impregnated products in order to evaluate the feasibility of using vacuum impregnation technique. In this practical work, mass gained, and water and soluble solid contents will be measured. From the obtained results and considering several data of vacuum impregnation parameters, the viability of using this solid–liquid operation for a specific purpose will be discussed, which has to be pre-defined by the students. Other physical parameters, such as color or mechanical properties could also be analyzed.

6.2. EXPERIMENTAL PROCEDURE

6.2.1. Equipment

- Vacuum impregnation equipment (where vacuum conditions can be applied and controlled) (Figure 6.1)
- Scale
- Caliper
- Refractometers
- Vacuum oven
- Chronometer

Figure 6.1. Vacuum impregnation equipment.

6.2.2. Material

Use different fruits and vegetables (products have to be porous) for each group such as:

- Pieces of several fruits (different geometries), such as apple (cylinders), pineapple (cross-slices), mango (slices), strawberry (halves), kiwi (cubes), peach (cubes).
- Sugar
- Distilled water

6.2.3. Procedure

Samples of these fruits are prepared and immersed on a solution obtained from distilled water and a specific solute (sugar) on a defined concentration (Brix). The system fruit-solution is subjected to vacuum pressure for 5 min and then left under atmospheric pressure for another 10 min. Operation conditions are fixed in order to compare the effect of the process on the physicochemical properties. Composition (water and soluble solids content), and weight are measured before and after the vacuum impregnation process.

Water content is determined in terms of weight losses after vacuum drying on a vacuum oven at 63°C until constant weight.

Soluble solid content is determined on a refractometer after extracting the fruit liquid phase.

6.3. DATA ANALYSIS AND QUESTIONS

1. From weight measurements, determine the mass gained by each fruit. Which one is most impregnated? Look at Table A6.1, where vacuum impregnation parameters are shown. Explain the possible reasons of the different response observed to impregnation in terms of % impregnation (% mass gained)
2. Determine the sensory color, texture, flavor and taste for each product before and after vacuum impregnation. Which one of these attributes were modified significantly by this process? Why? Determine the acceptability of each product.
3. Discuss possible applications of vacuum impregnations.

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7

Expression – Extraction of Pumpkin Oil

Elvyra Jarienė, Honorata Danilčenko,
Paulina Aleknvičienė, and Jurgita Kulaitienė

OBJECTIVE AND LEARNING OUTCOMES

1. Examine the uses of plant oils.
2. Review the mechanisms and Operations of oil pressing.
3. Calculate the optimum of screw rotation speed.
4. Calculate and compare the yield of oil and press cake.

7.1. INTRODUCTION

Fats and oils, in particular vegetable oil sources, have been a topic of keen interest over the past 20 years. The role of dietary fats in human nutrition has created widespread interest in fats and oils among consumers, clinicians, researchers, health educators, food producers, and food processors and distributors. Of particular interest have been the health effects of level of intake and type of fats and oils. Much of the current interest stems from the implication of dietary fat in the etiology of chronic diseases such as coronary heart disease (CHD), cancer, diabetes, obesity, and hypertension.

The pressing of nuts and seeds to extract their precious health sustaining oils, dates back thousands of years. The Egyptian civilization used oils for eating and cooking purposes and, of course, for body care, and so did the Roman and

ELVYRA JARIENE, HONORATA DANILCENKO, PAULINA ALEKNVICIENE, AND JURGITA KULAITIENE • Department of Horticulture, Faculty of Agronomy, University of Agriculture, ns@lzuu.lt

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the Greek civilization. Unrefined oils have always been the cornerstones of the Mediterranean peoples' diet. These oils were rich in nutrients and had particular individual taste, color, viscosity, and of course, unique aromas. All of this was carefully sabotaged by the industrial oil refineries who reduced all oils to the bland, colorless and flat tasting clear oils that flood the marketplace: inferior oils that are detrimental to health. However, in many places around the world, they have maintained traditional methods that produce high quality nutrient-rich unrefined oils that have incredibly delicious flavor. Since 1978, legislation in France states "virgin oils" must be obtained uniquely by mechanical means and filtered naturally without any chemical treatment or operation of refining. Good unrefined oils play a fundamental role in a balanced healthy diet.

7.1.1. Kinds of Plants for Pressing Oil

A huge list of seeds can be used to extract oil (Table 7.1).

Pressed oils can be characterized based on differences in their lipid composition (Table 7.2).

Table 7.1. Seeds from which oil is extracted

1	Apricot stones	7	Groundnut	13	Caraway seed	19	Pistachio nuts
2	Avocado	8	Hazel nut	14	Pumpkin seed	20	Rape seed
3	Cotton seed	9	Raspberry	15	Linseed	21	Sesame
4	Bilberry	10	Elderberry	16	Melon seed	02	Soybean
5	Calendula	11	Black currant	17	Nutmeg	23	Sunflower seed
6	Copra	12	Jojoba	18	Palm kernel	24	Walnut

Table 7.2. Characterization of vegetable oils

Fatty acid	Sun-flower	Soybean	Rapeseed	Olive	Peanut	Palm	Coconut	Pumpkin seed
C 12:0	–	–	–	–	–	–	47.0	–
C 14:0	–	–	–	–	–	–	17.5	–
C 16:0	6.5	11.0	4.0	10.0	10.0	45.0	9.0	12.07
C 18:0	4.0	3.5	1.5	3.0	3.0	4.5	3.0	3.21
C 18:1	21.5	22.0	61.5	77.0	42.0	38.0	7.0	20.45
C 18:2	66.0	54.0	20.0	8.0	38.0	10.0	1.8	63.16
C 18:3	0.5	8.0	10.0	0.5	–	0.5	0.1	0.29
Others	1.5	1.5	3.0	1.5	7.0	2.0	14.6	0.82

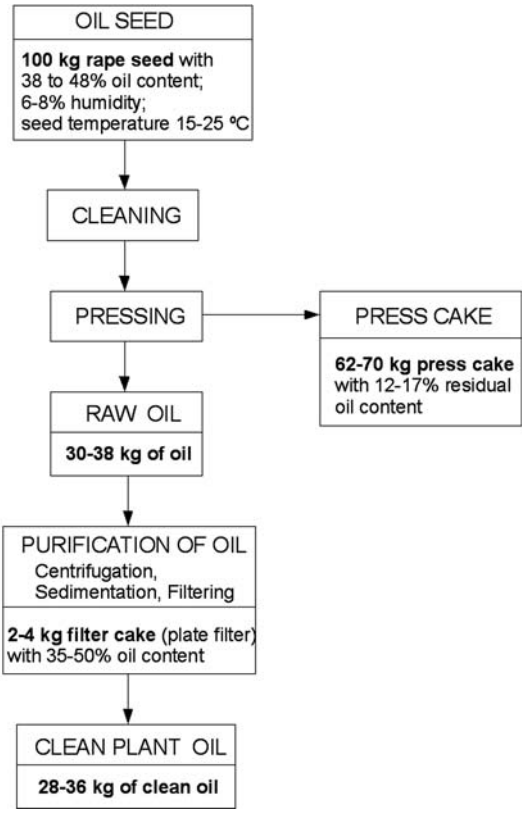

Figure 7.1. The scheme of cold pressing oil.

7.1.2. The Process of Cold Pressing Oil

Cleaning and pressing of oil seeds are the main steps for processing plant oil (Figure 7.1). It is important to clean seeds from stones, possible metal pieces and plant parts. This provides a more constant oil quality and reduces the risk of damaging the press. The contamination should be below 2%. A sieve is used to remove stones and plant parts, and a magnetic separator removes possible metal parts. The seed is prewarmed to about 20°C by a special unit or by a heat exchanger that makes use of the heat from the warm press cake. Preheating seeds to over 20°C has no additional benefit.

7.1.3. Parameters of the Press for Oil Extraction

There are two types of screw presses for production of cold-pressed vegetable oil. Screw rotations are adjustable by a friction ring gear between 20 and 100 rpm.

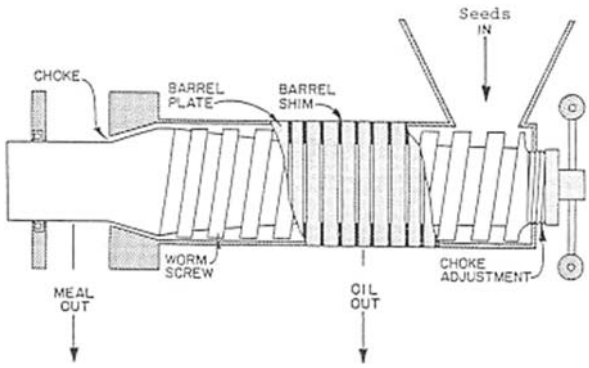

Figure 7.2. Screw press strainer

All screw presses operate electrically, with a diesel motor or can be hand driven. They differ in the screw and in the kind of oil outlet. The outlet can be built like a strainer, which means the press cylinder is created by metal-bars lying near to each other. The gaps between the bars form the oil outlet, which can be varied according to the kind of oil seed. The cake is pressed out of an adjustable choke formed into plates (a kind of chips) (Figure 7.2).

The diameter of the screw changes several times over its length to gradually increase the compression of the seed. This changing diameter can be formed several times on the screw. During the flow of the seed through the press, the oil is drained via the strainer, which surrounds the pressing space. The choke size can be adjusted to press the seeds harder.

Some machines can remove the heat generated around the strainers by a water cooler. For pressing several oil seeds, it is necessary to change the gap size of the strainer-bars, where the oil comes out, to get an optimal yield of vegetable oil. In some types of strainer presses, it is possible to change segments at the worm screw in order to change the compression of the seed. Other manufacturers offer extra screws. In addition, the choke size and the rotation speed should be adjusted by pressing different kinds of seeds. Strainer press types exist in a wide capacity range from approximately 15 to 2000 kg of seeds per hours.

The oil outlet from the other type of oil presses consists of drilled holes in a special part of the cylinder tube. The press cake is forced out in the form of pellets through a changeable nozzle at the end of the cylinder (Figure 7.3).

The oil outlet is in the form of ***holes*** in the press tube. The seeds suffer a gradual compression in direction of the press head. The oil is pressed out of the seeds near the outlet holes and drained to them. Special perforations in the tube avoid turning of the press cake/seed-mix with the screw. Otherwise, there will be no movement forewords. The press cake is pressed through changeable nozzles and formed into pellets. The nozzle, in most types, is heated to avoid blocking of

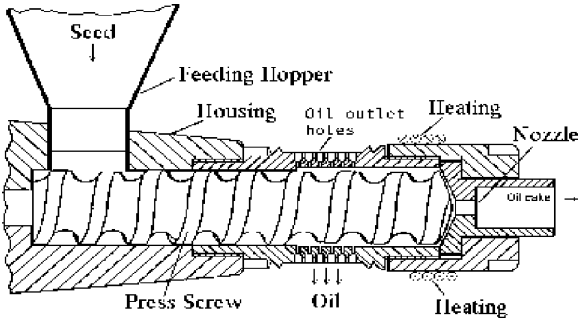

Figure 7.3. Screw press hole cylinder type.

the press cake. Most cylinder types exist for small capacities (up to approximately 100 kg of seeds per hour.

For some types of screw presses it is necessary to heat this part of the press, in order to avoid blockage of the press cake outlet. This heating should be in the range between 60 and 80°C. A higher temperature on the press cake outlet will lead to a higher phosphorous content in the oil. This temperature has an effect on the oil temperature, which should not rise over 40°C. With a lower temperature on the cake outlet the solid content in the oil rises.

The nozzle diameter and gap-size of the choke depends on the kind of seed. The optimum nozzle diameter varies between 6 and 8 mm. The phosphorous content in oil can be minimized with a 6 mm diameter a nozzle temperature of 60°C and a seed humidity. On the other hand there is a higher risk for blocking the nozzle under those conditions. The seeds are pressed harder by a smaller cake outlet and the yield rises beside a decrease in capacity.

An important parameter for the oil pressing process design for oil extraction is the residual oil in the press cake. By pressing very hard, a value of residual oil in the press cake, as low as 10%, can be obtained which and indicates a high percentage yield of oil (up to 38%). A harder pressing is obtained for a nozzle press by changing the nozzle diameter. For an increasing pressure, a strainer press is needed to adjust the cake outlet and by changing single segments of the screw.

If the throughput is reduced (e.g., screw rotation speed is reduced), yield of oil is increased but solid content in the oil is also increased. Alternatively with an increased in throughput, the yield is reduced and the solid content in the oil is reduced. It is possible to find an optimal compromise according to your individual aims with a revolution-regulated press-screw. This also allows pressing of a wide range of different oil seeds. Favorable rotation speed for the screw is between 20 and 50 rpm. In this range of rotation, a minimum energy demand is required. The higher the throughput of seed, the greater the capacity of the oil cleaning installation must be. With this increasing oil production, the total quantity of solids in the oil also rises.

7.1.4. Using the Press Cake

The press cake which is obtained by cold pressing contains more residual oil and has therefore a higher value for animal food purposes than the one which is obtained by warm pressing or normal industrial extraction with solvents. For example, by pressing 3 kg of rapeseed, you produce approximately 1 kg of rapeseed oil and 2 kg of press cake. The cake is the most valuable part of the process and the main product of the cold pressing.

The cake can be used as feed for cows, pigs, chicken, sheep and horses, and is an important source of nutrients and energy for animals. Rape and sunflower press cake is considered to be an optimal cattle fodder. The cold-pressed oil, especially the oil-rich sediment, is used as fodder oil to improve low-energy pig fodder. Also the filter cake from plate filters with 35–50% oil-content can be a valuable fodder.

The pellets should be stored cold, dark and dry. After pressing, the cake has a temperature between 40 and 60°C. Cooling down the press cake right after pressing is required for storage in silos. This cooling can be reached by transporting the cake with a conveyor belt to the silo.

7.1.5. Pumpkin Seed Oil

Some pumpkin cultivars are more suitable for oil pressing than others (Figure 7.4).

Pumpkin seed oil is one of the most nutritional oils available, and is an excellent source of essential fatty acids, antioxidants, vitamins, and sterols. It contains omega-3 and omega-6 fatty acids, which are known to promote energy levels, brain function, and overall health and vitality. It also contains high levels of vitamin E, as well as vitamins A and C, zinc, and other trace minerals and vitamins. Historically, pumpkin seeds (either the whole seeds or the pressed oil) have been used all around the world for healing purposes. They were used for a

Figure 7.4. Pumpkins cultivars *Miranda* (a), *Golosemianaja* (b), *Herakles* (c) for pressing oil.

variety of applications, including: to heal wounds and burns, as a diuretic, to dispel intestinal worms and parasites, hormone production, overactive bladder, and for prostrate problems. Today, this oil is used successfully in preventing and alleviating prostrate and bladder problems. Phytosterols present in the pumpkin seed oil, are also presently being studied for their role in lowering cholesterol levels. Pumpkin seed oil has a pleasant and mildly rich flavor. It is best to use the oil in its raw form, in a salad dressing, smoother, or drizzled over a freshly cooked meal just before serving, so that the important health benefits are not damaged by heat.

7.2. EXPERIMENTAL PROCEDURE

- Three different pumpkins cultivars: *Miranda*, *Golosemianaja*, *Herakles* pumpkins seeds – 300 g of each.
- Graduated cylinder (150 ml).
- Timer.
- Analytical balance (+/– 0.01 g).
- Thermometer.
- Screw press CA 59 G (Figure 7.5).

7.2.1. Procedure

Oil is made from seeds of various cultivars and using different screw rotation speed, yield of oil is different.

Figure 7.5. Screw press CA 59 G type (IBG Monforts Oekotec GmbH).

1. Clean seeds: Remove debris and foreign and damaged seeds.
2. Turn on the equipment. First experiment: Set screw rotation speed to 10 rpm.
3. Weigh 100 g of *Miranda* cv. seeds.
4. Continue the experiment until all seeds are pressed. Record the time.
5. Control the temperature of the press cake during oil pressing so that it is no greater than 60°C.
6. Collect the oil in a graduated cylinder.
7. Weigh 100 g of *Golosemianaja* cv. seeds.
8. Continue the experiment until all seeds are pressed. Record the time.
9. Control the temperature of the press cake during oil pressing so that it is no greater than 60°C.
10. Collect the oil in a graduated cylinder.
11. Weigh up 100 g *Herakles* cv. seeds.
12. Continue the experiment until all seeds are pressed. Record the time.
13. Control the temperature of the press cake during oil pressing so that it is no greater 60°C.
14. Collect the oil in a graduated cylinder.
15. Second experiment: screw rotation speed is set to 20 rpm.
16. Repeat all the operations from steps 4 to 15.
17. Third experiment: screw rotation speed is set to 30 rpm.
18. Repeat all the operations as in from steps 4 to 15.

7.3. DATA ANALYSIS AND QUESTIONS

For the each example:

1. Measure the yield of oil from three different cultivars in each experiment in ml using a graduate cylinder.
2. Weigh up the amount (g) of press cake from different cultivars in each experiment.
3. Calculate the yield of oil and press cake in %.

7.4. QUESTIONS

1. What types of screw presses are usually used for the production of cold-pressed vegetable oil ?
2. What is the relationship between screw rotation speed and yield of oil ?
3. Before pressing the nozzle is heated to avoid blocking of the press cake. What temperature is recommended and why ?
4. Compare the oil amount of press cake of different cultivars.
5. Which screw rotation speed is more efficient ?

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Part II
Preservation Operations

8

Dehydration I – Tray Drying of Apples

Margarida Vieira and Jorge Pereira

OBJECTIVES AND LEARNING OUTCOMES

1. Describe the drying process for a tray drier and indicate relevant variables that are used to control the process.
2. Calculate the drying times for different periods of a drying process.

8.1. INTRODUCTION

Dehydration is a food unit operation that is used to extend the shelf-life of a foodstuff by reducing its water activity (a_w). Drying with hot air using a tray or cabinet drier is one of the methods of food dehydration. Heat transferred to the surface of a foodstuff exposed to hot air, provides the latent heat of vaporization to evaporate moisture from the food surface. Water vapor diffuses through the film of air that is formed at the food surface and is subsequently removed by the circulating air. A region of low vapor pressure thus forms at the food surface, establishing a vapor pressure gradient between the inner humid layer and the outer layer of dry air. This gradient acts as the driving force of the drying process (Geankoplis, 1993, Heldman and Singh, 1981).

MARGARIDA VIEIRA AND JORGE PEREIRA • Universidade do Algarve, Escola Superior de Tecnologia, mvieira@ualg.pt • Universidade do Algarve, Escola Superior de Tecnologia

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8.1.1. Drying Curves

A drying process can be represented graphically by recording changes in moisture content of a foodstuff. Drying curves are typically represented as drying rate curves (Figure 8.1), showing different stages or periods of drying.

The drying process begins with an initial short period that allows for the food surface that is heated to reach similar conditions as the hot air (**A** to **B**). This initial period of drying is normally considered insignificant in comparison with the two main periods of the overall drying process (Brennan et al. 1990). The *constant rate period* (**B** to **C**) follows the initial period, where the drying rate is constant. At this stage the food surface is covered completely by a continuous film of free water (water that is not bound to the food surface). As water from the film evaporates, more water from the surface of the food replaces it, thereby keeping the food surface wet.

At a certain point in the drying process, the continuous film of water at the surface cannot be maintained, which results in a significant reduction in water and the surface begins to dry. It is at this point where the critical moisture content is reached and, as the rate of drying decreases, the *falling rate period* begins. A single falling rate period occurs with non-hydroscopic foods whereas with hydroscopic foods, two (**CD** and **DE**) or more periods may occur (Geankoplis, 1993, Fellows 2000).

In this period, the evaporation plane moves slowly from the surface to the interior of the food, as the latent heat of vaporization is transferred through the solid to the evaporation area, vaporized water moves through the solid to the circulating air.

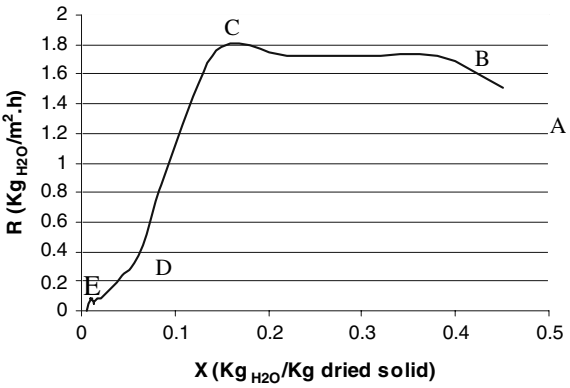

Figure 8.1. Drying rate curve for constant drying conditions.

8.1.2. Drying Time Calculation

In order to collect appropriate data from a drying experiment, of the loss of weight from a foodstuff, which can be used for constructing drying curves, the relative surface area of the material to be dried, relative air humidity and hot air flow rate should be kept constant during the drying process. The drying curve can be used directly to calculate the drying time or the data can be converted to plot a drying rate curve.

8.1.2.1. Determination of the Length of the Constant Rate Period

In this period, the rate of drying, R, can be expressed by:

$$R = -\frac{L_S}{A}\frac{dX}{dt} \tag{8.1}$$

where X is the free moisture content (kg water/kg of dry solids), t is the drying time (s) drying time, A is the exposed surface area (m^2), and L_S is the mass of dry solids (kg) (Geankoplis, 1993) .

By integration of Eq. 8.1 between $X = X_1$ for $t_1 = 0$ and $X = X_2$ for $t_2 = t$ the drying timedrying time is obtained from:

$$t = \frac{L_S}{AR_c}(X_1 - X_2) \tag{8.2}$$

where R_c is the constant drying rate (kg water $h^{-1}m^{-2}$)

8.1.2.2. Determination of the Length of the Falling Rate Period

For the falling rate period, the drying timedrying time can be obtained by integration of Eq. 8.1, taking the initial humidity, X_1, below the critical humidity, X_c (Eq. 8.3). This can also be obtained graphically, by representing $1/R$ as a function of X, and taking the area under the curve for that time period Geankoplis, 1993.

$$t = \frac{L_S}{A}\int_{X_2}^{X_1}\frac{dX}{R} \tag{8.3}$$

8.2. EXPERIMENTAL PROCEDURE

8.2.1. Tray Drier (Armfield)

The UOP8 is a small scale drier unit designed by Armfield. It has a tunnel in which an axial flow fan is mounted at one end (Figure 8.2). Before the fan there is a set of electrically heated elements which heat the passing through air stream before flowing to the drying chamber. The chamber contains a rack of trays

Figure 8.2. Tray drier.

(31.5 × 17.7 cm each) suspended from a scale mounted on top of the drier. The total capacity of the trays is approximately 3 kg of solids. A transparent access door, permits viewing the product while drying. A control panel mounted near the fan allows setting the air speed and air temperature by varying the power of the heater.

8.2.2. Additional Apparatus and Materials

The following additional apparatus and materials are required for the experiment:

- Anemometer used to measure the air flow in the drier.
- Manual Psychrometer (with an aspirated wet and dry bulb psychrometer).
- Infrared scale for humidity measurement, Mettler LP16-M.
- Thermocouples SSA-12100-G700-TS length 10 mm and 1.2 mm of diameter and data acquisition system for measuring and recording temperatures (Ellab CTF 9004).
- 1 kg of apples.

8.2.3. Procedure

1. Start the tray drier, set the knob to a flow rate between 5 and 6 to reach a flow rate of 1.48 m s^{-1}. The temperature knob was set at 12 (max.)

2. Place the trays on the shelves, after measuring the area of the shelves, and place a thermocouple on the middle shelf.
3. Cut the apples into round slices with a thickness of approximately 2 mm. Place the round apples slices on the trays and record the initial weight of the material to be dried. Place a thermocouple through an apple slice.
4. Measure the initial moisture content humidity of the apple slices using the infrared scale.
5. Measure the temperature and humidity with the psychrometer before and after the drier chamber before starting the drying process to check if the values are stable (values presented in Annex A8).
6. Turn the air flow rate knob to an intermediate position and record the air flow rate with the anemometer. Set the temperature controller to maximum. This is the initial moment of drying.
7. Register the weight of the apples and the temperature at the apple surface every 30 s for the first 10 min of drying. After this initial period, record these measurements every 2 min. Record the dry bulb temperature and the wet bulb temperature with the help of the psychrometer above the trays at 3-min intervals trays. Remember to check that the conditions of drying are kept constant by registering the temperature under the trays at 15-min intervals (values presented in Annex A8).
8. Continue registering the temperatures (values presented in Annex A8) until a constant weight is reached for the apple slices.
9. After completing the drying process, measure the moisture content of the sample of dried apple slices (values presented in Annex A8).

8.3. DATA ANALYSIS AND QUESTIONS

Using the data presented in Annex 8, represent graphically the relationship between the moisture content of the food sample and the drying time by plotting (i) the drying rate/area against time; and (ii) the drying rate against the moisture content of the apple slice. (Note: Take the surface area of drying as 1.5 times the total area of the trays.)

1. For each graph, identify the different periods of drying.drying
2. Calculate (i) the critical moisture content; (ii) the drying time during the constant rate period ; and (iii) the drying time during the falling rate period?

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9

Dehydration II – Mass Balances on a Combined Air-Microwave Drying Process

M. Eugenia Martin-Esparza, Chelo Gonzalez-Martinez, and Pedro J. Fito

OBJECTIVES AND LEARNING OUTCOMES

1. Calculate mass and energy balances during hot air drying of a solid food product.
2. Examine how to manage the variables involved in the operation, that is, room temperature and relative humidity, drying temperature and air rate, product mass losses, and time, in order to study the drying kinetics (drying curve and drying rate curves).
3. Determine the energy needs in a drying operation.
4. Evaluate an alternative procedure to obtain certain kind of products, such as candy products, with less energy consumption.

M. EUGENIA MARTIN-ESPARZA • Food Technology Department, Universidad Politécnica de Valencia. CHELO GONZALEZ-MARTINEZ • Food Technology Department, Universidad Politécnica de Valencia, cgonza@tal.upv.es. PEDRO J. FITO • Food Technology Department, Universidad Politécnica de Valencia

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9.1. INTRODUCTION

Water in food can be removed by mechanical operations (e.g., filtration or centrifugation) or by physico-chemical methods (e.g., surface evaporation, osmotic dehydration, freeze drying, adsorption or freezing). Recently, drying has been used as a tool in developing new products that could be used as food ingredients or be consumed directly (Barbosa-Canovas and Vega-Mercado 1995).

When drying with hot air, water is removed from the product by evaporation, resulting in an increase in air humidity. Several transport phenomena take place during the process (Fito et al., 2001), mainly:

- Heat transfer from the air to the product due to a temperature gradient (i.e., convection).
- Heat transfer inside the product, from the surface which is at a higher temperature (i.e., conduction).
- Mass transfer of water from the wet product to the air, which is associated with heat transfer, relating to the latent heat required to change the physical state of water (i.e., from liquid into water vapor).
- Mass transfer from within the product to the surface. Several transport mechanism are associated with this phenomena, such as capillary transport (i.e., when there is a high moisture content), and diffusion (i.e., predominantly occurring at a low moisture content).

The first step when studying the drying process of a particular food product (from the point of view of mass losses), is to plot the drying curve and the drying rate curve from data obtained experimentally. These graphs can aid in identifying the different mass transport phenomena that can be relevant to the drying process and in selecting the appropriate mathematical equations that model drying kinetics. Additionally, it is important in verifying the mass balances of the process.

9.1.1. Drying Curve

Product water content (x_{wt}) can be obtained from mass losses along the drying process, if the initial level (x_{wi}) is known (determined experimentally), by using the corresponding mass balances.

x_{wt} can be obtained from a dry mass balance between initial time and each process time t:

$$\mathrm{weight}_i \cdot (1 - x_{wi}) = \mathrm{weight}_t \cdot (1 - x_{wt}) \tag{9.1}$$

$\mathrm{weight}_{i,t}$ is the product mass in the initial and t process time, respectively. The percentage of weight loss can be obtained from Equation (9.2).

$$\%\,\text{weight loss} = 100 - \left(\frac{\text{mass}_t}{\text{mass}_i}\right) \cdot 100 \tag{9.2}$$

In order to check if experimental measurements have been correctly determined (no mass losses along the drying process), another mass (water) balance can be used (Equation 9.3).

$$\text{mass}_i - \text{mass}_f = \text{mass}_i \; x_{\text{w}i-\text{mass}_\text{f}} \; x_{\text{wf}} \tag{9.3}$$

Where *i* denotes initial and f denotes final.
Obtained water contents form Equation (9.1) will be expressed also on dry basis.

9.1.1.1. Drying Rate Curve

Product water loss rate is expressed as $-\text{d}X_\text{w}/\text{d}t = f(X_\text{w})$. This function can be plotted as an approximation by determining the ratio between water content increases (ΔX_w) and time increase (Δt), versus average product water content (ΔX_w) in each time period. Obtained values for water content (Equation 9.1) will be considered.

9.1.1.2. Air Conditions: Relative Humidity, Temperature, Enthalpy and Absolute Humidity. Mollier Diagram

During the drying process the product water content decreases and the evaporated water is transferred to the gas phase in contact with the product. Therefore, the absolute humidity increases. Also the enthalpy is changed when increasing the air temperature (before contacting the food product) and when "absorbing" the evaporated water from the product (Martinez-Navarrete et al., 1998). The evolution of the air during the drying process can be determined by defining three main points where air characteristics are changed: (1) room air; (2) air inside the drier before contacting the product; and (3) air after contacting the product.

9.2. EXPERIMENTAL PROCEDURE

9.2.1. Apparatus

- Drier (Fig 9.1).
- Vacuum oven.
- Scale.
- Refractometer.
- Balance.

Figure 9.1. Microwave drier.

9.2.2. Material

- Apples

9.2.3. Procedure

The temperature (T), the relative (RH) and absolute (X) humidity, and the enthalpy (h) of the gas phase in the three points mentioned in 9.1.1.3 should be obtained from the actual conditions in the room where the experiment was run (average values) and from the Mollier psychometric diagram. The obtained results should be filled in Table A9.1.

9.2.3.1. Air Volumetric and Mass Flow. Consumed Electric Power on the Process

The electric power (P) necessary to increase the air temperature until the operating value, and to keep it along all the drying process, can be obtained (Equations (9.4)–(9.7)) from the mass flow (Q_{mass}) and the increase in the air enthalpy (related to the increase in air temperature from the room to the working value). This electric power is supplied by the electric resistances of the drying equipment. Enthalpy (h) can be determined from the psychometric Mollier diagram.

$$P = Q_{\text{mass}}\ \Delta h \tag{9.4}$$

$$Q_{\text{mass}} = Q_{\text{volumetric}}.\rho_{\text{air}} \tag{9.5}$$

$$Q_{\text{volumetric}} = \text{air velocity} \times \text{Drier section} \tag{9.6}$$

Air density can be obtained from Equation (9.7):

$$\rho_{\text{air}} = \frac{PM_{\text{air}} \cdot P_{\text{atm}}}{R \cdot T} - \frac{RH \cdot P_{\text{s}}\rho}{R \cdot T} \cdot (PM_{\text{air}} - PM_{\text{water}}) \tag{9.7}$$

where

$PM_{air} = 29\ \text{g} \cdot \text{mol}^{-1}$,

$PM_{\text{water}} = 18\ \text{g} \cdot \text{mol}^{-1}$, $R = 0.082\ (\text{atmL} \cdot \text{mol}^{-1} \cdot \text{K})$, T = air temperature expressed on K ($T_S = T_2$), RH_2 = relative humidity at drying temperature

P_{atm} = 1 atm, P_s = see Table A9.1 (values must be recalculated based on atm).

9.2.3.2. Mass Balance on the Process

Mass balance on the process will be checked by comparing the induced increase on air humidity ($X_3 - X_1$) and induced decrease on product water content (ΔX_{w}).

The raw materials must be characterized by means of evaluating the following parameters:

1. Weight.
2. Water content.
3. Soluble solids content.
4. Volume.
5. Thickness.

Along the drying process, every 30 s, the following parameters must be monitored (data presented on Table A9.3):

1. Room relative humidity.
2. Room temperature.
3. Drying temperature.
4. Air velocity.
5. Mass loss (balance).
6. Incident and Reflected Microwave Power.

The dried material must be characterized by means of evaluating the following parameters:

1. Weight.
2. Water content.
3. Soluble solids content.
4. Volume.

9.3. DATA ANALYSIS AND QUESTIONS

From results presented in Annex 9, represent the Product weight versus process time and:

1. Plot the drying curve: $X_w = f(t)$.
2. Plot the drying rate curve: $-\Delta X_w/\Delta t = f(X_w)$.
3. Describe the different drying steps observed on the drying rate curve.
4. From mass balance on the process, what conclusions can be deduced?
5. Repeat the experiment combining hot air with the microwave. Determine the increase on drying rate?

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10

Spray Drier – Atomization of Milk

Cristina Luisa Silva, Magda Navarro de Noronha and Alexandra Morim

OBJECTIVES AND LEARNING OUTCOMES

1. Review the principles of operating a spray drier.
2. Identify the main variables affecting the process.
3. Compare the obtained milk powder by atomization and the original commercial one, concerning the quality attributes of colour, smell and granulation.

10.1. INTRODUCTION

Spray drying, by atomization, transforms a liquid feed into a spray of very small droplets in a continuous one-step operation. These droplets have a very large surface area and evaporation is completed rapidly, producing a dry powder. The necessary small droplet sizes are produced by rotating the vaned wheel at high speeds. The resulting evaporation rates are high enough to enable completion of moisture removal from the droplets, even though the small chamber volume gives short droplet residence times in the dryer. During the evaporation stage, there is an accompanying cooling effect on the droplets, and with the short duration of the product residence time in the chamber, heat damage of the product is prevented (Earle, 1983).

CRISTINA LUISA SILVA • Escola Superior de Biotecnologia, Universidade Católica Portuguesa, clsilva@esb.ucp.pt MAGDA NAVARRO DE NORONHA AND ALEXANDRA MORIM • Escola Superior de Biotecnologia, Universidade Católica Portuguesa

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The separation of the dried product from the is achieved in a cyclone separator, connected with the drying chamber. Drying by atomization is a method for liquid foods that minimizes the changes of aroma. This method is used in the production of the milk powder, instant coffee and other thermal sensitive products.

10.2. EXPERIMENTAL PROCEDURE

10.2.1. Apparatus

- Spray Drier NIRO Atomizer, Figure 10.1 (details in Annex 1) (Niro Atomizer, 1992).

10.2.2. Material

- Distilled water
- One litre of milk, prepared using commercial light milk powder (solids 20% in w/w).
- Water.

Figure 10.1. Spray Drier NIRO ATOMIZER.

10.2.3. Procedure

1. Turn on the air aspiration blower, turning the button (14) from position 0 to M. To start air heating, turn on the resistances, turning on the same button to position II.
2. Turn the temporization button to position 6, in order to adjust the temperature of the air entrance (between 200 and 250°C).
3. Start the atomization procedure (turn the blue valve until 4 bar pressure) with the heater and blower turned on.

   Ensure that the air pressure must be kept constant, in order to obtain an efficient liquid atomization (observe the manometer). If pressure goes down, the atomization will loose velocity.
4. Feed the atomization with distilled water (to adjust the drying conditions). Keep a gradual strain of water until the desired temperature (100°C).
5. Prepare 1 L of light milk with approximately 20% w/w of solids (use commercial milk powder and hot water) and filter the milk using a net filter.
6. Change the feed water in the spray drier with the product to be dried.

   Be sure to keep the air exit temperature constant during the drying operation, to maintain the water content constant of the dried product (verify the temperature).
7. Complete the drying of the milk.
8. Change the atomizer feed with distilled water, and change the product collecting bottle.

   The quantity of feed water must be regulated not to lower the exit temperature. Keep the unit operating with distilled water for another 5–10 min.
9. Turn off the distilled water feed to the atomizer.
10. Turn off the heat resistance, turning the button to position M, to keep only the air aspiration.
11. After 3 min, turn off the atomizer (blue valve) and keep the cold air circulating in the drier until the exit temperature is approximately 50°C.
12. Open the chamber's upper cover using button 12. Verify that the feed pump is not over the cover.
13. Turn off the air aspiration blower (turn the button from position M to O).

10.3. DATA ANALYSIS AND QUESTIONS

1. Describe the main observations during the process; principles of operation, main variables affecting the process.
2. Determine the total drying time.

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11

Operating a Fluidized Bed Drier

Semih Otles

OBJECTIVES AND LEARNING OUTCOMES

1. Operate a fluidized bed drier.
2. Describe safe laboratory operations.
3. Determine the heat transfer coefficients in a fluidized bed dryer.
4. Describe the analysis of drying curves for various food matrices by using fluidized bad dryer.

11.1. INTRODUCTION

Fluidized bed drying of food and food products is one of the most efficient drying methods, which is finding ever-growing applications in diverse industries, especially in the pharmaceutical and food industries. One promising drying process is the drying of food product in a fluidized bed, which gives better heat and mass transfer, shorter drying time, better quality products and shorter reconstitution time. Fluidized bed dryers are used to dry loose granular solids and are characterized by a large exchange area between the particles and the gas, and thus, by high heat and mass transfer coefficients. Hence, they provide uniform wet masses for drying, high drying rate and short drying time. A fluidized bed dryer significantly reduces drying time compared to a tray dryer or vacuum dryer. It also exposes the entire product surface area to the high volume air stream and heat is transferred to the product surface by convection. However, if the inlet air velocity is not properly selected, consistent and uniform drying will not be obtained Heldman and Singh, 1981 and Geankoplis, 1983.

SEMIH OTLES • Department of Food Engineering, Ege University, Semih.otles@ege.edu.tr

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11.1.1. Equipment

The laboratory fluidized bed dryer (Figure 11.1) is a simple, compact design, conveniently portable and easy to operate, with the only requirement being a main power supply. The cabinet contains the air distribution system and electrical controls. Air is drawn in through a mesh filter in the base of the cabinet and blown by the centrifugal fan over a 2 KW electrical heater and through a stainless steel filter gauze at the base of the dryer body.

The tub unit consists of a container with a fine mesh nylon gauze air distributor and stainless steel support gauze. This channel over the top of the tube, retains any particles expelled from the fluidized bed. Filter bags are available in nylon, polyethylene terephthalate (PET) and polypropylene to suit a range of process conditions.

The dryer cabinet incorporates an electrical heater, temperature controller, timer and air blower fan.

The air blower is controlled by a thyristor circuit to give a smooth variation over a wide range of motor speeds. This enables efficient fluidization of the drying temperature.

Some, particulate materials, can be easily uniformely distributed. However, a more violent slugging or spouting type of fluidization may be obtained, which also

Figure 11.1. Fluidized bed drier.

has good heat transfer characteristics and has applications in drying of granular materials and foodstuffs, coating of tablets, and granulation of fertilizers.

The heater is a 2 KW finned element which gives air inlet temperatures to the fluidized bed of up to 200°C. A two-term temperature controller gives an accuracy of +1°C over the complete operating range. Adjustment is made by a simple thumb wheel movement for both analogue and the optional digital temperature display.

Air velocity is variable using the blower speed control, which is graduated from 1 to 10 for ease of operation. Table A11.1 shows the relationship between blower speed and air velocity.

The unit can be used in either manual or automatic mode. The sequence timer allows the user to pre-set any cycle duration time between 6 s and 6 h. An audible alarm will sound at the end of each drying cycle and the unit will automatically switch off to allow weighing of the sample. After each timed cycle the unit must be reset using the stop–reset control button.

11.1.2. Range of Materials

The wide range of materials which can be dried includes fine powders, coarse particles, crystals, granules, slurries or pastes (after decanting, or pre-drying or by spraying into bed of initially dried materials). Materials with moisture content up to 80% such as some polymers, dyestuffs and molecular sieve catalysts can also be accommodated. In addition, heat-sensitive materials including foodstuffs such as peas, wheat and lentils, may be dried at relatively low temperatures.

11.1.2.1. Optimum Bed Depth

The optimum bed depth is that which can be fluidized at the required temperature by relatively high air velocity, bearing in mind that as drying proceeds the bed becomes easier to fluidize and the air velocity can be progressively reduced. This optimum bed depth will vary appreciably with the material – an initial bed depth of about 75 mm is typical and a trial and error procedure is generally used to identify the optimum. Particle sizes in the range 0.1–5 mm are ideal with the largest size ratio.

11.2. EXPERIMENTAL PROCEDURE

1. Determine the moisture content of the sample to be dried.
2. Remove any excess water from the sample by decanting and/or using a filter pump.
3. Place the sample in the dryer at a pre-determined bed depth compatible with the operating range of the dryer as previously indicated. In some

cases the moisture content may be too high for immediate fluidization to be effected, but after some initial drying fluidization becomes possible.

4. Weigh the empty container and then again together with the material to be dried.
5. Fit a clean bag over the container, locating the sealing ring into its groove on the tub.
6. Switch on the mains supply.
7. Select the drying temperature.
8. Select manual or automatic mode of operation. Press the cycle start button.
9. Select blower and heater settings. Adjust blower speed to achieve good fluidization as determined by observation. The blower speed will correspond to the required fluidization velocity which will normally be above the minimum fluidization velocity (*umf*) and in the range of 1–2 *umf*.
10. In the manual mode, when drying reaches the desired composition as judged by the temperature of the outlet air or visual observation, press the stop button to stop cycle. Remove the filled tub unit and weigh it, continue repeating the drying cycle until a constant weight is obtained. For each weight measurement the dryer is stopped and restarted.

    In the automatic mode, once the pre-set time has elapsed the cycle will automatically stop and the alarm will sound. This can be reset by pressing the red stop button.
11. For simple drying, place the sample of material in the tub to an appropriate bed depth through trial and error.
12. Weigh the empty tub unit and again then with the material. Fit a filter bag over the tub unit locating the sealing ring into the groove.
13. Switch on the mains supply and select the required drying temperature as in (1).
14. Using a suitable hygrometer, measure and record note the wet and dry bulb temperatures (and thus humidity) of the inlet air to the fan and outlet air from the fluidized bed.
15. Weigh the filled tub unit at 2 min intervals for about 15 minutes (or as long as it takes to attain constant weight) recording the wet and dry bulb temperature before removing the tub for weighing. Continue taking measurements at 5 min intervals until constant weight is achieved indicating that the equilibrium moisture content has been reached. Record the drying time and moisture content.

11.3. QUESTIONS AND CALCULATIONS

1. Calculate the heat transfer coefficients for the constant rate period and falling rate period.
2. Determine the mass measured as a function of drying time.

3. Plot drying curves of moisture content versus time and drying rate versus free moisture content.
4. Read the drying time from the graph.
5. Calculate the theoretical drying time and compare it with the experimental time, discuss the reasons for difference.
6. Discuss the effects of the characteristics of the material on drying conditions.
7. How can you minimize experimental errors?
8. Suggest different substances that can be dried with this type of dryer.
9. Describe the advantages and disadvantages of fluidized bed dryers.

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12

Freeze Drying Foods

Lea Hyvönen and Kirsi Jouppila

OBJECTIVES AND LEARNING OUTCOMES

1. Review the theory of freeze drying.
2. Examine the critical requirements for successful freeze drying.
3. Calculate the diffusion coefficient and mass transfer coefficient.
4. Determine and compare the physical and mechanical properties of the freeze-dried products with those of the heated air-dried products.

12.1. INTRODUCTION

Freeze drying is a method of water removal from a frozen product mainly by sublimation. The frozen water, ice, changes directly to the gas phase without passing through the liquid phase. Sublimation is only possible under the conditions below the triple point of water (temperature 0.01°C; pressure 610.5 Pa) at which all three phases of water are in equilibrium (Figure 12.1). Any material must be adequately pre-frozen before starting the freeze drying process. A maximum amount of ice is obtained when material is frozen at temperatures between the glass transition temperature of maximally freeze concentrated material (T_g') and the onset temperature of ice melting in maximally freeze concentrated material (T_m'). Table 12.1 shows T_g' and T_m' values for different sugars and for selected food materials. Sugar containing materials are very difficult to freeze-dry, because their onset temperatures of ice melting in maximally freeze concentrated systems are very low. It is important to cool down the frozen product below the glass transition temperature, before starting the freeze drying process.

LEA HYVÖNEN AND KIRSI JOUPPILA • Department of Food Technology , University of Helsinki, lea.hyvonen@helsinki.fi

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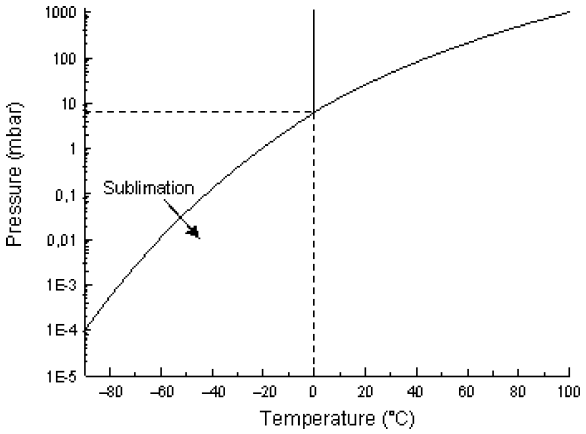

Figure 12.1. Phase diagram of water showing sublimation of ice (triple point of water shown by dotted lines).

Table 12.1. Glass transition temperatures (T_g', onset) and onset temperature of ice melting (T_m') of maximally freeze-concentrated matrix for various sugars and selected food materials determined using DSC

Material (reference)	T_g' (°C)	T_m' (°C)
Fructose (Roos, 1993)	–57	–46
Glucose (Roos, 1993)	–57	–46
Lactose (Roos, 1993)	–41	–30
Maltose (Roos, 1993)	–42	–32
Sucrose (Roos & Karel, 1991a)	–46	–34
Freeze-dried skim milk (Jouppila & Roos, 1994)	–50	–32
Freeze-dried skim milk with hydrolyzed lactose (Jouppila & Roos, 1994)	–65	–40
Maltodextrin M365, DE35 (Roos & Karel, 1991b)	–43	–28
Maltodextrin M200, DE20 (Roos & Karel, 1991b)	–31	–19
Maltodextrin M100, DE10 (Roos & Karel, 1991b)	–23	–13
Maltodextrin M040, DE5 (Roos & Karel, 1991b)	–15	–11
Gelatinized corn starch (Jouppila & Roos, 1997)		–11

After pre-freezing, the ice can be removed from the frozen product via sublimation by controlling the temperature and pressure of the freeze drying system (Figure 12.2 and 12.3). The water vapour pressure of a material must be below 6.105 mbar (triple point). The rate of sublimation of ice from frozen product depends on the difference in vapour pressure of the product compared to the vapour pressure on the ice collecting refrigeration coils (condenser). Water molecules migrate from the higher pressure product to a lower pressure

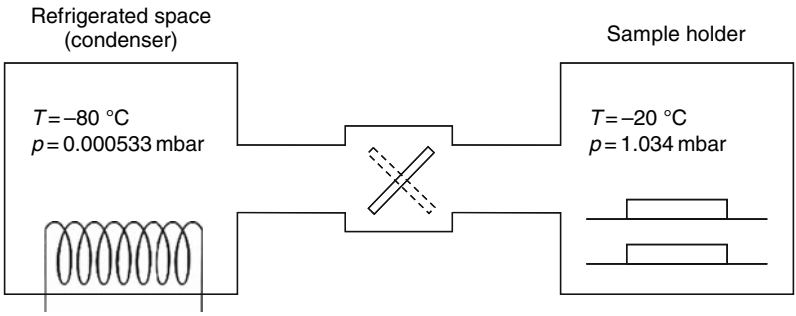

Figure 12.2. Scheme of a freeze dryer.

Figure 12.3. Freeze drying equipment.

condenser. Because vapour pressure is related to temperature, the product temperature must be significantly higher than the condenser temperature (at least 20°C).

It is important that the temperature at which a product is freeze dried is balanced between the temperature that maintains the frozen integrity of the product and the temperature that maximizes the vapour pressure of the product. The product temperature must remain below a critical collapse temperature. Table 12.2 shows examples of collapse temperatures for some food materials in freeze drying.

Freeze drying conditions must be created to help the free flow of water molecules from the product. A vacuum pump is used to lower the pressure of the environment around the product and a condenser is used to condense and collect

Table 12.2. Collapse temperatures for selected foods in freeze (Fellows, 2000)

Food	Collapse temperature(°C)
Coffee extract (content of solids, 25%)	–20
Apple juice (content of solids, 22%)	–41.5
Grape juice (content of solids, 16%)	–46
Tomato	–41
Sweet corn	–8 to –15
Potato	–12
Ice cream	–31 to –33
Cheddar cheese	–24
Fish	–6 to –12
Beef	–12

the moisture that leaves the frozen product. The vacuum pump also removes all non-condensable gases.

Heat must be applied to the product to remove water in the form of vapour from the frozen product. The amount of heat must be carefully controlled. If applying more heat than the one that the evaporative cooling can remove, the product warms above the onset temperature of ice melting, which can lead to an irreversible collapse of the food structure, restricting the rate of vapour transfer and effectively ends the freeze drying process. Ice melting in frozen solutions may cause foaming during freeze drying. Heat can be applied directly through a thermal conductor shelf, by radiant heaters or by microwaves.

After all ice has sublimed, some unfrozen water is still present in the product. The residual water content may be even 7–8%. Continued drying at higher (ambient) temperatures is needed for desorption of unfrozen water whilst retaining the low pressure.

The endpoint of the primary drying (sublimation) can be determined as follows: (1) when the minimum pressure attainable by the system is reached, no more water vapour is leaving the product or (2) when the evaporative cooling ends the product temperature rises to equal the shelf temperature.

12.1.1. Freeze DryingTime

The factors that control the freeze drying time are described in the Equation (12.1):

$$t = \frac{x^2 \rho (m_1 - m_2) H_s}{8 k_d (T_s - T_i)} \tag{12.1}$$

where t (s) is the drying time, x (m) the thickness of food, ρ (kg m^{-3}) the bulk density of dry food, m_1 the initial moisture content and m_2 the final moisture content in dry layer, H_s (J kg^{-1}) the latent heat of sublimation, k_d ($W \cdot m^{-1} \cdot K^{-1}$)

the thermal conductivity of the dry layer, T_s (°C) the surface temperature and T_i (°C) the temperature at the sublimation front.

The basic assumption of Equation (12.1) is that the rate of freeze drying is mainly dependent on the velocity of heat transfer through the already dried material from the surface to the inner part and on the velocity of the water vapour transfer to the opposite direction. When heat is transferred through the dry layer, the relationship between the pressure in the chamber and the pressure at the ice surface follows the Equation (12.2):

$$p_i = p_s + \frac{k_d}{bH_s}(T_s - T_i) \tag{12.2}$$

where p_i (Pa) is the partial pressure of water at the sublimation front, p_s (Pa) the partial pressure of water at the surface and b ($kg \cdot s^{-1} \cdot m^{-1}$) the permeability of the dry layer.

The drying event is characterized by the diffusion coefficient, D (Equation 12.3) and the mass transfer coefficient, k, which can be determined using the Equation (12.4):

$$1 - Y = \frac{2MV_wD(p_i - p_a)t}{x_L^2RT_a(1 - Y)} - \frac{2D}{k_m x_L} \tag{12.3}$$

$$K = \frac{k_m}{RT} \tag{12.4}$$

where M ($kg \cdot kmol^{-1}$) is the mole mass of water, V_w ($m^3 \cdot kg^{-1}$) is the relation of the volume of the dry matter to the mass of water before drying, D ($mol \cdot s^{-1} \cdot m^{-1}$) is the diffusion coefficient, p_i (Pa) is the partial pressure of water vapour in the frozen matter, p_a (Pa) is the partial pressure of the water vapour in the surrounding atmosphere in the chamber, t (s) is the time, x_L (m) is the total thickness of the material layer, R (8.314 $J \cdot mol^{-1} \cdot K^{-1}$) is the gas constant, T_a (°C) is the temperature of the frozen material, $Y = y/x_L$ (dimensionless) the fraction of the frozen material layer (Figure 12.4) and k_m ($m\ s^{-1}$) is the coefficient.

V_w ($m^3 \cdot kg^{-1}$) is the ratio of the volume of the dry material to the mass of water in the beginning of freeze drying and can be calculated using Equation (12.5):

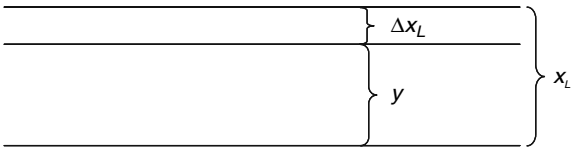

Figure 12.4. Dimensions of the material to be freeze dried; total thickness of the material layer (x_L), thickness of the dry material layer Δx_L) and thickness of the frozen material layer (y).

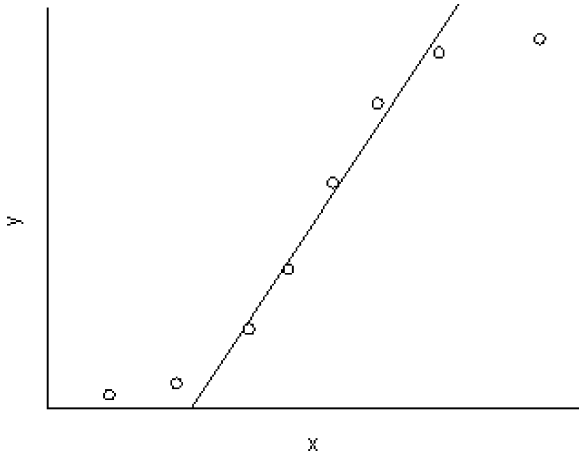

Figure 12.5. Graphic presentation of the freeze data; equation $y = kx + b$ is fitted to the data.

$$V_w = \frac{1}{(\omega_0 - \omega_f)\rho_d} \tag{12.5}$$

where ω_0 (kg H_2O/kg dry matter) is the moisture content in the beginning, ω_f (kg H_2O/kg dry matter) is the moisture content at the end of freeze drying and ρ_d (kg·m^{-3}) is the density of the ground, freeze-dried material.

Coefficient k_m and diffusion coefficient D are determined using the graph drawn using the values for y and x shown in Equations (12.6) and (12.7), respectively (Figure 12.5):

$$y \cong (1 - Y) = \frac{m_0 - m_i}{X_w m_0} \tag{12.6}$$

where m_0 is the mass of material in the beginning, m_i is the mass of material at the moment $t = i$ ($i = 0, 2, 4, 6, 8, 16$ h. . .) and X_w is the moisture content of material in the beginning of freeze drying.

$$x \cong \frac{t}{(1 - Y)} \tag{12.7}$$

Slope k and constant b obtained (Figure 12.5) are used in the calculation of values for diffusion coefficient D (Equation 12.8), coefficient k_m (Equation 12.9) and mass transfer coefficient K (Equation 12.4).

$$k = \frac{2MV_w D(p_i - p_a)}{x_L^2 RT_a} \tag{12.8}$$

$$b = -\frac{2D}{k_m x_L} \tag{12.9}$$

The diffusion coefficients and mass transfer coefficients obtained using various freeze drying conditions (i.e., various shelf temperatures and chamber pressure) are shown in table and their effect on freeze drying ratedrying rate may be predicted.

12.2. EXPERIMENTAL PROCEDURE

12.2.1. Apparatus

- Freezers at –20°C and at –80°C.
- Freeze dryer (FD8, Heto-Holten A/S, Denmark).

12.2.2. Material

Both solid, semi-solid and liquid material can be freeze dried. Important for a successful freeze drying process is an even and rather small (1–2 cm) thickness of the frozen food layer (e.g. apple slices), preferably 0.5–1 cm for frozen solutions (e.g. apple juice).

12.2.3. Procedure

1. Freeze the materials at around –20°C for 2–3 h and then at –80°C for at least 20 h (6×3 samples for each freeze drying conditions).
2. Cool down the condenser to about –80°C and the shelves to the selected temperature (e.g. –20, –10 and 0°C). Cooling of shelves to –20°C takes about 3 h.
3. Place the weighed, frozen material in Petri dishes onto the shelves of the freeze dryer.
4. Seal the chamber and adjust the pressure to the selected value (e.g., 0.2 and 0.5 mbar).
5. Heat from the shelves causes the ice to change phase.
6. Water vapour condenses onto the condenser in solid ice form.
7. Continue drying for 2, 4, 6, 8 and 16 h or until the drying process is finished (starting every time from the initial stage with new frozen samples of the same lot of food material).
8. Release the vacuum before opening the chamber.
9. Pack the dried material into plastic bags to avoid moisture transfer from the air.

12.2.4. Measurements

1. x_L (m), thickness of the material layer is measured before and after freeze drying by ruler.
2. X_w (kg H_2O/[kg H_2O + kg dry matter]), moisture content of the material is determined before starting the freeze drying in vacuum drying oven (at 60°C for 5 h or to constant weight). ω_0 (kg H_2O/kg dry matter), the moisture content in the beginning is calculated using the same data.
3. m_i (kg), the mass of the material is weighed at the moment $t = i$ ($i = 0, 2, 4, 6, 8, 16$ h...).
4. After freeze drying, $t = 16$ h, the residual moisture content, ω_f (kg H_2O/kg dry matter), is determined by drying in the vacuum drying oven (at 60°C for 5 h or to constant weight).
5. ρ_d (kg m^{-3}), density of the ground, freeze-dried material is determined using cylindrical glass and scales.

12.3. DATA ANALYSIS AND QUESTIONS

Decrease of the mass of the drying material is followed as a function of time. What is the effect of the:

(a) Drying parameters (p, T).
(b) Thickness of material layer.
(c) Structure of food material.
(d) Composition of food material on freeze drying rate.

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13

Determining Freezing Times of Food with a Plate Freezer

Cristina Luisa Silva, Magda Navarro de Noronha, and Alexandra Morim

OBJECTIVES AND LEARNING OUTCOMES

1. Determine experimentally freezing points and freezing times for different food products, using a plate freezer.
2. Compare the freezing points and freezing times of different water content food products.
3. Examine the applicability of empirical formula to estimate freezing times.

13.1. INTRODUCTION

Freezing is an excellent method for food preservation, because it reduces or stops the microbial action, chemical and enzymatic reactions and preserves the aroma, the nutritional value and other quality attributes.

During freezing storage, adequate package and the maintenance of lower and stable temperatures are essential factors for the preservation of quality and nutritive value of foods (Fellows, 1988).

CRISTINA LUISA SILVA, MAGDA NAVARRO DE NORONHA AND ALEXANDRA MORIM • Universidade Católica Portuguesa, Escola Superior de Biotecnologia, clsilva@esb.ucp.pt • *Universidade Católica Portuguesa, Escola Superior de Biotecnologia*

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Figure 13.1. Plate freezing equipment.

Since damages caused in food texture and other deterioration changes, due to concentration effects, happen more quickly during the "critical phase" of the temperatures' history (which occurs from –1 to –5°C), the going through this phase rapidly ensures that minimum quality changes during the freezing operation.

Plate freezing is a fast freezing method, once the foodstuff is in direct contact with two cold metallic surfaces. This process is used when the food geometry has two flat and parallel surfaces. The smaller the thickness of the food between these two surfaces, the faster will be the freezing process. The metal plates are good heat conductors and remove the heat from the food to the cooling fluid in an efficient way (Heldman, Dennis and Hartel, 1998).

The plate freezer used in this work is shown in Figure 12.1. It consists of refrigeration system (compressor, condenser, expansion valve and evaporator), using a refrigerant 12 as the cooling fluid. The compressor and the condenser are shown on the right side of Figure 13.1.

13.1.1. Freezing Plots

To understand the freezing process, it is essential to be familiar with the temperature change during food products freezing. If the temperature is monitored at the thermal centre of a food (the point that cools most slowly) as heat is removed, a characteristic curve is obtained (Figure 13.2) (Heldman and Singh, 1981):

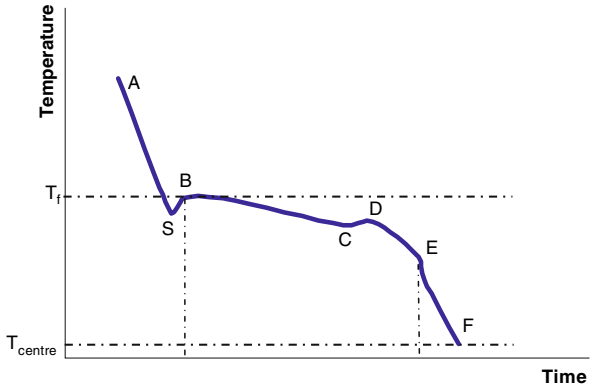

Figure 13.2. Time–temperature data during freezing.

AS – the food is cooled to a temperature below its freezing point, without ice formation (removal of sensible heat above freezing point). At point S the water remains liquid, although the temperature is below the freezing point. This phenomenon is known as supercooling and is never more than 10°C below the freezing point.

SB – the crystallization begins at point S. The release of latent heat of crystallization causes a rapidly rise in the temperature until the initial freezing point-freezing point (B). The first ice crystals begin to form.

BC – during this stage the major content of water (3/4) is crystallized. Latent heat is removed and ice forms, but the temperature remains almost constant. The ice formed during this stage results only in a moderate concentration of the solutes in the unfrozen phase.
CD – one of the solutes crystallizes out due to supersaturation releasing the latent heat of crystallizationcrystallisation causing a rise in the temperature to the eutectic temperature for that solute. After point D, each gram of ice formed is responsible for the successive and substantial increase of the frozen phase concentration, lowering the freezing pointfreezing point.
DE – crystallization of water and the solutes continues. The total time taken (the freezing plateau) is determined by the rate at which heat is removed.

EF – the temperature of the ice–water mixture falls to the temperature of the freezer (removal of sensible heat below freezing).

13.1.2. Freezing Time Estimation

The freezing time is the critical factor required for freezing systems design and selection. The freezing time also establishes the system capacity. The estimation

of freezing times very important to assure the efficient selection of freezing systems.

The first and the most popular formula to estimate freezing times was proposed by Plank in 1913, and it was adapted for foods by Ede in 1949 (Ede, 1994),

Plank's equation:

$$t_f = \frac{\rho\,\lambda}{T_f - T_\infty}\left(\frac{P\,a}{h_c} + \frac{R\,a^2}{k_{frozen}}\right) \tag{13.1}$$

where a is the sample diameter or thickness (m), h_c is the convective heat transfer coefficient (W/(m^2 °C)), k_{frozen} is the thermal conductivity of the frozen product (W/(m °C)), M_w is the fraction of water content in the product, T_f is the the product freezing point (= –1.8 ° + °M_w) (°C), t_f is the freezing time (s), T° is the temperature of the freezing environment (°C), P, R are constants depending on product shape, ρ is the frozen product density (kg m^{-3}) and λ is the latent heat of fusion (J kg^{-1})

The magnitude of the constants (*P and R*) in Plank's equation depends on product geometry. The values for three common product shapes (infinite slab, infinite cylinder and sphere) are presented in Table 13.1.

More recently, Cleland and Cleland (1992, 1994) developed the following equation to estimate the freezing times. This equation can be applied to any product geometry:

$$t_f = \frac{1}{E}\left[\frac{\Delta H_1}{\Delta T_1} + \frac{\Delta H_2}{\Delta T_2}\right]\left[\frac{R}{h} + \frac{R^2}{2^* k_{frozen}}\right] \tag{13.2}$$

where E is the shape factor, h is the surface heat transfer coefficient (W m^{-2} °C^{-1}), k_{frozen} is the thermal conductivity of the frozen product (W m^{-2} °C^{-1}), R is the shortest distance between the product geometric centre and the surface (m) and t_f is the freezing time (s).

The other equation terms are defined as follows:

$$\Delta H_1 = \rho\, C_{p,unfrozen}(T_{initial} - T_{fm}) \tag{13.3}$$

$$\Delta H_2 = \rho\left[\lambda + C_{p,frozen}(T_{fm} - T_{centre})\right] \tag{13.4}$$

Table 13.1. Geometry constants for Plank's equation

Geometry	*P*	*R*
Infinitive plate	1/2	1/8
Infinite cylinder	1/4	1/16
Sphere	1/6	1/24

The average freezing temperature T_{fm} is given by:

$$T_{fm} = 1.8 + 0.263\, T_{centre} + 0.105\, T \tag{13.5}$$

$$\Delta T_1 = \frac{T_{initial} + T_{fm}}{2} - T_{\infty} \tag{13.6}$$

$$\Delta T_2 = T_{fm} - T_{\infty} \tag{13.7}$$

where $C_{p,\ unfrozen}$ is the sensible heat capacity above freezing point (J/(kg °C)), $C_{p,\ frozen}$ is the sensible heat capacity below freezing point (J/(kg °C)), λ is the latent heat of fusion ($J \cdot kg^{-1}$), ρ is the frozen product density ($kg \cdot m^{-3}$) and T_{centre} is the final temperature at the product centre.

It must be emphasized that the estimation of freezing time by the Plank's equation takes into consideration just the time from points B to E in Figure 13.2. On the other hand, the Cleland and Cleland's equation considers the time from points A to F in Figure 13.2, therefore it requires the specification of the initial and final product temperatures ($T_{initial}$ and T_{centre}).

13.2. EXPERIMENTASL PROCEDURE

13.2.1. Apparatus

- Plate freezer Armfield (Figures 13.3–4).

The fluid coming from the high-pressure condenser, passes through the expansion valve and is broken up into two chains (one for the superior plate and another one for the inferior). Afterwards, the fluid is evaporated in the pipes of each plate's evaporator (see Figure 13.4). This absorbs heat from the plane metallic surfaces in contact with the food. The gas of the evaporators returns to the compressor to complete the cycle. In the entry and exit, of the evaporator, there are measures of pressure. This operation could be shown in a P-H diagram (pressure versus enthalpy) of R-12 as shown in annex book.

- Six thermocouples and a time–temperature registration apparatus.
- Mix of ice–water for the calibration of thermocouples.
- Mercury thermometer for the calibration of thermocouples.
- Balance.
- Petri dish and vacuum stove for water content determination in mashed food.

13.2.2. Materials

- Two mashed food products, with different water content: e.g., potato and carrot purees.
- Adherent film.

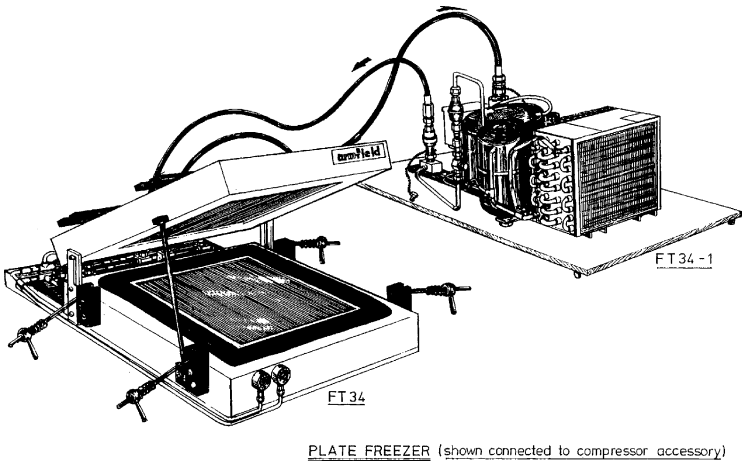

Figure 13.3. Plate Freezer.

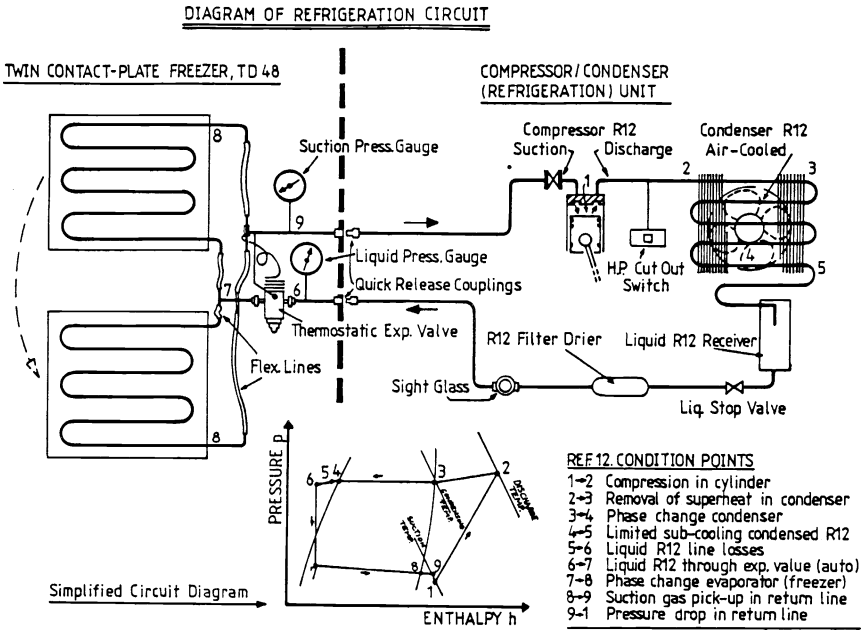

Figure 13.4. Plates evaporator.

13.2.3. Procedure

1. Turn on the plate freezer 15 min before the start of the experiment (please, ask for technical help).
2. Calibrate the thermocouples.
3. Measure the temperature of the ice–water mixture using the mercury thermometer (leave enough time so that the equilibrium is reached).
4. Immerse all the thermocouples into the ice–water mixture (leave enough time so that the equilibrium is reached).
5. Record the temperatures indicated by the thermocouples during 3 min.
6. For each thermocouple, calculate the average and the standard deviation from the real temperature.

13.2.4. Food preparation

1. Weight equal amounts (500 g) of the two mashed products and place each of them in plastic bags.
2. Compact the mashed samples to achieve a final approached thickness (infinite geometry plate, E = 1).
3. Measure, or estimate, important thermo-physical properties of the mash products.
4. Place both packages in the plate freezer.
5. Insert thermocouples between the packed mashed products and each surface.
6. Close the plate to compress the samples to the final required form and open them immediately.
7. Place a thermocouple in the centre of each package, confirming that the thermocouple is at the centre of the samples.
8. Close the plate freezer again.
9. Register the data of T *vs* t during the freezing process. Register also the values of pressures at 10 min intervals.
10. Stop the operation when the temperature in the center of the samples reaches below –10°C.
11. Turn off the plate freezer and open the plates.
12. Measure the dimensions of the samples and remove the packages.
13. Remove the thermocouples only when the samples have sufficiently defrosted.
14. Leave the freezer to defrost and then clean and dry the metallic surfaces.

SAFETY ASPECTS

The equipment uses refrigerant (cooling) 12, which is harmful to the ozone layer in case of leakage. Only qualified personnel can release connections, or carry out any maintenance.

Please call management personnel to check any possible leakage.

13.3. DATA ANALYSIS AND QUESTIONS

Please refer to the experimental data in Annex 13.

1. Plot the freezing data (temperature at the centre of the food items versus freezing time) of the two mashed foods.
2. Determine the surface temperature ($T_{surface}$) of the metal plates.
3. Draw the operation on the P-H diagram (pressure versus enthalpy).
4. Estimate the ideal operation coefficient of performance (COP).
5. Determine or estimate the important thermo-physical properties of the of the mash products.
6. Compare the results obtained experimentally with the freezing time-freezing time estimated using the equations of Plank and Cleland & Cleland.
7. Discuss any deviation between the estimates and the experimental results.
8. Observe the original food, frozen and after thawing and discuss their quality attributes.

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14

Comparing Air Blast and Fluidized Bed Freezing

Margarida Vieira and Jorge Pereira

OBJECTIVES AND LEARNING OUTCOMES

1. Examine the freezing process in an air blast and fluidized bed freezer and how to control this operation.
2. Verify the accuracy of existing correlations' accuracy on the prediction of food freezing times.
3. Examine the way the refrigeration unit works with special emphasis to the kind of refrigerant used, *134a*, stressing the fact that *Freon 12* was the former refrigerant used, which was very harmful to the environment, namely the ozone layer and therefore had to be replaced some years ago.

14.1. INTRODUCTION

Freezing is a unit operation where the temperature of the food is decreased below its freezing point and during which part of the water content changes state, forming ice crystals (generally in foodstuff frozen at –29°C about 90% of the water content is frozen). The immobilization of water in ice and the consequent concentration of the solutes disolved in non-frozen water, lower the food a_w, inhibiting the growth of microorganisms and enzyme

MARGARIDA VIEIRA AND JORGE PEREIRA • Universidade do Algarve, Escola Superior de Tecnologia, mvieira@ualg.pt • Universidade do Algarve, Escola Superior de Tecnologia

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activity. The preservation is therefore reached by the combination of temperature, reduction of a_w and in some foods the applied pre-treatment (blanching).

The freezing process includes initially a removal of the sensitive heat until the food reaches its freezing temperature and from then on a removal of the latent heat of crystallization. At this stage, the freezing temperature starts decreasing slowly due to the crystallization of other food components leading to the concentration of the solutions. Finally, there is a period where the temperature keeps going down until the temperature of the freezer is reached. The heat removed is mostly due to the latent heat of freezing (335 $kJ \cdot kg^{-1}$) later used for the refrigerant gases compression in the freezing equipment.

14.1.1. Freezing Time

Once the latent heat of freezing is present in the non-steady state freezing process, the standard equations for heat conduction in non-steady state and the existent charts cannot be used to predict freezing times. In 1913, Plank derived an approximate solution for the freezing time, which is used frequently in engineering problems Equation (14.1). This equation was obtained after the following assumptions were taken:

- Food is at the freezing temperature although still unfrozen.
- The thermal conductivity of the frozen part is constant.
- All the components freeze at the frozen point with a constant latent heat.

Heat transfer by conduction in the frozen layer occurs so slowly that it can be considered in steady state:

$$t = \frac{\lambda \rho}{T_f - T_\infty}\left[\frac{Pa}{h_c} + \frac{Ra^2}{k}\right] \tag{14.1}$$

where *P and R* are constants that depend on the geometry of the food to freeze (Table 14.1).

For food in brick shape, for example, *P and R* are obtained through Figure 14.1 prepared by Ede, in 1952, where β_1 is the ratio between the second

Table 14.1. Values of for different geometries

	Slab inf.	Sphere	Cylinder inf.
P	1/2	1/6	1/4
R	1/8	1/24	1/16

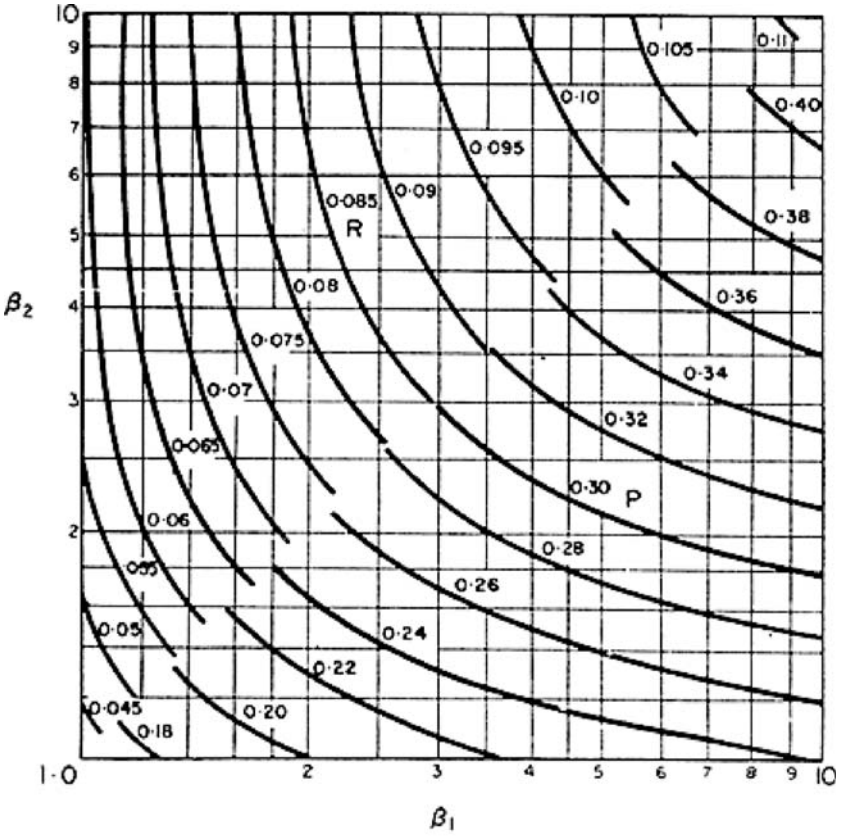

Figure 14.1. Values of for a product in a brick (based on Ede, 1949).

larger size and the smaller size and β_2 is the ratio between the larger size and the smaller size, which gives a *P* or *R* value.

The Plank equation does not take into account the food cooling time from its initial temperature *T*o to the freezing *T*f. However, the time can be determined by using charts for non-steady state, assuming that there is no freezing and using the thermophysical properties of unfrozen food. In 1955, Nagaoka et al. proposed a change to the Plank equation, based on data obtained from, fish freezing, Equation (14.2):

$$t = \frac{\triangle H' \rho}{T_f - T_\infty}\left[\frac{Pa}{h_c} + \frac{Ra^2}{k}\right] \tag{14.2}$$

where,

$$\triangle H' = \lfloor 1 + 0,008(T_o - T_f) \perp c_{pd}(T_i - T_f) + \lambda + c_{pc}(T_f - T) \rfloor \tag{14.3}$$

In this equation, Nagaoka et al. (1955), take into account the sensible heat above and below the initial freezing point, cp_d and cp_c, assuming that all the latent heat is removed at constant temperature T_f, taking into account the final temperature of the product, T and adjusting the value of the latent heat of fusion according to the water content of the product.

Cleland and Earle, in 1979, proposed new changes to the Plank equation and rewrote it in a dimensionless form:

$$N_{Fo} = \frac{P}{N_{Bi}\,N_{St}} + \frac{R}{N_{St}} \tag{14.4}$$

where N_{Fo} is the Fourier number ($\alpha t/a^2$), N_{Bi} is the Biot number (ha/k), N_{St} is the Stefan number ($Cpf(Tf\text{-}Tm)/\lambda$), NPk is the Plank number ($Cpu(Ti\text{-}Tf)/\lambda$) and a is the thermal diffusivity.

with *P and R* estimated using the following equations:

Slab

$$P = 0,5072 + 0,2018N_{Pk} + N_{St}(0,3224N_{Pk} + \frac{0,0105}{N_{Bi}} + 0,0681) \tag{14.5}$$

$$R = 0,1684 + N_{St}(0,2740N_{Pk} + 0,0135) \tag{14.6}$$

Cylinder

$$P = 0,3751 + 0,0999N_{Pk} + N_{St}(0,4008N_{Pk} + \frac{0,071}{N_{Bi}} - 0,5865) \tag{14.7}$$

$$R = 0,0133 + N_{St}(0,0415N_{Pk} + 0,3957) \tag{14.8}$$

Sphere

$$P = 0,1084 + 0,0924N_{Pk} + N_{St}(0,231N_{Pk} - \frac{0,3114}{N_{Bi}} + 0,6739) \tag{14.9}$$

$$P = 0,0784 + N_{St}(0,0386N_{Pk} - 0,1694) \tag{14.10}$$

The choice of freezing equipment depends on:

The freezing speed required,
Packaging requirements, size and shape of food.
Size of production, i.e., number and kind of products and option of operation (batch or continuous).

Taking these parameters into account, freezes can be classified into:

Figure 14.2. Air blast and fluid bed freezer.

1. Mechanical freezers in which the air blast, the fluidized bed and the plate freezers are included.
2. Cryogenic freezers, which utilize CO_2 and liquid N_2 as a refrigerant.

14.1.2. The Air Blast and Fluidized Bed Freezer

This type of freezers, utilize cold air at high speeds and may be designed in various configurations. They are used to freeze very dense products or products packed in big packages which can be placed in trays (batch system, Figure 14.2) or conveyor belts (continuous system) and exposed to the high speed flowing air.

The fluidized bed freezer is only a modified version of an air blast freezer in which the products are frozen by fluidization in air at very low temperature. The size (density) of the product to be frozen is limited by the energy needed to produce the required air velocity for fluidization. The frozen product is commercially designated by "instant-quick-frozen" (IQF) (Heldman and Singh, 1982).

14.2. EXPERIMENTAL PROCEDURE

14.2.1. Apparatus

- Air blast freezer Armfield.
- Temperature registrator Ellab.
- Thermocouple.

14.2.2. Material

- One meat ball.
- One sausage.
- One fish filet.
- Potatoes.
- Green peas.

14.2.3. Procedure

1. Open the tap water. The compressor cannot work if the water is not running to cool down the machine!
2. Press the refrigeration button of the air blast freezer after turning on the main switch and the compressor switch.
3. Set the temperature in the thermostat to –25°C. As soon as the temperature of the compressor is reached the compressor stops working continuously, although the evaporator fan keeps working. At this point, the freezer is ready to start freezing rapidly.

14.2.3.1. Tray Freezing with Forced Air Ventilation

1. Introduce one meat ball, one sausage and one fish filet in alternate trays. Write down the dimensions of the food items to freeze considering the meat ball as a sphere, the sausage as a cylinder and the filet as a slab.
2. Insert a thermocouple in the thermal centre of each food item and another one inside the chamber.
3. Set the temperature registering device to register the temperature every minute. Wait until the food items reaches a constant temperature.

14.2.3.2. Fluidized Bed Freezing

1. Place the diced potatoes or peas, previously drained to avoid too much water over the grid where the fluidized air runs. Insert a thermocouple in the centre of the peas and another one in the diced potatoes. Write down the dimension of the food items Place a thermocouple over the grid to indicate the freezer temperature.
2. Loosen the screw on the side of the protection guard, raise it and then tighten again. Do not forget to place back the acrylate plate to cover the tray chamber.
3. Set the temperature registering device to register the temperature every 30 s. Wait until food reaches a constant temperature.
4. The air flow rate from the fan can be adjusted using a handwheel which regulates a plate inside the damper. The maximum air rate that can be reached is about 10 $m \cdot s^{-1}$.

14.3. DATA ANALYSIS AND QUESTIONS

1. Plot the temperature at the centre of the food items as a function of the freezing time.
2. Determine the freezing time.
3. Determine the freezing time using the equations of Plank and the Nagaoka et al. (1955) for each one of the food items. Compare with the result obtained experimentally and discuss any differences.
4. Using the experimental results estimate the convective coefficient of heat transfer.

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15

Pasteurization with a Plate Heat Exchanger

Margarida Vieira, Jorge Pereira and Cornelius Lucher

OBJECTIVES AND LEARNING OUTCOMES

1. Demonstrate the operation of a plate exchanger, examining fundamental principles of thermal process engineering, namely, heat transfer, residence time and thermal degradation (of ascorbic acid).
2. Discuss the thermal energy in an efficient way.

15.1. INTRODUCTION

Plate heat exchangers are used throughout the food industry to transfer heat from one medium to another. They are commonly used for low-viscosity fluids and in the food industry they are often used for pasteurization of liquid foods.

MARGARIDA VIEIRA AND JORGE PEREIRA • Universidade do Algarve, Escola Superior de Tecnologia, mvieira@ualg.pt, jape@ualg.pt. CORNELIUS LUCHER • Technische Universität Berlin Department of Food Biotechnology and Food Process Engineering, cornelius.luscher@tu-berlin.de

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15.1.1. Thermal Preservation with Heat Exchangers

By definition, thermal pasteurization results either in the destruction of pathogenic organisms in non-acid foods, or reducing the number of spoilage organisms and/or inactivateing enzymes in acidic foods. In the USA 98% of juices are pasteurized and the Food and Drug Administration has stated (FDA, 1998) that any juice should receive a 5 D reduction in spoilage or pathogenic microorganisms. In fruit juices processing, if an hygienic procedure is used throughout the whole process, including fruit surface treatments, such as sanitizers and mechanical scrubbers, a pasteurization process should, therefore, be sufficient to produce commercially sterile juice during the pre-established shelf-life period and until the package is opened (Mcintyre et al., 1995). Milk is also often pasteurized when distributed in a cold chain.

15.1.2. Thermal Processing Effects on Quality and Safety Attributes

To date, the design of food thermal processes has assumed conservative safety factors, which, although reliable, result in over processing that adversely affect food quality. Reduction of microbial loads to a safe level (pasteurization or commercial sterilization), while avoiding major changes in quality attributes of the product, nutritional attributes (e.g., vitamins), as well as sensorial attributes (e.g., texture), became one of the most important fields in food research. Generally, a 10°C increase in the process temperature only doubles the rate of reaction for chemical degradation (e.g., vitamins or colour pigments degradation) whereas for bacteria and spores, a tenfold increase is often obtained (Holdsworth, 1985). This behaviour allows the finding of the conditions for thermal processing, ensuring a commercially sterile food product, with a maximum retention of quality. A High Temperature Short Time process is in most cases the best choice. Therefore, knowledge of thermal kinetic parameters of the quality and safety attributes, along with a reaction model, in a food product allows prediction of concentration retention or microbial survivors for a given thermal process.

15.1.3. Kinetics of Chemical Reactions and Microbial Destruction in Food

A kinetic study of a chemical reaction involves the evaluation of the rate of change of a chemical reactant (C) with time (t). These changes can be expressed mathematically by a generalized equation that relates the rate of degradation with environmental factors (E_i), such as temperature and pressure, and composition factors (F_j), such as a_w and pH (Eq. 15.1) (Saguy and Karel, 1980).

$$\frac{dC}{dt} = f(E_i, F_j) \tag{15.1}$$

Table 15.1. Order of important reactions that affect food quality

Quality loss reaction	n
Non-enzymatic browning, quality of frozen food	0
Browning, vitamins degradation, pigments degradation	0–1
Texture loss , microbial death or growth	1

In a more simplified way, if a reactant A degrades, during thermal processing or during storage, forming B, the rate of change of A can be expressed by Equation (15.2):

$$\frac{\mathrm{d}C_A}{\mathrm{d}t} = -k\,C_A^n \tag{15.2}$$

where k is the the reaction rate constant and n is the order of reaction with respect to reactant A.

The order of reaction, n, usually varies from 0 to 1 for the most well known reactions in food (Table 15.1) (Taoukis *et al.*, 1997).

By integrating Equation (15.2), Equation (15.3) is obtained:

$$\int_{C_0}^{C} \frac{\mathrm{d}C_A}{C_A^n} = -k \int_0^t \mathrm{d}t \tag{15.3}$$

In Table 15.2 we can see the equation form for $n = 0$ (zero-order) or 1 (first order reaction).

The inactivation of microorganisms, although not yet fully understood, is known to be logarithmic and can be treated as an unimolecular reaction of first order taking into account any deviation (Etsy and Meyer, 1922; Stumbo, 1973; Geankoplis, 1993). Usually, the designation C for concentration of reactants is replaced by N for number of microorganisms (Equation 15.4).

$$\ln \frac{N}{N_0} = -k\,t \tag{15.4}$$

However, thermobacteriologists prefer to express the inactivation in terms of a logarithmic reduction, being 1 log reduction the time required to destroy 90% of the cells, designated by the D-value (decimal reduction time). If a

Table 15.2. Integrated forms of the rate of reaction, as function of the reaction order

N	Equation form
0	$C_A - C_{A_0} = -kt$
1	$\ln \frac{C_A}{C_{A_0}} = -k\,t$

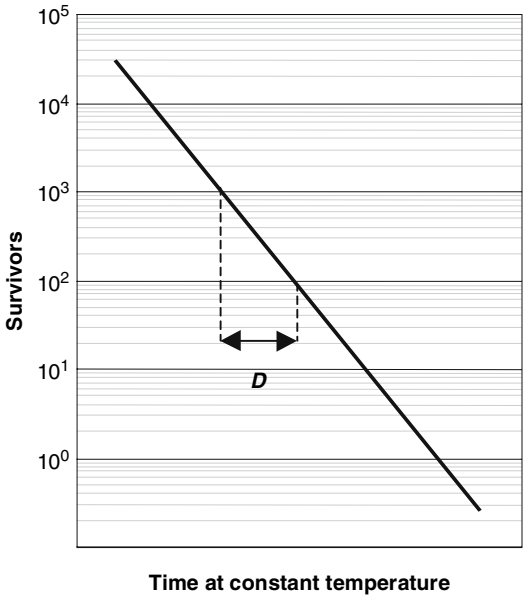

Figure 15.1. Survivors curve.

survivor curve is considered Figure15.1 the slope of this curve is given by Equation (15.5):

$$\frac{\log N - \log N_0}{t} = \frac{1}{D} \tag{15.5}$$

Equation (15.6) can be given in a form similar to Equation (15.4):

$$\log \frac{N}{N_0} = -\frac{t}{D} \tag{15.6}$$

The relationship between k and D can be derived and is given by Equation (15.7):

$$D = \frac{2.303}{k} \tag{15.7}$$

15.1.4. Temperature Dependence

The temperature dependence of the rate of change of any reactant (quality factor) is the most important environmental factor to be studied in food undergoing thermal preservation. Among several existing models, that were developed to describe it, the Arrhenius model is the most used, because it has the broadest temperature range of application although it is difficult to visualize. The Bigelow Method can be applied in temperature ranges of no more than 30°C.

15.1.4.1. The Arrehnius Model

The temperature dependence of the rate constant can be described by the Arrhenius law equation,

$$k_{(T)} = k_0 \exp^{\left(-\frac{Ea}{RT}\right)} \tag{15.8}$$

where T is the absolute temperature (K), Ea is the Arrhenius activation energy ($kJ \cdot mol^{-1}$), k_0 is the frequency constant (min^{-1}) and $R$is the Universal Gas constant (8.314 $J \cdot mol^{-1} \cdot K$–1)

Ea is assumed to be constant over the temperature range. This method was proved to be valid over a large range of temperatures (4–160°C), as mentioned before (Lund, 1986).

Due to the large difference in magnitude between Ea and k_0, a reference temperature, T_{ref}, was introduced to rescale the parameters (Nunes *et al*., 1993). This transformation not only brings stability to the numerical integration and parameter estimation, but also, by using a reference rate constant, k_{ref}, the equation can be applied in the most interesting range of temperatures including the reference temperature T_{ref}.

$$k_T = k_{ref} \exp\left[-\frac{Ea}{R}\left(\frac{1}{T} - \frac{1}{T_{ref}}\right)\right] \tag{15.9}$$

15.1.4.2. The Bigelow Model

Bigelow (1921) introduced the thermal death time (TDT) concept, where curves that represent the lethal effect of temperature on the resistance of microorganisms, often called thermal death time curves or TDT curves, are obtained for a specific microorganism by plotting D-values against the respective temperatures (Figure 15.2).

The slope of the TDT curve is, therefore, given by:

$$\frac{\log D_2 - \log D_1}{T_2 - T_1} = \frac{1}{z} \tag{15.10}$$

where z accounts for the sensitivity of the microorganisms to the different temperatures, and is defined as the number of degrees Centigrade or Fahrenheit needed for the TDT curve to cross a log cycle (Stumbo, 1973).

Being the general equation of the TDT curve given by:

$$\log D_2 - \log D_1 = \frac{1}{z}(T_1 - T_2) \tag{15.11}$$

This equation can be presented in a similar way the Arrhenius equation as:

$$D = D_{ref} 10^{\frac{(T\,ref - T)}{z}} \tag{15.12}$$

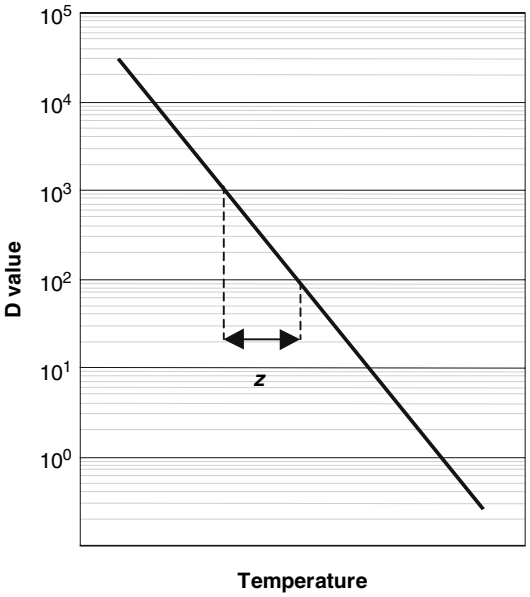

Figure 15.2. Thermal Death time (*TDT*) curve.

where D_{ref} is the decimal reduction time at T_{ref} (min) and T_{ref} is the reference temperature (°C).

15.1.5. Continuous Thermal Processes

In thermal processing of liquid foods, the food industry prefers increasingly continuous flow combined with aseptic processing, as opposed to batch processing of pre-packaged products, such as can, glass jar and retortable pouch processing. This preference is mainly due to the possibility of achieving a higher quality, together with energy savings and a more streamlined process (Swartzel, 1984). Another advantage comes from the fact that continuous flow allows different kinds of packaging shapes and materials to be used and, consequently, the consumer is provided with more attractive and convenient packages.

15.2. EXPERIMENTAL PROCEDURE

"The total time of the experiment should be around 6 h. The training exercise is one of 5 days of practical training on "Thermal Process Engineering"."

15.2.1. Apparatus

- One plate heat exchanger.
- Two pumps with tanks.

- One three-way valve.
- Gauges as indicated.
- Various tubes/hoses and fittings.

15.2.2. Materials

- Media: water, hot water, salt, ascorbic acid.
- Analysis: HPLC.

The system consists of a plate heat exchanger (Alfa – Laval) with two heat exchange sections ($A_A = 1.39\ m^2$; $A_B = 0.7\ m^2$). Hot water (95–98°C) is used as heating medium in section *B*, whereas heat recovery of the product is applied in section *A* (Figure 15.3).

After section *B*, holding tubes of different length can be used to achieve holding times at high temperatures.

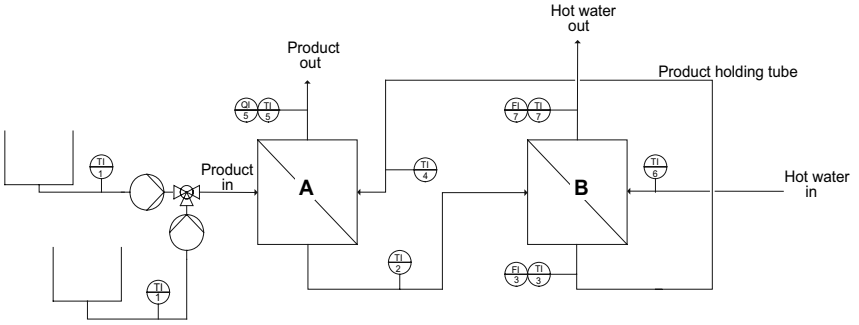

Figure 15.3. Schematic view (counter current flow) of a plate heat exchanger.

Table 15.3. Measuring points

Name	Description
T1	Product in
T2	Product between the sections
T3	Product before holding tube
T4	Product after holding tube
T5	Product out
T6	Hot water in
T7	Hot water out
F3	Flow rate product
F7	Flow rate hot water
Q5	Conductivity at product out

The temperatures are measured either by thermocouples or are read off at the temperature displays of the flow/conductivity gauges. Flow is measured by Coriolis gauges.

15.2.3. Procedure

15.2.3.1. Heat Transfer

Water is used as the liquid product. The exchanger is operated as shown in the scheme (counter current). The product flow rates is set to approximately 3, 3.6 , 4.8 and 8.4 $\cdot$ h^{-1}, the flow of the hot water is adjusted in the way that T6 is approximately constant. When steady state is reached, temperatures and flow rates are noted.

Afterwards the setup of the system is changed in the way that the heat recovery in section *A* is operated in co-current flow. When steady state is reached, temperatures and flow rates are noted.

15.2.3.2. Residence Time Distribution

The goal is to determine the residence time distribution of the experimental set-up at two different product flow rates: $V_1 = 3 \cdot h^{-1}$; $V_2 = 8.4 \cdot h^{-1}$. A tracer (5% of NaCl) and detection method (electrical conductivity, Crison conductometer) are used. A calibration curve was first done with different concentrations of NaCl in water as a funtion of their respective conductivity. The results obtained, for each flow rate at the exit of the cooling tube, showed a RTD curve with a symmetrical Gaussian-like shape and an extended tail, revealing that part of the flowing fluid was held back in dead regions of the equipment (Figure A15.1).

15.2.3.3. Thermal Degradation Kinetics

The degradation kinetics of ascorbic acid (vitamin C) shall be used as an indicator to quantify the thermal load. For this purpose the rig is operated with a solution of ascorbic acid ($c = 6\ g \cdot L^{-1}$). In order to simulate the conditions of a fruit juice the pH is set to pH 3 by addition of acid. Vitamins are otherwise (at neutral pH) not destroyed by a pasteurization process like the one analysed here. Additionally, the highest possible thermal load achievable in this unit should be realized by operating at a low flow rate. Two different flow rates shall be used.

The degradation is analysed by HPLC, (Zapata and Dufour, 1992). First of all a calibration line must be determined by analysing six dilutions of ascorbic acid with known dilution factors. Their concentration is now correlated to measured peak area. By using the calibration line the ascorbic acid content of the samples can be determined.

The inactivation of microorganisms can be described by the *D*-value. Similarly the *D*-value of other compounds (like vitamins) can be determined, which described the thermal stability of that compound at a given temperature. The *D*-value of ascorbic acid shall be determined approximately with the two concentrations measured by HPLC at the temperature in the holding tube (average temperature in the tube).

15.3. DATA ANALYSIS AND QUESTIONS

15.3.1. Heat Transfer

1. Calculate the following values for co-current and counter-current flow in section A.

   (a) $\dot{Q}_{A1}$ Heat flow [$kJ \cdot h^{-1}$]

   (15.1) $$\dot{Q} = \dot{m} \cdot c_p \cdot (T_2 - T_1)$$

   (b) Thermal efficiency of the heat recovery [–].

   (15.2) $$\varepsilon_A = \frac{\dot{m} \cdot c_p \cdot (T_2 - T_1)}{\dot{m} \cdot c_p \cdot (T_4 - T_1)}$$

   Where is the mass flow rate product, T_2, T_1, T_4 are specified above and given $C_P = 4.19\ kJ \cdot kg^{-1} \cdot K^{-1}$.

2. Calculate the heat flow [$kJ \cdot h^{-1}$] in counter-current flow for both sections, and for both media.

   $\dot{Q}_{A1} = \dot{m} \cdot c_p \cdot (T_2 - T_1)$ $\qquad$ $\dot{Q}_{B1} = \dot{m} \cdot c_p \cdot (T_3 - T_2)$

   mass flow rate HW:

   $\dot{Q}_{A2}{}^{\dot{m}} = \dot{m} \cdot c_p \cdot (T_5 - T_6)$ hot water $\qquad$ $\dot{Q}_{B2} = \dot{m}_{HW} \cdot c_p \cdot (T_6 - T_7)$

   T temperatures as specified above

3. Calculate the heat loss in both sections [$kJ \cdot h^{-1}$].

   (15.3) $$\Delta Q = \left|Q_{\text{uptake}}\right| - \left|Q_{\text{released}}\right| = |Q_1| - |Q_2|$$

4. Calculate the heat transfer factor k ($W \cdot m^{-2} \cdot K^{-1}$) in section A (counter-current flow).

   (15.4) $$\dot{Q} = k \cdot A_A \cdot \Delta T_m$$

   (15.5) $$\Delta T_m = \frac{(T_4 - T_2) - (T_5 - T_1)}{\ln \frac{T_4 - T_2}{T_5 - T_1}}$$

5. Calculate the resistance due to fouling R_B [$m^2 \cdot KW^{-1}$] in section A. What is its percentage of the total heat transfer resistance? (counter-current flow)

   (15.6) $$R_B = \frac{1}{k} - \frac{1}{k_{th}} = \frac{1}{k} - \left(\frac{1}{\alpha_1} + \frac{1}{\alpha_2}\right)$$

k_{th} theoretical heat transfer coefficient, assuming heat conduction without loss in the plate
$\alpha_1 = \alpha_2$ assumed heat transfer coefficient α with $\alpha\ \alpha = 2000\ \mathrm{W \cdot m^{-2} \cdot K^{-1}}$) on both sides of the plate.

6. Calculate the heat loss of the product [kW] in the holding tube (counter-current flow)

15.3.2. Residence Time Distribution

1. Determine graphically (in the diagram) approximate values of $t_{\min}$, $t_{\max}$, and t_m(sec).
   Determine the relative distribution (RD) and the efficiency of the hot dwell time (HT).

$$RD = \frac{\Delta t}{t_{\mathrm{m}}} = \frac{t_{\max} - t_{\min}}{t_{\mathrm{m}}} \tag{15.7}$$

$$HT = \frac{t_{\min}}{t_{\mathrm{m}}} \tag{15.8}$$

   Subscripts: m is measured values, w is water and s is salt solution.

2. Calculate an approximate value for both flow rates of t_{m} (s) and of the average velocity of flow w_{m} ($\mathrm{m \cdot s^{-1}}$) in the holding tube.

$$t_{\mathrm{m}} = \frac{L}{w_{\mathrm{m}}} = \frac{V}{\dot{V}} \tag{15.10}$$

   Given: V is the volume of the holding tube, L is the length of the tube, must be measured (about 8 m) and thediameter of the tube $d = 25$ mm.
3. Calculate for both flow rates the Reynolds number in the holding tube. Which state of flow (laminar/turbulent) is present at the respective flow rates?
   Given:

kinematic viscosity of water at 90°C, $\nu = 3.3 \times 10^{-7}\ \mathrm{m^2 \cdot s^{-1}}$

4. Describe in your own words the effect of increasing the flow rate on the state of flow, the Reynolds number and the resulting residence time distribution. Which kind of residence time distribution is desired in the food processing industry? Explain your answer.
5. The inactivation of microorganisms can be described by the D-value. Similarly the D-value of other compounds (like vitamins) can be determined, which described the thermal stability of that compound at a given temperature. Calculate the D-value of ascorbic acid approximately with

the two concentrations measured by HPLC at the temperature in the holding tube (average temperature in the tube) based on the data present in the annex 15 Tables A15.1 and A15.2.

15.3.3. Determination of the D Value of Ascorbic Acid

$T_3 =$ $T_4 =$ Average temperature $T_M =$

Prepare a diagram, which describes in an appropriate way the dependence of the concentration of ascorbic acid to the treatment time and determine the *D*-value graphically.

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16

Sterilization in Cans

Albert Duquenoy

OBJECTIVES

1. Visualized and understand the thermal behaviour of different types of contents.
2. Characterize the effect of a non-isothermal treatment on micro-organisms.
3. Characterize and quantify the thermal behaviour of a can.
4. Apply these characteristics to help finding better heat treatments that may achieve:
   - A sufficient effect on micro organisms so that almost all cans will be sterile or biologically stable (no micro organism growth).
   - A sufficient cooking without over-cooking.
   - The shortest treatment time, saving investment or energy.
5. Evaluate the theoretical microbiological impact of the thermal process.

16.1. INTRODUCTION

The canning process has several objectives: sterilization, cooking, conservation. These objectives are fulfilled by a two-step procedure: heating and cooling. The total effect of the procedure depends on the thermal behaviour of the product contained in the package.

ALBERT DUQUENOY • UMR Génie Industriel Alimentaire, ENSIA, duquenoy@ensia.fr

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The thermal behaviour of a canned product can be described by two simple models based on the way heat is transmitted inside the can: the perfect convection model (case of a low-viscosity liquid) and the pure conduction one (case of a solid). The second model is more complex and not very easy to use when the cooling phase occurs while the can content is not uniform in temperature.

The method described hereafter does not need any hypothesis on the heat transfer mechanism and thus can be applied to almost every case. It is commonly used in the canning industry as it is a very efficient tool to help establishing the best heat treatment.

This method needs to proceed to an experiment performed within conditions as close as possible to the industrial ones: same can, content, heating media and operating conditions (such as type and speed of rotation for example).

16.1.1. The Essential Concepts: The Sterilisation (or Pasteurisation or Cooking Value)

Note: The treatment temperature must be understood as the temperature at which the product is maintained.

This general concept considers that, irrespective of the treatment applied, an elevation of z°C will amplify the effect of a heat treatment by a factor of 10. The z-value may depend on the effect to be considered, but it takes some "standard" values, for instance a z-value of 10°C is considered for the destruction of spores and although this is not absolutely true for all spores, it is "sufficiently true" to be used efficiently. The z-value can have values ranging from 7 to 10 depending on the type of food (e.g., pH, ° Brix, a_w, etc.) or stage of the micro organism (spores or vegetative cells) (Ball and Olson, 1957). For many cooking effects such as colour, chemical or textural changes, enzymes inactivation, z is close to 30°C (very important as it determines the chemical stability to the canned food).

All these effects are expressed as the time of treatment at a certain temperature, so called "reference temperature" quoted T_{ref} hereafter, that would have the same effect as the treatment actually performed.

For example, keeping a product 110°C for 10 min will have the same effect on spores it contains, than a treatment at 120°C for 1 min. Two thermal treatments will have the same effect as long as:

$$t_1 \cdot 10^{\frac{T_1 - T_{ref}}{z}} = t_2 \cdot 10^{\frac{T_2 - T_{ref}}{z}} \tag{15.1}$$

The choice of T_{ref} is not critical as a change of its value will change both sides of the equality by the same factor. Nevertheless it is usual to choose the following values:

These concepts are particularly useful to quantify the effect of a variable temperature treatment, such as the one observed inside a canned food placed in a retort. In this case, the "values" would be defined as:

Table 16.1. T_{ref} values for different kinds of thermal processing

T_{ref} (°C)	z (°C or K)	Denomination of computed time
121,1	10	Sterilization value
75	7	Pasteurization value
100	30	Cooking value

$$\text{Value} = \int 10^{\frac{T(t)-T_{ref}}{z}}.\mathrm{d}t \tag{16.2}$$

where $T(t)$ is the temperature evolution inside the canned product. When this temperature is recorded with a time interval Δt the value is simply computed as a sum:

$$\text{Value} = \sum_i 10^{\frac{T(i.\Delta t)-T}{z}}.\Delta t \tag{16.3}$$

When the temperature is not uniform inside the can the coldest point inside the can should be used to determine the lowest value, thus the lowest effect. In many cases this point is not known: it can be inside a particulate which moves inside a sustaining liquid. Therefore the centre of the can will be used as a reference point.

Applying this last formula to the temperature evolution given in annex 16 will lead to the results shown in Table 16.2.

All these values apply to the sauce, not to the particulates. However they are good indications of the effect of the heat treatment. It is even used in home cooking! It is common to read in recipes for pasta cooking, the sentence "put the product in boiling water during x minutes" thus referring to the temperature of the heating medium, not the temperature of the pieces of food.

Theoretically the computed value could be "translated" in a result in terms of sterility, biological or chemical stability or degree of cooking. Actually it is not so easy to do so, and the computed values will be used as comparative or relative values: treatments with different temperature evolutions will have merely the same effect on the spores or flora provided that they "give" the same sterilization or pasteurization values. The same goes for cooking.

Table 16.2. Values for Can 1

Values (min)	Can 1
Sterilization	4.50
Pasteurization	$7.51\ 10^6$
Cooking	97.5

16.1.2. Quantifying the Thermal Behaviour: f and j Values

Heat transfer between two media separated one from the other by a conductive interface is proportional to their temperature difference implying that the heating rate is proportional to their difference. This leads generally to an asymptotic exponential temperature evolution leading to a null temperature difference. By applying this general rule to the difference between the outer media (heating or cooling) and the can temperature.

First the treatment must be characterized by the average value of the temperature plateau. Then the decimal logarithm of the difference between the plateau and the can temperature (its opposite at cooling) is plotted as a function of time (Figure 16.1). A straight line is obtained at least for the largest times. The line must be fitted to the end of the curve as the asymptotic behaviour is observed after some delay in the case when heat is transmitted slowly (conduction or poor convection) to the can centre. This is illustrated hereafter with doted

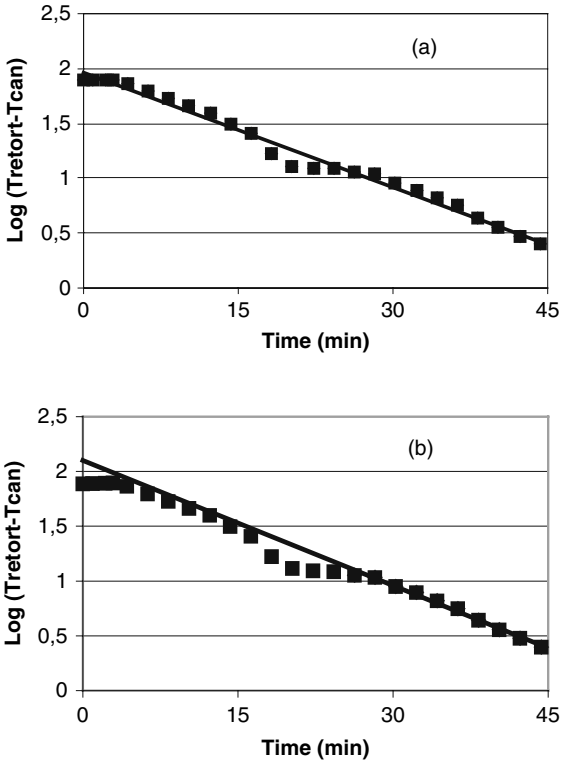

Figure 16.1. Plots of decimal logarithm of the difference between the plateau and the can temperature (its opposite at cooling) as a function of time.

experimental points. The line on the right is better fitted than the left one. For example, MS Excel, can be used to fit a line through the points after discarding the "bad" points (initial and sometimes irregular ones).

The line drawn as a slope α and intersects at $t = 0$ (beginning of the treatment stage) β from which we compute two other parameters:

$$f = -1/\alpha \tag{16.4}$$

and

$$j = \frac{10^{\beta}}{T_{\text{plateau}} - T_{\text{can}}(t = 0)} \tag{16.5}$$

These parameters characterize the thermal behaviour of the can in the conditions of the thermal process. They are quite independent of the values of the plateau and also of the initial (i.e., at the beginning of each stage) temperature of the can. Usually they receive a suffix 'h' for heating and 'c' for cooling, except for j_h which remains j

From the curves obtained with the temperatures recorded at annex 16 we can compute the following for can 1*:*

	f_h (min)	j	f_c(min)	j_c
Can 1	16.2	0.769	61.7	1.07

Generally the closer j is to 1, the better the temperature uniformity and reciprocally. This is observed for the cans containing only the sauce and can be explained by the rotation of the cans. For Can 1, j is even lower than 1. This is because of a fast initial evolution that slows down after a while. This is frequently the case when natural convection occurs, or a change in the sauce viscosity occurs (effect of cooking).

The values at cooling are very different for identical cans. This is often observed and can be attributed to the heat chock created by the cold media which disrupts the measurement of temperature by thermocouples as well as the movements of the product inside the can: there can be boiling inside as the cold can wall initiate condensation of the inner steam.

16.1.3. Using f and j to improve the Heat Treatment: The Ball Method

To find a thermal process that will achieve a desired sterilization value as well as a desired cooking value several trials should be performed in order to get closer and closer to the target. Ball's method enables us to choose a trial that will lead closer to this target as it uses what has been learnt from the previous trials about

the thermal behaviour of a canned product. The method uses many tables that differ according to the z-valuez value to consider mainly, but also some operating parameters. Canners generally use one table presented in annex 16 and is valid for $z = 10$, i.e., for a sterility target.

Ball's method is based on the hypothesis that f and j will remain constant if we only change the retort temperature or initial temperature of the can. A change in the come up time of the retort can also be taken into account. The principle of the method is based on two considerations. First f_h is used as a time base so that all cans will have the same new f_h of 1. Second, the retort temperature is used as the reference temperature.

To find the heating time necessary to obtain a target sterilization value F_0 with a retort temperature T_{retort}, the several steps of the Ball's Method should be followed in this way (Ball and Olson 1957):

1. Compute U applying the formula:

$$U = Fo \cdot 10^{\frac{121,1 - T\text{retort}}{10}} \tag{16.6}$$

   (U is the sort of sterilization value expressed with T_{retort} as the reference temperature).

2. Compute f_h/U (the inverse ratio would be the modified sterilization value expressed with the new time unit).
3. Go to the relevent table to find g that corresponds to the f_h/U ratio. g is the temperature difference between the can and the retort at the end of the heating stage. The higher the sterilization objective, the lower the f_h/U ratio, the longer the plateau must be and the closer the can comes to the retort and g is small. This can be seen in the table.
4. The line that has been fitted to the heating curve is defined by Equation (16.7):

$$Log(T\text{retort} - T\text{can}) = \beta + \alpha.t \tag{16.7}$$

And considering Equations (16.8), (16.9) and (16.10):

$$f = -1/\alpha \tag{16.8}$$

$$\beta = Log(j \cdot (T\text{retort} - T\text{can}(t = 0))) \tag{16.9}$$

$$j = \frac{10^{\beta}}{T\text{retort} - T\text{can}(t = 0)} \tag{16.10}$$

g should be close to this line so that we can find the time t necessary to come to this point (eq. 16.11):

$$t = \frac{1}{\alpha}(Log(g) - \beta) = -\frac{1}{\alpha}(\beta - Log(g)) \tag{16.11}$$
$$= f_h \cdot (Log(j \cdot (T\text{retort} - T\text{can}(t=0))) - Log(g))$$

Finally, t is given by Equation (16.12):

$$t = f_\text{h} \cdot Log(j \cdot \frac{T\text{retort} - T\text{can}(t=0)}{g}) \tag{16.12}$$

It is then also possible to take into account a chance in the initial temperature of the product.

All tables are built considering that the *f*-values are identical for heating and cooling. For the case when it is not so, the tables also indicate for every g, the portion, named *frac* in our table, of the total U/f_h reached at the end of heating. For the same value of g, the contribution of cooling will be the portion $(1 - frac)$ of U/f_c .For every g we thus obtain the total effect of heating and cooling by the sum of the two U-values thus computed. This computation will be iterated until the g-value leads to a total effect equal to the fixed target.

As an example of application of this method we shall determine the heating time of the can no. 1 containing raviolis so that we obtain a final sterilization value of 3 min instead of 4.5 with a retort temperature of 115°C instead of 117°C. The initial temperature will remain at 53.5°C, and the water for cooling at 17°C. The method gives:

1. Compute U : $U = Fo \cdot 10^{\frac{121.1 - T\text{retort}}{10}} = 3 \cdot 10^{\frac{121.1-115}{10}} = 12.2\,\text{min}$
2. Compute f_h/U: $f_\text{h}/U = 16.2/12.2 = 1.33$

(refer to the table in annex 16 to find that this ratio is in the interval):

f_h/U	G
1.25	0.49
1.50	0.69

Interpolating for $f_h/U = 1.33$ is found: $g = 0.49 + (0.69 - 0.49) \cdot ?\frac{1.33-1.25}{1.50-1.25} = 0.55$

4. Compute t:

$$t = f_\text{h} \cdot Log\left(j \cdot \frac{T_\text{retort} - T_\text{can}(t=0)}{g}\right) = 16.2 \cdot Log\left(0.769 \cdot \frac{115 - 53.5}{0.55}\right)$$
$$= 31.3\,\text{min}$$

Is this estimation a good one? Do we fulfil the conditions necessary to use the table? These conditions are: z = 10°C that is the case; $T_{retort} - T_{cooling} = 100°C$: we have 115 – 17 very close to 100; $j_c = 1.4$: that is not the case. As we do not have a better table we shall use it but there is still a condition not fulfilled concerning the equality of f between heating and cooling. Fortunately we can take this into account with our table.

The g-value we have used also correspond to a value of $frac = 0.901$ (by interpolation). From this we can compute the U/f_h effect during heating: $U/f_h = 0.901.(1/1.33) = 0.677$ and during cooling: $U/f_c = (1 - 0.901).(1/1.33) = 0.074$

The corresponding U-values are:
$U = f_h \cdot 0.677 = 16.2 \cdot 0.677 = 10.96$ for heating
and
$U = f_c \cdot 0.074 = 61.7 \cdot 0.074 = 4.59$ for cooling
The total U comes to 10.96 + 4.59 = 15.1 min which is too large as we expect $U = 12.2$ min.
So, trying again a higher g: (e.g.: 0.90) gives $f_h/U = 1.75$ and $frac = 0.882$: then
$U/f_h = 0.882.(1/1.75) = 0.504$
So that for heating, $U = 16.2 \cdot 0.504 = 8.15$
$U/f_c = (1 - 0.882).(1/1.75) = 0.067$
and for cooling, $U = 61.7 \cdot 0.067 = 4.16$
The total U is now the sum of the two terms (heating and cooling) 8.15 + 4.16 = 12.3 min, which is satisfactory. The heating time can be calculated now:

$$t = 16.2 \cdot \mathrm{Log}\left(0.769 \cdot \frac{115 - 53.5}{0.90}\right) = 27.8\,\mathrm{min}$$

The reader may practice this method on can nos 2–4 with the same objectives as we have used for can no. 1.

It must be underlined that the contribution of cooling to the total effect is small when the f-values are small: this is the case for fluid product in agitated cans. On the other hand, a large part of the effect will be gained during the cooling stage for conductive product. This makes the control of the retort more critical for this kind of product.

All computations above are only a mean to select the best trials to be performed which should never be replaced by them. The computed heating times must be validated even when the most sophisticated calculations are performed.

16.2. APPARATUS AND MATERIALS

Trials consist to process four cans, equipped with thermocouples, into a rotary pilot retort. Two cans are filled with a tomato sauce, the two others also contain dry pasta pieces (raviolis). They all contain the same mass of product. Two different trials can

be realized, with different temperature or rotation speed, according to the duration of the practical sequence. Cans can be rotated axially or end-over-end.

The use of can replicates allows to show the effect of the type of rotation (if different) on the importance of the different heat transport phenomena. Alternatively, it allows showing the repeatability of the process if the cans have the same type of rotation.

To change the heating temperature, together with the duration of heating so as to obtain the same final sterilization value, allows to show a differential effect on cooking.

16.2.1. Procedure

16.2.1.1. Sterilization of Tomato Sauce or Sauce with Pasta

1. Prepare the tomato sauce and heat it up to 60°C.
2. Fill all the cans with the same total mass of product, sauce or sauce with pasta.
3. Place thermocouples in the cans.
4. Seam the cans and place them in the rotary autoclave.
5. Adjust pressure and temperature levels to regulators.
6. Process the can with pure steam until the sterilization value attains a required value (to be defined previously in order to obtain a sterilization value of 3 min at the end of cooling).
7. Allow cans to cool.

16.2.2. Analysis of Final Result

Open the cans: observe their content. Measure the "tenderness" of pasta using any kind of "texturometer". These last measures can be (or cannot) related to a cooking value depending on the kind of rotation or the steam temperature.

16.3. DATA ANALYSIS AND QUESTIONS

Based on data in Annex 16,

1. Compute and compare f_h and f_c values. What do they depend on?
2. Is it easy to stop heating at a precise sterilization value?
3. Are thermal processes reproducible?

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Part III
Food Processing Operations

17

Ingredients in Infant Foods – Pregelatinized Amaranth Flour Using a Drum Dryer

Gernot Zweytick

OBJECTIVES AND LEARNING OUTCOMES

1. Review familiar with the drum drying technology.
2. Examine familiar with the measurement of hot paste viscosity.
3. Calculate the specific capacity of the heating surface.
4. Calculate the specific load of the heating surface.

17.1. INTRODUCTION

Amaranth is a plant originally grown in South America. It is a pseudocereal and has a starch content of about 60%. It does not contain gluten and thus, it can be used by people suffering from celiac disease and its use in infant food should be forced.

Amaranth grain (*Amarantus cruentus*) could be the basis of infant formula because of its combination of high digestibility and nutritional quality. For the use in infant food, the amaranth flour should be pregelatinized. The degree of gelatinization depends on the method. In comparison to other methods like extrusion cooking the drum drying technology reaches a degree of gelatinization of almost 100%.

GERNOT ZWEYTICK • Department of Food Science and Technology, University of Natural Resources and Applied Life Sciences, gernot.zweytick@boku.ac.at

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A disadvantage of the drum drying is the high energy input and the low rate of yield relating to the time. There is also a high thermal impact.

The responding factor hot paste viscosity is dependent on the raw materials used, the temperature of the surface of the drum dryer and the contact time. The influence of the raw material cannot be calculated or compared to other flours and mixtures.

Basically starch and its gelatinization degree is responsible for the hot paste viscosity but there are also some other components in cereals, which have some influence like pentosanes and other hydrocolloids.

The configuration of the drum dryer (heat, speed of rotation, dimensions of the drum) influences the product in terms of its degree of gelatinization.

During one rotation of the drum the temperature of the product reaches the temperature of the drum.

17.2. EXPERIMENTAL PROCEDURE

17.2.1. Apparatus

- Pin mill – Pallmann.
- Drum dryer – Goudsche Machinenfabriek, Gouda – heated by steam (steam pressure is 1.5–2 $\times$ 10^5 Pa and the surface temperature of the drum is about 130°C, speed of the drum: 1.5 rpm).
- Brabender Viscograph: Volume of the sample: 100 ml.

17.2.2. Materials

- Amaranth: 10 kg.
- Water.
- PE bags.

17.2.3. Procedure

1. Grind the Amaranth kernel using a pin mil comminution, product should contain all parts of the kernel.
2. Mix the flour with the same amount of water.
3. The drum dryer has to be heated up to the working temperature, step by step. Every 10 min, the pressure of the steam can be raised minimally (1×10^4 Pa).

When the drum reaches the desired temperature, the slurry of amaranth flour and water can be spread on the hot drum. During the rotation the slurry

is dried and a film of pregelatinized flour can be scraped off with a special knife. The obtained film has to be cooled by compressed air.

Before it is packaged or used for further mixtures, the dried product has to be milled again in a pin mill. The pregelatinized amaranth flour is then packed into PE bags which have to be hot sealed.

In Figure 17.1 the flow chart of the drum drying process is shown.

17.2.3.1. Analysis

For the *Analysis of the hot paste viscosity* it is necessary to observe the degree of gelatinization as this is related to the solubility of the end product.

A sample volume of 100 ml has to be filled into the viscometer. Then the heating and cooling system has to be started and the parameters (heating and cooling rate, time to keep the temperature) for the analytical process have to be adjusted in the corresponding software.

The temperature profile has to be adjusted as follows:

- Heating from 30 to 90°C within 8 min (7.5°C min^{-1}).
- Keeping the temperature for 4 min.
- Cooling from 90 to 30°C within 8 min (7.5°C min^{-1}).

In Figure 17.2 the hot paste viscosity of two different pregelatinized flours is shown.

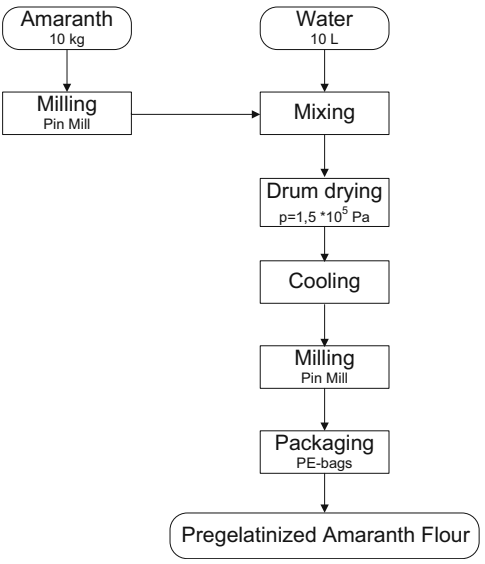

Figure 17.1. Flow chart of the drum drying process.

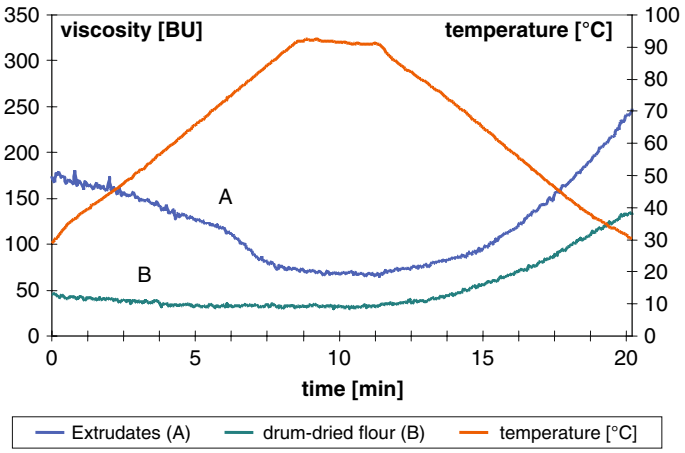

Figure 17.2. Analysis of the hot paste viscosity of two different pregelatinized flours.

Figure 17.3. Drum dryer. (Pilot plant-University of Natural Resources and Applied Life Sciences)

1. Specific capacity of the heating surface (h_m):

h_m describes the amount of water, which is evaporated per hour and per m^2. It is calculated as kg m^{-2} h^{-1} using Equations. (17.1), (17.2) and (17.3):

$$h_m = \frac{W}{F} \ [\mathrm{kg \cdot m^{-2} \cdot h^{-1}}] \tag{17.1}$$

where W is the amount of water which is converted to steam and F is the surface of the drum.

$$W = \frac{\frac{(3600 \cdot G)}{z} \cdot (\eta_b - \eta_e)}{(100 - \eta_e)} [\text{kg h}^{-1}] \tag{17.2}$$

where G is the amount of material before drying, z is the time used for drying, η_b is the water content before drying and η_e is the water content at the end (after drying).

$$F = \frac{(2 \cdot r \cdot \pi \cdot l \cdot \varphi)}{360} [\text{m}^2] \tag{17.3}$$

where r is the radius of the drum, l is the length of the drum and φ is the angle between feeding of the wet material and drop of the dried flour.

2. Specific load of the heating surface (q_m):

It is the heat quantity Q, which has to be applied to remove the amount of water W per hour. It is calculated as J kg^{-1} using eq. 17.4 and eq. 17.5.

$$q_m = \frac{Q}{F} [\text{kJ m}^{-2} \text{h}] \tag{17.4}$$

where Q is the heat quantity.

$$Q = W \cdot R \cdot G \cdot c_p (t_w - t_b) [\text{J kg}^{-1}] \tag{17.5}$$

where R is the evaporation heat, C_p is the specific heat of the wet material, t_w is the boiling temperature of pure water and t_b is the temperature of the wet material before feeding.

17.3. DATA ANALYSIS AND QUESTIONS

1. What are advantages and disadvantages of drum-dried products in comparison to spray-dried or extruded products?
2. What is the advantage using amaranth?
3. Why are pregelatinized flours used in infant food?
4. What is the main difference between the curves A and B in Figure 17.2?

5. Calculate the specific capacity of the heating surface (use values of Table. A17.1)
6. Calculate the specific load of the heating surface (use values of Table. A17.1)

18

Wheat Crisps – Extrusion Cooking Technology

Gernot Zweytick

OBJECTIVES AND LEARNING OUTCOMES

1. Review extrusion cooking technology.
2. Examine the texture measurement of extrudates.
3. Calculate the bulk density.
4. Calculate the expansion index.

18.1. INTRODUCTION

Spelt wheat is an ancient and under-utilized crop. As only wheat, rice and corn are of general interest worldwide the aim is the production of spelt wheat crisps for further use in granola bars. The texture, the bulk density and the expansion index of the extruded material will be measured.

Times of overproduction and environmental problems remind people of less productive but ecological beneficial crops. Furthermore they increase the interest in healthy nutrition and in food cultivated and produced without fertilizers or plant-protective agents.

Spelt wheat is ancient wheat covered with husks and able to grow even in colder climates and on poorer soils. But due to more difficult cultivation and processing, spelt wheat still remains a cereal for speciality products – though it was the main crop in Central Europe for centuries. Spelt wheat originates from South-west Asia; there it was developed from Emmer, which is a tetraploide wheat and *Aegilops squarrosa*, a wheat related grass variety. Following the

GERNOT ZWEYTICK • Department of Food Science and Technology, University of Natural Resources and Applied Life Sciences, 1190 Vienna – Austria, gernot.zweytick@boku.ac.at

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Danube it found its way to Central Europe. A change in climate favoured the cultivation of spelt wheat above the two other mentioned cereals.

During the last years environmental and economical problems of agriculture as well as the increasing demand for healthy food in industrial countries, caused a revival of spelt wheat as this cereal has some mentionable characteristics in chemical composition, besides the agricultural beneficial aspects of robustness, modesty and the hull, protecting against pollution or infections, (Qing-Bo-Ding et al., 2005).

Extrusion cooking is an effective and economic continuous process in which the physical, nutritional, and sensory properties of the raw material can be changed to a desired degree. Positive effects include increased digestibility, destruction of anti-nutritional constituents, toxic substances and micro organisms (Schiavone, et al., 2001).

The responding factors texture, bulk density and expansion index are dependent on the raw materials used and the configuration of the extrusion cooker. As only spelt wheat flour is used, the influence of the raw material cannot be calculated or compared to other flours and mixtures.

Starch and similar components like maltodextrines are responsible for higher expansion indices and lower bulk density while components like fat, sugar and salt have the contrary effect. Regarding the formation of water vapour during the discharge of the molten material through the dies the water content in the flour and water addition have the greatest influence on the expansion.

The influence of the configuration of the extruder on the product is more complex but in general the geometry of the screws, the temperature profile and the diameter of the dies have the greatest effect.

Mass temperature and mass pressure in the extruder depend on both – raw material and configuration of the equipment.

18.2. APPARATUS AND MATERIALS

18.2.1. Apparatus

- Pin mill – Pallmann.
- Extrusion cooker – Cincinnati Milacron: the used extrusion cooker is a conical counter rotating twin-screw extruder with four temperature zones, which can be adjusted separately.
- The configuration of the extruder is:
- Temperature zone 1: 70°C.
- Temperature zone 2: 100°C.
- Temperature zone 3: 130°C.
- Temperature zone 4: 160°C.
- Temperature of the screws: 70°C.
- Speed of the screws: 80 rpm.
- Screw type: 300 with two shearing zones.

- Die: 16×1 mm.
- Texture Analyzer TA-XT2i, Stable Micro Systems.
- Sliding caliper.
- Tubs.
- Scales.

18.2.2. Materials

- Spelt wheat: 20 kg.
- Water.
- PE bags.

18.3. PROCEDURE

For the preparation of the raw material the hulled whole kernels have to be milled using a pin mill. The obtained flour is then filled into the hopper of the extrusion cooker.

The system has to be heated up to the adjusted temperatures and then the engine of the screws has to be started. The dosing device and the pump for water addition are then started. The gravimetric dosing has to be adjusted to a value of 30 $kg \cdot h^{-1}$, the water dosing pump has to be started with maximum flow. When the product comes out first in a line it is quite wet and knead able. Depending on the original water content of the flour, the water addition has to be reduced until the product coming out of the extruder expands and feels dry and less compact. If the water content in the flour is higher than 12 %, no water addition is necessary. At this time the pelletizing head can be closed and started.

The obtained extruded material must be cooled for about 2 h and can then be packed or used in the mixture of granola bars. Figure 18.1 shows the scheme of the used extrusion cooker.

In Figure 18.2 the flow chart of the extrusion process is shown.

18.3.1. Analysis

18.3.1.1. Bulk Density

Tubs for measurement of the volume and scales for the weight are used. The tub is filled with extrudates and the weight is determined. The bulk density has to be expressed as $g \cdot l^{-1}$ using eq. 18.1.

$$\text{bulk density} = \frac{\text{weight [g]}}{\text{volume [L]}} \tag{18.1}$$

Weight: 783 g in 5 L.

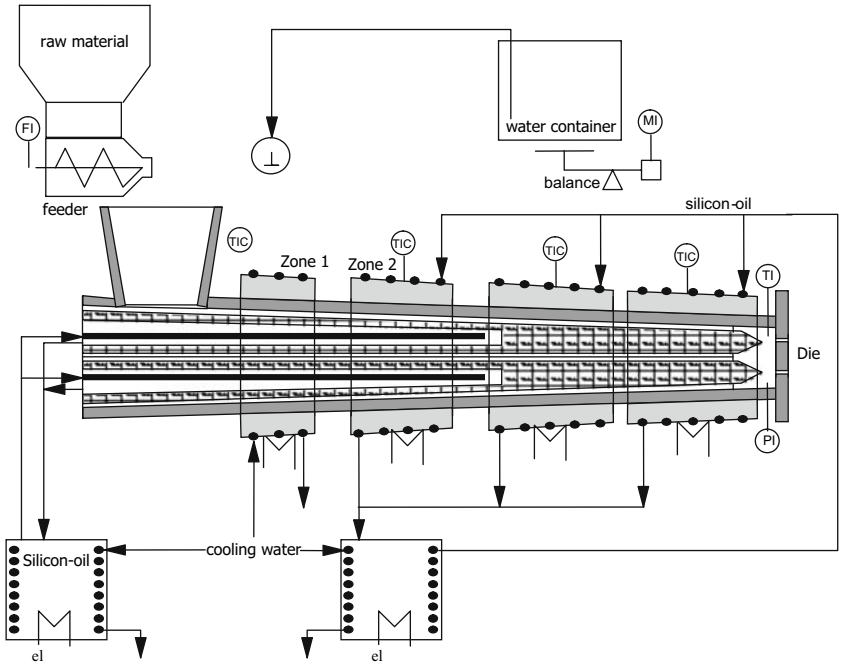

Figure 18.1. Scheme of the extrusion cooker.

18.3.1.2. Expansion Index

A sliding caliper is used to determine the diameter of extruded material and diameter of the die. The result is a dimensionless value calculated as shown in Equation 18.2.

$$\text{expansion index} = \frac{\text{diameter of the extrudates}}{\text{diameter of the die}} \tag{18.2}$$

Diameter of the die: 1 mm.
Diameter of the extrudates: 4.3 mm.

In Figure 18.3, examples (a) and (b) of two different samples are shown.

18.4. DATA ANALYSIS AND QUESTIONS

1. Which parameters are dependant on the expansion of the extruded material dependant ?

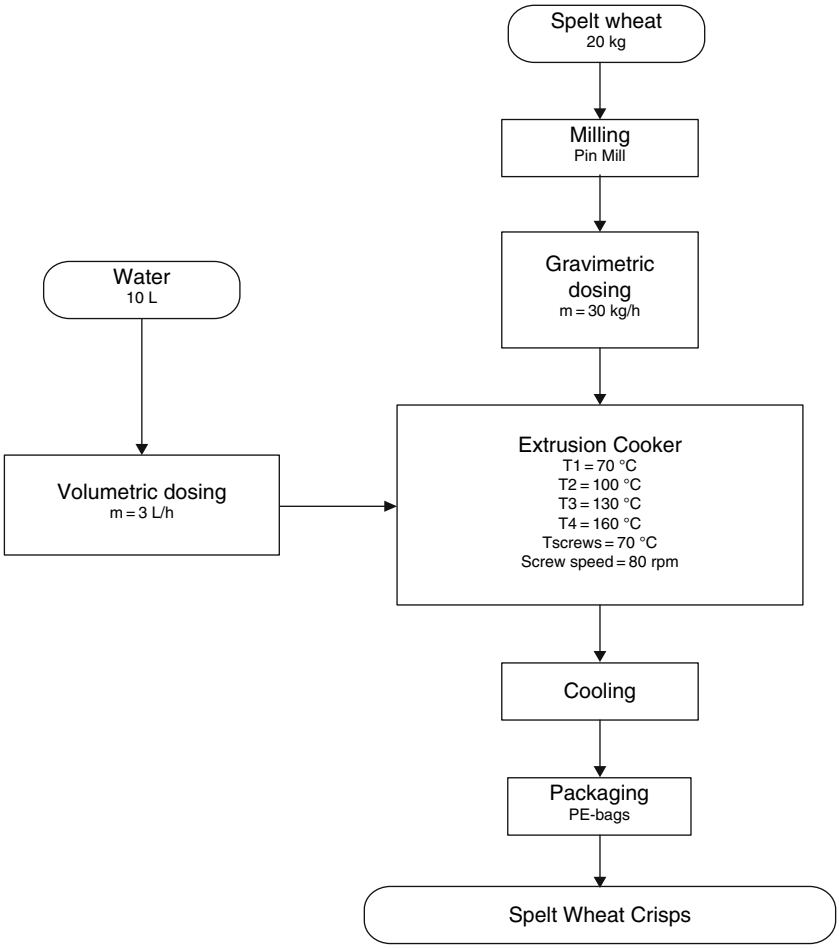

Figure 18.2. Flow chart of the extrusion process.

2. Why is the temperature in the first temperature zone lower than in the fourth ?
3. Which kinds of forces are occurring in the extruder ?
4. What can the number of peaks in texture measurement tell you about crispness ?
5. In Figure 18.3, what is the difference between (a) and (b) regarding crispness and maximum force ?
6. Calculate the bulk density and the expansion index of the obtained extruded material (Table A 18.1).
7. Which parameters are dependent on is the expansion of the extruded material dependant ?

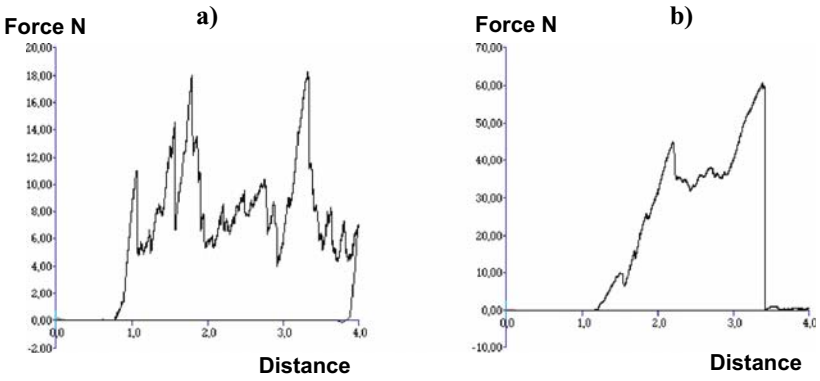

Figure 18.3. (**a**) Sample A and (**b**) Sample B.

Figure 18.4. Extrusion cooker (Pilot plant – University of Natural Resources and Applied Life Sciences).

8. Why is the temperature in the first temperature zone lower than in the fourth ?
9. Which kinds of forces are occurring in the extruder ?
10. What can the number of peaks in texture measurement tell you about crispness ?

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19

Semolina – Milling and Sieving

Maria Papageorgiou

OBJECTIVES AND LEARNING OUTCOMES

1. Evaluate the yield and quality parameters of durum wheat semolina.

19.1. INTRODUCTION

Milling tests performed on experimental equipment are useful for comparing different varieties and for obtaining comparison data among different samples of durum wheat.

Durum wheat mills have the task of freeing the wheat from additions and foreign seeds and preparing it, by dampening to the best condition for grinding. They then detach the protective hull so as to free the farinose kernel, after which reduction and cleaning are used to produced the finished product, semolina.

The production cycle of these operations is composed of three basic processes: (1) reception of cereal, sampling, cleaning and conditioning of (2) grinding of by-products and final products (3) sieving of semolina (Bizzari and Morelli, 1988).

Semolina that is produced in this way must fulfill certain requirements that can be placed into two groups according to how they affect: (a) the milling performance of the wheat, i.e., the proportion of semolina of the desired degree of refinement that can be extracted from the wheat (semolina milling quality), (b) the ability of the extracted semolina to give pasta with the required appearance, resistance to breakage and cooking tolerance (pasta quality) (Feillet and Dexter, 1996).

MARIA PAPAGEORGIOU • National Agricultural Research Foundation, Cereal Institute, Laboratory of Cereal Chemistry and Technology, tech.ci@nagref.gr

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The factors that are usually determined in assessing semolina quality are (a) those affecting dough development and some quality characteristics of pasta, i.e., granulation and particle size distribution, ash content and pigment content (b) those associated with cooking quality of pasta, i.e., rheological characteristics through the performance of mainly farinograph test of semolina dough for levels of absorption used in pasta processing (30–35% absorption levels), protein and gluten quality, experimental pasta making tests followed by assessment of pasta quality.

19.2. EXPERIMENTAL PROCEDURE

19.2.1. Conditioning the Wheat

- Cleaning

The grain must be carefully cleaned and all foreign grains (cereals other than wheat and fodder plants), stones and metallic particles are removed. The cleaning operations separate the impurities and foreign seeds by dimension, weight and form.

- Conditioning to obtain correct moisture level

Milling is carried out using 500 g of durum wheat. Weigh 500 g wheat and put it in a plastic bag. By either damping or drying the moisture capacity of the grains must be fixed at 16.5%. Add the estimated amount of water and shake the bag in order to distribute the water evenly in the bag. Seal the bag and leave it for about 24 h. The amount of water that is to be added is given by the following formula:

$$X = 16.5\,\mathrm{MS}/83.5 - A$$

where X is the amount of water we have to add to 100 g of wheat, MS is the dry matter % and A is the initial moisture content of wheat %.

19.2.2. Milling

Model: Laboratory Mill Chopin -Dubois CD2/Durum Wheat.

- The 500 g used in the first stage are poured through the spout into the feed-hopper at right. The spout, which is removable, has on the bottom edge a magnet for catching metallic particles, which is not removed during the cleaning process.

- *Break*. Start the grinding by turning the right-hand electric starting switch (clockwise) which simultaneously controls the distributor micromotor and the grinder and sieve motor. Grinding time varies with the size of the grains (weight of 1.000 g). This takes about 3–3½ min. After grinding 500 g wait approximately 1½ min so that the sieving is completed. Then empty the sieve.

Two main fractions are obtained:

– at the end, coarse and fine bran and
– coarse semolina in the central collecting pan.

- *Reduction*. Pour the semolina into the left feed-hopper of the reduction system (with a flour scoop). Start the motor by turning on the left-hand switch (clockwise) which controls the distributor-reduction roll and sieve unit. Reduction time varies, depending on the amount of semolina extracted during the break stage (3–5 min).

When the semolina has been reduced, wait approximately 1½ min for the sieving to be completed and for the sieve to empty itself.

Three fractions are obtained:

– at far end, medium coarse semolina,
– in the left collecting-pan, reduction flour and
– in the right collecting fine semolina.

19.2.3. Sieving

Model: Sieve Chopin SA

The fractions obtained from the mill are used in the following way:

- The reduction flour is ready and can be collected in a plastic bag.
- The other two fractions are mixed together and they are poured in the sieve.
- Turn on the button and start sieving. When the mixture is reduced the machine should be stopped.
- Three fractions are obtained in the three drawers of the sieve.
- Mix the one in the middle with the one at the right and pour the mixture to be sieved for one more time.
- When the sample is reduced, wait for about 1 min in order to collect as much sample as possible.
- Finally turn off the machine and collect the three fractions in the same bag where the flour was collected from the second stage.

19.3. DATA ANALYSIS

1. How much water would you need to add to 100 g of a wheat sample of initial moisture of 11%?
2. Calculate the semolina extraction rate.

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20

Semi-Hard Cheese – Cheese Making Technology

Victoria Ferragut and Toni Trujillo

OBJECTIVE AND LEARNING OUTCOMES

1. Produce a rennet coagulated cheese made from cow's milk for obtaining a semi-hard, pressed curd cheese.
2. Review the role of the ingredients used in cheese-making.
3. Understand the importance of each step of the process and its influence on the characteristics of the final product.

20.1. INTRODUCTION

Cheese making is the process of removing water, lactose and some minerals from milk to produce a concentrate of milk fat and protein. The essential ingredients for cheese are milk, rennet, starter cultures and salt. Each step of the process has a relevant importance to obtain a good quality and characteristic cheese.

20.1.1. Pasteurization

This preservation process is one of the major critical control points in the cheese making process. It destroys the pathogenic micro-organisms present in the raw milk. Pasteurization also contributes to the increase of yield by promoting the

VICTORIA FERRAGUT AND TONI TRUJILLO • Universitat Autònoma de Barcelona Centre Especial de Recerca Planta de Technologia dels Aliments (CERPTA), Department de Ciència Animal i dels Alimetns, Facultat de Veterinària

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association of whey proteins and casein in a favour of a higher water retention. The starter culture is added from cultures prepared from inoculates of bacteria, in this case mesophilic and thermophilic heterofermentative bacteria, which have the purposes to develop acidity and to promote ripening. Acid production by bacteria cultures is essential to aid in the expulsion of whey from the curd and largely determines the final cheese moisture, flavour and texture.

20.1.2. Protein Coagulation

During cheese production, the rennet (coagulating enzymes) is stirred into the milk. Under certain conditions (T, acidity) rennet hydrolyses casein causing the gel formation. $CaCl_2$ is also added to promote the gel formation, the curd.

20.1.3. Cutting

Proper cutting is important to both quality and yield. Both early cutting when the curd is fragile and late cutting when the curd is brittle cause losses of particles. The curd is ready to cut if it breaks cleanly. Curd size has a great influence on moisture retention.

20.1.4. Cooking

The cooking and stirring will cause an increase in the acidity. Therefore, the moisture and soluble compounds will be expelled.

20.1.5. Curd Washing

Part of the whey is removed and substituted by warm water. This step contributes to remove some lactose and controlling pH of the curd.

20.1.6. Drainage

Whey is removed and curds are allowed to settle.

20.1.7. Pre-pressing and Moulding

Contributes to pre-forming and remove whey from the curd.

20.1.8. Pressing

Pressing is increased gradually to avoid moisture trapping inside the cheese which would be undesirable.

20.1.9. Salting

This step contributes to inhibit the growth and activity of pathogenic micro-organisms; controls the activity of various enzymes in cheese; changes proteins which influence cheese texture and protein solubility; contributes to the initial rind formation of cheese and affects cheese flavour.

20.1.10. Ripening

The prepared curd is exposed to certain environmental conditions (T, RH) for several months to several years depending on the cheese type. The purpose is to take place glycolysis, proteolysis and lipolysis which produce the compounds responsible of texture and flavour of cheese.

Attention has to be drawn to the hazards to human health due to the potential presence of pathogenic bacteria from the raw milk used in some cheese production. As cheese making is a process usually open, hygienic conditions of installations must be extremely controlled and adequate personnel training performed. Recommendations have been given for safe production of cheese applying Hazard Analysis Critical Control Point principles.

Potential environmental problems of cheese making may result from the lack of a managing system for the whey, as the main by-product of the process, and in a lesser extent, from brine used in the salting step.

20.2. EQUIPMENT

20.2.1. Materials

- Raw cow's milk.
- Calf rennet (Renifor-15/E, Lamirsa, Spain) containing 780 mg chymosinl^{-1}.
- Starter containing *L. lactis* subs. *lactis*, *L. lactis* subs. *cremoris* and *S. thermophillus*.
- $CaCl_2$ (food quality grade, 35%w/v).
- Brine solution (19% NaCl, $\rho = 1.143$).
- 1 kg perforated plastic moulds.

- Cotton gauzes.
- Strainer.
- Test tubes.
- Glasses.
- pH-meter.
- Thermometer.
- Latex gloves.
- Lab coat.
- Lab cap.
- Wellington boots.

20.2.2. Apparatus

- Pasteurizer.
- Steam/water jacket tank with agitation system, wire knives (horizontal and vertical) and plate for drainage.
- Ripening chamber.

20.3. EXPERIMENTAL PROCEDURE

20.3.1. Main Raw Material: Milk

To assess the milk quality some parameters are commonly used.

1. pH: potentiometrical determination.
2. Microbial quality of raw and pasteurized milk assessment by enumerating total counts on PCA medium incubated for 48 h at 30̊C (IDF, 1991).
3. P Protein content (IDF, 1993).
4. Fat content (ISO, 1976).
5. Qualitative analysis of the activity of alcaline phosphatase in raw and pasteurized milk (IDF, 1987).

20.3.2. Process

1. x l of milk are used to process y cheeses.
2. Pasteurize cow milk at 72°C, 15 s in a plate and frame pasteurizer.
3. Transfer the milk to a tank and add (2% w/w) of starter.
4. Then add $CaCl_2$ (food quality grade 0.015% v/w).
5. Rennet (0.02% v/w; 520 mg/L active chymosin).

6. Setting (31°C, 30–45 min).
7. Cut the curd with the wire knives (horizontal and vertical) ..... (pea size).

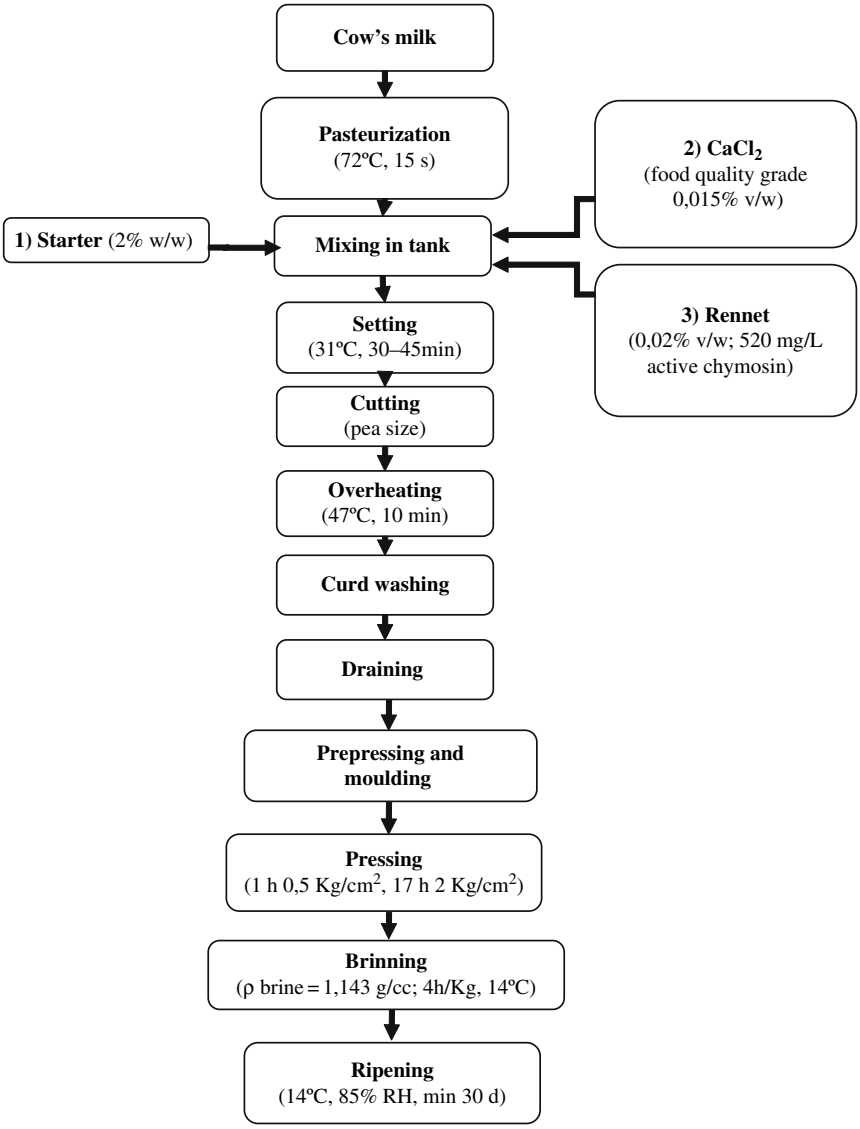

Figure 20.1. Flow chart of the process for cheese production.

20.3.3. Final Product: Cheese

The methods for cheeses analysis, consist in an overall quality control to assure established standard requisites for a specific type of cheese at its optimum time for consuming, in this case 30 days.

1) Total solids content (IDF, 1982).
2) Fat content (% on dry basis). Van Gulik method (ISO, 1975).
3) pH: potentiometrical determination on a slurry obtained by homogenizing 10 g of cheese and 10 ml of deionized water in a pH meter.
4) Sensory evaluation to asses the typical characteristics.

20.4. QUESTIONS

1. Indicate the steps of the process in which temperature control have a special relevance and justify the answer.
2. Indicate in which steps of the process it would be interesting to control pH for an optimum ripening evolution of cheese.
3. Why it is required to add $CaCl_2$ into the milk for the gel formation?
4. Try to find the critical control points for this process and your specific working conditions.
5. Why are chamber conditions of temperature and relative humidity relevant for the ripening process. Indicate what defects would be developed when RH is higher or lower than optimal conditions.

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Annexes

ANNEX 1. MIXING OF SOLID PARTICLES

Angle of repose of bulgur (after classification): 33°
Angle of repose of semolina (after classification): 37°
Hectolitre-weight of bulgur (after classification): 77 gmL^{-1}
Hectolitre-weight of semolina (after classification): 73 gmL^{-1}

Table A1.2. Data for cone type tumbling mixer

	Spot 1		Spot 2		Spot 3		Spot 4		Spot 5	
Time (min)	Bulgur	Semolina	Bulgur	Semolina	Bulgur	Semolina	Bulgur	Semolina	Bulgur	Semolina
2	6.2	18	18.4	8.2	3	19	4.1	17.5	2	24
4	7.3	19	9.3	16.1	12.6	14.8	6.8	15.3	3	22
6	9	17	13.4	10.9	13.5	12.9	14.1	10.1	5	20
8	7.2	10.9	9.8	11.9	11.8	14.9	8.6	17.6	5	11.5
10	13	12	14	14.5	11.9	12.9	17.5	18.6	8.9	9.6

I – 0.45A
V – 220 V

ANNEX 2. FILTERABILITY

Table A 2. Data obtained from experiments A and B

	C_o(FTU)	C(FTU)	Q(cm^3min^{-1})	H(mm)	t(s)	T(°C)
EXPERIMENT A	54	42	93	30	595	19
EXPERIMENT B	57	2.5	93	23	660	21

ANNEX 3. CAKE FILTRATION OF KIESELGUHR SUSPENSION ON PLATE-AND-FRAME FILTER

$\Delta P = 0.85$ bar
Cell radius = 4 cm

Table A3.1 Experimental data for filtration

	K-150		T-1000	
$V(m^3)$	t(s)	$t/V(s.m^{-6})$	t(s)	$t/V(s.m^{-6})$
$0.1\ 10^{-3}$	30	$0.30\ 10^6$	6	$0.60\ 10^5$
$0.2\ 10^{-3}$	65	$0.32\ 10^6$	15	$0.75\ 10^5$
$0.3\ 10^{-3}$	102	$0.34\ 10^6$	27	$0.90\ 10^5$
$0.4\ 10^{-3}$	145	$0.36\ 10^6$	41	$1.02\ 10^5$
$0.5\ 10^{-3}$	189	$0.38\ 10^6$	58	$1.16\ 10^5$
$0.6\ 10^{-3}$	235	$0.39\ 10^6$	77	$1.28\ 10^5$
$0.7\ 10^{-3}$	285	$0.41\ 10^6$	98	$1.40\ 10^5$
$0.8\ 10^{-3}$	336	$0.42\ 10^6$	123	$1.54\ 10^5$
$0.9\ 10^{-3}$	393	$0.44\ 10^6$	150	$1.67\ 10^5$

Average value of $r = 6.2\ 10^{10}\ m.kg^{-1}$

COFRAM-SEITZ FILTER

$S = 0.09\ m^2$

Table A3.2 Set of data for the pilot experiment

First stage		Second stage	
constant flow rate filtration $Q = 400\ L.h^{-1}$		constant pressure filtration $\Delta P = 1$ bar	
t(s)	ΔP (bar)	t(s)	$dV/dt\ (m^3.s^{-1})$
75	0.2	495	1.0
100	0.3	495	$1.10\ 10^{-4}$
166	0.4	508	$0.97\ 10^{-4}$
221	0.5	565	$0.92\ 10^{-4}$
280	0.6	720	$0.83\ 10^{-4}$
350	0.7	911	$0.78\ 10^{-4}$
392	0.8	1140	$0.75\ 10^{-4}$
439	0.9	1421	$0.69\ 10^{-4}$

First stage $r = 5.6\ 10^{10}\ m.kg^{-1}$
Second stage $r = 7.2\ 10^{10}\ m.kg^{-1}$

ANNEX 4. CENTRIFUGATION OF MILK

Table A4.1. Experimental data for milk separation

		Milk flow measurement		Milk		Cream Flow measurement		
Valve position	Milk T °C	V(mL)	t(s)	ρ(gmL^{-1})	%fat (w/w)	V(mL)	t(s)	Cream %fat(w/w)
1	51.4	1890	9.23	1.030	3.8	228	30.00	71
2	45.7	1990	7.20	1.032	3.6	760	24.26	35
3	38.5	2000	6.40	1.032	3.8	1560	33.78	30

Other data needed for calculations:
$\rho_{\text{milk fat}} = 9.15$ g.mL^{-1}
$\mu_{\text{milk}} = 1.42 \times 10^{-3}$ Pa.s
$\mu = 8110$ rpm
$R = 0.045$ m (location of the centre of the hole)
$r = 0.0045$ m (radius of the hole)
$V = 0.0032$ m^3 (volume of the centrifuge)

ANNEX 5. DIRECT OSMOTIC CONCENTRATION

Table A5.1. Detailed experimental conditions for comparative treatments (Dova et al., 2006b)

T Code No	Feed Liq.	molality	F_2 (m^3h^{-1})	T_f (°C)	Osm. medium NaCl μ_2(Pas)10^3	molality	F_1(m^3\h)	$T_{o.m.}$(°C)	μ_1(Pas) 10^3	ΔΠ (atm)	ΔP, (atm)	ΔP_{net}
1	Water		0.680	27.0	0.85	4.922	0.138	33.4	1.16	247.8	1.1	246.7
2			0.666	25.6	0.88	4.868	0.131	32.2	1.19	244.3	1.1	243.2
3			0.713	26.7	0.87	3.074	0.119	33.1	0.91	142.4	1.1	141.3
4			0.698	24.9	0.89	4.235	0.115	32.0	1.19	208.6	1.0	207.6
5	Sucrose	0.087	0.605	23.3	0.97	5.111	0.132	31.0	1.21	280.8	1.1	279.7
6		0.186	0.583	25.2	1.01	5.100	0.115	32.4	1.18	281.4	1.1	280.3
7		0.602	0.601	25.0	1.53	4.692	0.130	33.1	1.17	273.2	1.1	272.1
8		0.622	0.644	23.5	1.60	1.990	0.122	30.7	0.95	80.1	1.05	79.05
9	Glucose	0.351	0.637	23.0	1.10	4.863	0.112	30.3	1.23	263.2	1.05	262.15
10		0.311	0.644	23.3	1.07	1.939	0.120	29.7	0.97	85.1	1.05	84.05
11		1.021	0.673	19.8	1.60	1.929	0.119	26.6	1.03	69.3	1.05	68.25
12	Water		0.558	21.5	0.97	5.221	0.402	30.4	1.23	282.9	0.94	281.96
13			0.576	20.0	1.03	5.213	0.245	27.5	1.30	279.3	0.90	278.4
14			0.580	20.5	1.00	5.036	0.126	26.9	1.31	251.5	1.1	250.4
15	Glucose	0.276	0.792	19.7	1.15	5.192	0.131	26.7	1.32	287.7	0.67	287.03
16		0.281	0.526	19.6	1.16	4.942	0.130	27.1	1.31	268.8	0.90	267.9
17		0.281	0.227	19.5	1.16	5.079	0.131	29.9	1.21	279.5	0.77	278.73

Table A5.2. Measured flux values for experimental treatments (Dova et al., 2006b)

T Code #	Feed Liquid	molality	F_2 (m^3h^{-1})	T_f (°C)	μ_2 (Pa.s) 10^3	Osmotic medium	NaCl(molality)	F_1(m^3h^{-1})	$T_{o.m.}$ (°C)	μ_1(Pa.s) 10^3	ΔP_{net}	Flux, kgm^{-2} h^{-1}
1	Water		0.680	27.0	0.85	4.922		0.138	33.4	1.16	246.7	4.16
2			0.666	25.6	0.88	4.868		0.131	32.2	1.19	243.2	4.15
3			0.713	26.7	0.87	3.074		0.119	33.1	0.91	141.3	3.62
4			0.698	24.9	0.89	4.235		0.115	32.0	1.19	207.6	3.88
5	Sucrose	0.087	0.605	23.3	0.97	5.111		0.132	31.0	1.21	279.7	1.66
6		0.186	0.583	25.2	1.01	5.100		0.115	32.4	1.18	280.3	1.53
7		0.602	0.601	25.0	1.53	4.692		0.130	33.1	1.17	272.1	0.55
8		0.622	0.644	23.5	1.60	1.990		0.122	30.7	0.95	79.05	0.41
9	Glucose	0.351	0.637	23.0	1.10	4.863		0.112	30.3	1.23	262.15	1.27
10		0.311	0.644	23.3	1.07	1.939		0.120	29.7	0.97	84.05	0.69
11		1.021	0.673	19.8	1.60	1.929		0.119	26.6	1.03	68.25	0.07
12	Water		0.558	21.5	0.97	0.97		0.402	30.4	1.23	281.96	2.22
13			0.576	20.0	1.03	1.03		0.245	27.5	1.30	278.4	2.84
14			0.580	20.5	1.00	1.00		0.126	26.9	1.31	250.4	4.30
15	Glucose	0.276	0.792	19.7	1.15	5.192		0.131	26.7	1.32	287.03	1.20
16		0.281	0.526	19.6	1.16	4.942		0.130	27.1	1.31	267.9	1.35
17		0.281	0.227	19.5	1.16	5.079		0.131	29.9	1.21	278.73	1.53

ANNEX 6. VACUUM IMPREGNATION

Table A6.1. Vacuum impregnation parameters for some fruits (Salvatori, 1997)

	ρ_a	ρ_r	X	γ	ε_e
Apple G. Smith	802	1052	19	–0.6	21
Apple R. Chief	830	1059	17.9	–2.4	20.3
Apple Golden	787	1055	11.2	–6	17.4
Mango Tommy Atkins	1130	1130	14.2	8.9	5.9
Strawberry Chandler	984	1050	1.9	–4	6.4
Kiwi Hayward	1051	1076	1.09	0.8	0.7
Peach Miraflores	1038	1065	6.5	2.1	4.7
Peach Catherine	987	1070	4.4	–4.2	9.1
Pineapple Española Roja	1030	1051	5.7	2.3	3.7
Pear Passa Crassana	1030	1070	5.3	2.2	3.4

Table A6.2. Results from experimental work

	x_w	°Brix	Mass gained %
Apple G. Smith	0.86	0.104	19.0
Apple R. Chief	0.86	0.130	17.9
Apple Golden	0.84	0.153	11.2
Mango	0.79	0.178	14.2
Strawberry	0.91	0.072	0.2
Kiwi	0.82	0.143	0.89
Peach Miraf.	0.82	0.150	6.5
Peach Cath.	0.88	0.119	4.4
Pineapple	0.89	0.897	5.7
Pear	0.80	0.160	5.3

ANNEX 7 EXTRACTION OF PUMPKIN OIL

Table A7.1. The influence of pumpkin cultivar and screw rotation speed on oil and press cake yield (an example)

	Pumpkin Seeds cv. Yield					
	Miranda		*Golosemianaja*		*Herakles*	
Screw Rotation Speed (rpm)	Oil (ml)	Press cake (g)	Oil (ml)	Press cake (g)	Oil (ml)	Press cake (g)
10	40	60	45	55	50	60
20	39	57	48	51	54	57
30	33	62	43	57	48	62

ANNEX 8 TRAY DRYING OF APPLES

Table A8.1. Drying data for apples*

Drying time (sec)	Product weight (Kg)	$T_{Drybulb}$ (°C)	$T_{Wetbulb}$ (°C)	Drying time (sec)	Product weight (Kg)	$T_{Drybulb}$ (°C)	$T_{Wetbulb}$ (°C)	Drying time (sec)	Product weight (Kg)	$T_{Drybulb}$ (°C)	$T_{Wetbulb}$ (°C)
0	0.094	40	36	960	0.077			3720	0.037		
30	0.094			1080	0.075	47	36	3840	0.036		
60	0.093			1200	0.072			3960	0.034	49	36
90	0.093			1320	0.07			4080	0.033		
120	0.092			1440	0.068	48	36	4200	0.032		
150	0.092			1560	0.066			4320	0.031	49	36
180	0.091	43	35	1680	0.064			4440	0.03		
210	0.091			1800	0.062	48	36	4560	0.029		
240	0.090			1920	0.06			4680	0.028	49	36
270	0.090			2040	0.059			4800	0.027		
300	0.089			2160	0.057	48	36	4920	0.026		
330	0.088			2280	0.055			5040	0.025	49	36
360	0.088	45	35	2400	0.053			5160	0.025		
390	0.087			2520	0.051	48	36	5280	0.024		
420	0.087			2640	0.05			5400	0.023	49	36
450	0.086			2760	0.048			5520	0.023		
480	0.085			2880	0.047	49	36	5640	0.022		
510	0.085			3000	0.045			5760	0.022	49	36
540	0.084	46	35	3120	0.044			5880	0.021		
570	0.084			3240	0.042	49	36	6000	0.021		
600	0.083			3360	0.041			6120	0.02	49	36
720	0.081	47	35	3480	0.04			6240	0.02		
840	0.079			3600	0.038	49	36	6360	0.02		

*- Operation conditions: average air speed = 1,47 ms^{-1}

ANNEX 9 MASS BALANCES ON A COMBINED AIR-MICROWAVE PROCESS

Drying temperature = 40 °C x_w (g water total mass^{-1}) = 0.87
P_{sat} (T_2) = 4.3993 atm Dry mass (g) = 2.86

Table A 9.1. Saturation pressure and density of water vapor for a common temperature range

Temperature (°C)	Saturation Pressure (Pa)	Density (kg/m^3)
0	603	0.005
10	1212	0.009
20	2310	0.017
30	4195	0.030
40	7297	0.051
50	12210	0.083
60	19724	0.13
70	30866	0.20
80	46925	0.29
90	69485	0.42
100	100446	0.59

Table A 9.2. Time-temperatures profile during the drying process of apple slices in an air microwave drier

Time (s)	Mass (g)	Air velocity (m s^1)	Room temperature (°C)	RH_1 (%)	Time (s)	Mass (g)	Air velocity (m s^1)	Room temperature (°C)	RH_1 (%)
30	21.78	5.08	17.58	36.34	870	19.6330	5.03	17.81	36.65
60	21.69	5.06	17.51	36.43	900	19.5900	5.07	17.78	35.31
90	21.61	5.06	17.67	36.3	930	19.5180	5.05	17.69	36.46
120	21.53	5.03	17.49	36.85	960	19.4320	5.05	17.58	36.41
150	21.45	5.04	17.47	37.05	990	19.3850	5.07	17.59	36.66
180	21.35	5.05	17.58	36.79	1020	19.32	5.02	17.55	36.59
210	21.29	5.05	17.49	36.49	1050	19.24	5	17.56	36.85
240	21.21	5.14	17.64	37.03	1080	19.18	5.08	17.59	36.5
270	21.13	5.03	17.53	36.27	1110	19.11	5.03	17.59	35.37
300	21.04	5.03	17.59	36.1	1140	19.05	5.02	17.58	35.71
330	20.97	5.07	17.68	36.01	1170	18.99	5.04	17.62	35.77
360	20.89	5.08	17.57	36.74	1200	18.93	5.03	17.56	36.1
390	20.82	5.04	17.55	36.51	1230	18.80	5.01	17.59	36.48
420	20.73	5.08	17.6	36.46	1260	18.80	5.02	17.58	35.92
450	20.67	5.05	17.57	36.13	1290	18.72	5.03	17.91	35.29
480	20.59	5.02	17.56	36.49	1320	18.67	5.03	17.6	35.81
510	20.50	5.02	17.45	36.95	1350	18.59	5.05	17.63	36.08
540	20.45	5.05	17.54	36.42	1380	18.55	4.95	17.55	35.6
570	20.370	5.04	17.6	36.44	1410	18.48	5	17.7	35.44
600	20.30	5	17.76	34.96	1440	18.42	5.04	17.61	35.96
630	20.22	4.98	17.55	36.3	1470	18.36	4.99	17.68	35.45
660	20.15	5.03	17.48	36.53	1500	18.31	5.03	17.59	35.43
690	20.08	5.05	17.46	36.54	1530	18.250	4.98	17.61	35.48
720	20.01	5.05	17.52	36.36	1560	18.18	5.08	17.66	35.35
750	19.94	5.04	17.87	35.24	1590	18.14	5.03	17.62	35.47
780	19.8590	5.07	17.73	35.44	1620	18.07	4.92	17.6	36.47
810	19.7810	5.04	17.59	36	1650	18.00	4.99	17.58	35.43
840	19.7230	5.04	17.58	35.82	1680	17.95	5.06	17.59	35.53
1710	17.89	5.05	17.79	34.72	1980	17.3980	5.01	17.61	35.48
1740	17.84	5.06	17.58	34.93	2010	17.3330	5.08	17.62	35.89
1770	17.79	5.03	17.58	35.18	2040	17.2890	5.09	17.64	35.82
1800	17.7220	5.04	17.61	35.4	1890	17.5680	5.04	17.61	35.3
1830	17.6750	4.98	17.61	35.55	1920	17.5090	5	17.6	35.31
1860	17.6080	5.07	17.62	35.4	1950	17.4500	5.02	17.61	36.2
1890	17.5680	5.04	17.61	35.3	1980	17.3980	5.01	17.61	35.48
1920	17.5090	5	17.6	35.31	2010	17.3330	5.08	17.62	35.89
1950	17.4500	5.02	17.61	36.2	2040	17.2890	5.09	17.64	35.82

ANNEX 11 EXPERIMENTAL DATA FOR FLUID BED DRYING

Table A11.1. Relationship between drying rate & time

Drying rate (kg water $m^{-2}h^{-1}$)	Time (min)
10	0
8	10
5	20
3.5	30
3	40
1.5	50

*Conditions: 50°C and 0.9 ms^{-1} air velocity

ANNEX 13 FREEZING – I. DETERMINING FREEZING TIMES OF FOOD WITH A PLATE FREEZER

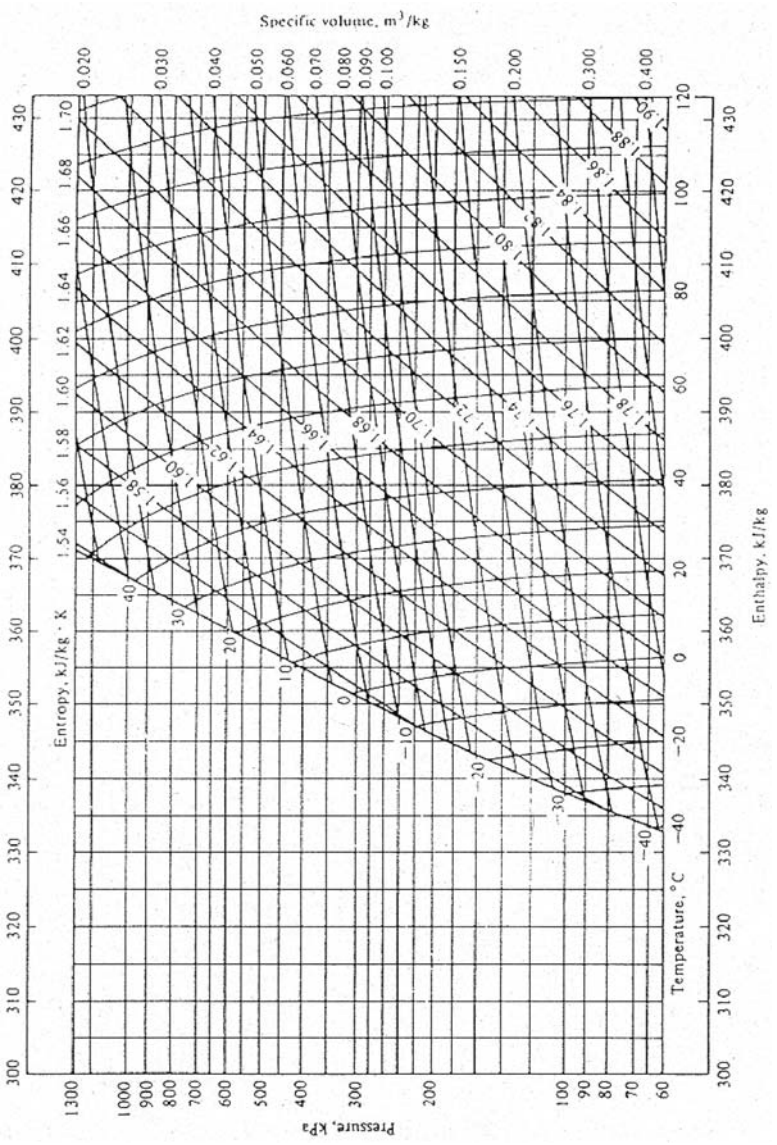

Figure A13. Pressure-enthalpy diagram of superheated R-12.

Table A13.1 Data for the determination of the fraction water content of potato and carrot purees

	M_1(G)	M_2(G)	M_3(G)
POTATO	55.02	69.46	55.18
CARROT	54.35	68.13	55.47

M_1 – Petri box weight; M_2 – Petri box weight + potato/carrot puree; M_3 – Petri box weight + potato/carrot after 48 hours in the stove

Table A13.2 Time-temperatures profile during the frozen process of potato puree

Time (min)	$T_{\text{bottom surface}}$ (°C)	T_{centre} (°C)	$T_{\text{top surface}}$ (°C)	Time (min)	$T_{\text{bottom surface}}$ (°C)	T_{centre} (°C)	$T_{\text{top surface}}$ (°C)
0	21	23	17.6	35	–11.8	4.6	–30.2
1	–5.6	22.8	–10.2	36	–12	4.4	–28.2
2	–4.4	22	–9	37	–12.6	3.8	–26.4
3	–3.6	21.6	–10	38	–12.8	3.6	–24.8
4	–4.4	20.6	–18.2	39	–13.4	3.2	–33.6
5	–4.8	19.8	–16.2	40	–13.4	3.2	–32.8
6	–5	18.8	–14.6	41	–13	2.8	–28.8
7	–5.8	18	–20.4	42	–13.4	2.6	–27.2
8	–6.4	17.4	–26.4	43	–13.6	2.2	–25
9	–6.8	16.6	–25	44	–14	2	–24.2
10	–7.2	16	–21.8	45	–14.4	1.8	–32
11	–7.4	15.4	–19.8	46	–14.4	1.6	–31.2
12	–7.6	14.8	–27	47	–14	1.2	–28
13	–8.2	14	–28.6	48	–14.4	1.2	–26.4
14	–8.2	13.6	–26.6	49	–14.6	0.8	–24.8
15	–8.4	13	–24	50	–14.8	0.8	–23.8
16	–8.6	12.6	–21.6	51	–15.2	0.6	–23
17	–9	12	–30.2	52	–15.4	0.6	–23.4
18	–9.4	11	–30.4	53	–15.2	0.2	–23
19	–9.4	10.8	–26.6	54	–15.4	0.2	–22.4
20	–9.4	10	–24	55	–15.4	0	–21.6
21	–10	9.8	–22.2	56	–15.6	–0.2	–26.2
22	–10	9.6	–29.2	57	–15.8	–0.2	–27
23	–10.2	9	–31.8	58	–15.6	–0.2	–25.6
24	–9.6	8.6	–28	59	–15.6	–0.6	–24.2
25	–10	8.4	–25	60	–15.8	–0.6	–23.4
26	–10.2	7.8	–23.4	61	–15.2	–0.6	–23
27	–10.4	7.4	–30.2	62	–16.6	–0.8	–26.6
28	–10.8	7	–35	63	–17.8	–0.6	–25.6
29	–10.4	6.6	–32.6	64	–16.6	–0.8	–24.8
30	–10.4	6.4	–29.2	65	–16.4	–0.8	–23.8

Table A13.2 (continued)

Time (min)	$T_{bottom\ surface}$ (°C)	T_{centre} (°C)	$T_{top\ surface}$ (°C)	Time (min)	$T_{bottom\ surface}$ (°C)	T_{centre} (°C)	$T_{top\ surface}$ (°C)
31	–11	5.8	–27.2	66	–16.6	–0.8	–22.6
32	–11.2	5.6	–25.8	67	–17	–1	–22.6
33	–11.8	5	–34.4	68	–16.6	–1	–22.6
34	–12	4.8	–35	69	–16.6	–0.8	–22.4
70	–17	–0.8	–21.6	97	–18.8	–3.6	–29
71	–17	–1	–21.2	102	–19.6	–4	–28.6
72	–17.4	–1	–20.8	109	–19.8	–4.6	–28.2
73	–17.8	–1.2	–21.2	112	–20	–4.8	–27.4
74	–17.4	–1.2	–21.4	117	–20.6	–5.4	–29
75	–17.4	–1.2	–21.4	122	–20.8	–5.6	–28.6
76	–17.4	–1.6	–20.8	127	–22.2	–5.8	–29
77	–17.4	–1.6	–20.4	132	–21.8	–6.4	–30.4
78	–17.8	–1.6	–20.2	137	–23	–7.2	–30.6
80	–18	–1.8	–20.4	142	–23.4	–7.8	–29.8
82	–17.8	–2	–24.2	147	–24.6	–9	–29
87	–18	–2.6	–28.6	152	–24.8	–10.8	–30.2
92	–18.4	–3	–29.2				

Table A.13.3. Temperatures in the carrot puree

Time(min)	$T_{bottom\ surface}$ (°C)	T_{centre} (°C)	$T_{top\ surface}$ (°C)
0	14.6	13.8	14
1	–8.2	13	6.6
2	–5.6	12.4	6.4
3	–4.4	11.6	6.4
4	–4.8	10.8	6
5	–5	10	5.8
6	–5	9.4	5.4
7	–5.6	8.8	5.4
8	–6	8.6	5
9	–5.8	7.8	4.6
10	–5.6	7.6	4.6
11	–5.8	7.4	4
12	–6.4	6.8	3.8
13	–6.6	6.6	3.6
14	–6.6	6	2.8
15	–6.4	6	2.6
16	–6.8	5.8	2.2

Table A.13.3. (continued)

Time(min)	$T_{bottom\ surface}$ (°C)	T_{centre} (°C)	$T_{top\ surface}$ (°C)
17	–7.2	5.4	2
18	–7.4	5	1.2
19	–7.2	4.6	0.8
20	–7.4	4.6	0.8
21	–7.4	4.4	0.6
22	–7.8	3.8	0.6
23	–8.2	3.8	0
24	–7.8	3.6	–0.6
25	–7.6	3.2	–0.6
26	–7.8	3.2	–0.8
27	–8.2	3	–1
28	–8.4	2.8	–1
29	–8.2	2.8	–1.8
30	–8.2	2.6	–1.8
31	–8.2	2.2	–2
32	–8.4	2	–2
33	–8.6	2	–2.2
34	–8.6	1.8	–2.8
35	–8.4	1.6	–2.8
36	–8.4	1.6	–3
37	–8.6	1.2	–3.2
38	–9	1.2	–3.2
39	–9.2	1	–3.6
40	–9	1	–4
41	–8.6	0.8	–4
42	–9	0.6	–4
43	–9.2	0.6	–4.4
44	–9.2	0.6	–4.6
45	–9.2	0.6	–4.6
46	–9.4	0.2	–4.6
47	–9.2	0	–5
48	–9.4	0	–5
49	–9.4	0	–5
50	–9.6	0	–5.4
51	–10	–0.2	–5.4
52	–10	–0.2	–5.4
53	–9.6	–0.6	–5.6
54	–9.6	–0.6	–5.8
55	–10	–0.6	–5.6
56	–10.2	–0.6	–5.8
57	–10.4	–0.6	–5.8
58	–10	–0.6	–6
59	–10	–0.8	–6
60	–10.2	–0.8	–6.4

Table A.13.3. (continued)

Time(min)	$T_{bottom\ surface}$ (°C)	T_{centre} (°C)	$T_{top\ surface}$ (°C)
61	–10.4	–0.8	–6.4
62	–10.8	–0.8	–6.4
63	–10.4	–0.8	–6.4
64	–10.4	–1	–6.4
65	–10.4	–0.8	–6.4
66	–10.8	–0.8	–6.4
67	–10.8	–1	–6.4
68	–11	–0.8	–6.4
69	–11	–1	–6.6
70	–11	–1	–6.6
71	–11.2	–1	–6.4
72	–11.2	–1.2	–6.6
73	–11.4	–1.2	–6.6
74	–11.2	–1.2	–6.6
75	–11.4	–1.6	–6.6
76	–11.4	–1.6	–6.6
77	–11.4	–1.6	–6.6
78	–11.8	–1.6	–6.6
80	–12	–1.8	–6.6
82	–11.8	–2	–6.6
87	–12	–2.6	–7.2
92	–12.8	–3	–7.6
97	–13	–3.6	–7.8
102	–13.4	–4	–8.2
109	–13.8	–4.8	–8.4
112	–14	–5	–8.4
117	–14.6	–5.8	–8.6
122	–15.2	–6	–8.4
127	–15.4	–6.6	–9
132	–15.8	–7.2	–9.4
137	–16.6	–8.2	–9.6
142	–17.4	–9.2	–10
147	–18.4	–10.4	–10.8
152	–19.6	–11.8	–11.4

Water content on commercial milk powder: 5.57%
Water content on milk powder obtained after the process: 4.06%

ANNEX 14 FREEZING – II. COMPARING AIR BLAST AND FLUIDIZED BED FREEZING

Table A14.1. Freezing data for meat balls

Time	T_{Env}	$T_{meat\ ball}$	time	T_{Env}	$T_{meat\ ball}$
0	–18,9	15,7	16	–25,5	–14
1	–23,9	10,7	19	–23,4	–17,6
2	–25,8	6,7	24	–26,7	–22,8
3	–23,9	3,4	25	–23,5	–23,1
4	–20,8	0,8	26	–21,3	–22,8
5	–25	–2,1	27	–24,9	–23,1
6	–25,4	–4,1	28	–26,2	–23,8
7	–21,8	–5	29	–23	–23,7
8	–23,9	–5,9	30	–21	–23,1
9	–26,2	–7	31	–25,5	–23,5
10	–23,8	–7,4	32	–25,4	–24
11	–21,1	–7,6	33	–22,7	–23,8
12	–25	–8,9	34	–21	–23,1
13	–26,3	–10,2	35	–25,7	–23,5
14	–22,9	–11,3	36	–25,3	–24
15	–20,9	–12,2	37	–22,5	–23,8

Table A14.2. Freezing data for meat balls

Sausage					
Time	T_{env}	$T_{center\ sausage}$	time	T_{env}	$T_{center\ sausage}$
	–23	23,1	19	–23,4	–3,6
0	–18,9	23,1	20	–26,1	–4,8
1	–23,9	22,6	21	–24,3	–6,6
2	–25,8	20,5	22	–21,6	–9
3	–23,9	17,6	23	–24,3	–12,2
4	–20,8	14,3	24	–26,7	–15,3
5	–25	11,2	25	–23,5	–17,9
6	–25,4	8,4	26	–21,3	–19,6
7	–21,8	5,9	27	–24,9	–20,4
8	–23,9	3,8	28	–26,2	–21,5
9	–26,2	2,1	29	–23	–22,6
10	–23,8	0,7	30	–21	–22,9
11	–21,1	–0,4	31	–25,5	–22,8
12	–25	–1,1	32	–25,4	–23,5
13	–26,3	–1,6	33	–22,7	–24
14	–22,9	–1,9	34	–21	–23,8
15	–20,9	–1,9	35	–25,7	–23,4
16	–25,5	–2	36	–25,3	–24,1
17	–25	–2,1	37	–22,5	–24,4
18	–22,1	–2,7			

Table A14.3. Freezing data for fish fillets

Fish Filets					
Time	$T_{env.}$	$T_{fish\ filet}$	time	$T_{env.}$	$T_{fish\ filet}$
	–23	24,5	19	–23,4	–22,6
0	–18,9	12,3	20	–26,1	–23,8
1	–23,9	1,2	21	–24,3	–24,5
2	–25,8	–0,8	22	–21,6	–23,4
3	–23,9	–1,5	23	–24,3	–22,7
4	–20,8	–5,4	24	–26,7	–24,2
5	–25	–14,8	25	–23,5	–24,4
6	–25,4	–21,1	26	–21,3	–23,1
7	–21,8	–22,5	27	–24,9	–22,9
8	–23,9	–22,3	28	–26,2	–24,3
9	–26,2	–23,6	29	–23	–24,2
10	–23,8	–24,3	30	–21	–22,8
11	–21,1	–23	31	–25,5	–23,1
12	–25	–23	32	–25,4	–24,5
13	–26,3	–24,3	33	–22,7	–24,1
14	–22,9	–24,2	34	–21	–22,7
15	–20,9	–22,7	35	–25,7	–23,2
16	–25,5	–23,3	36	–25,3	–24,5
17	–25	–23,7	37	–22,5	–24
18	–22,1	–24,4			

ANNEX 15. HEAT PROCESSING – I. PASTEURIZATION WITH A PLATE HEAT EXCHANGER

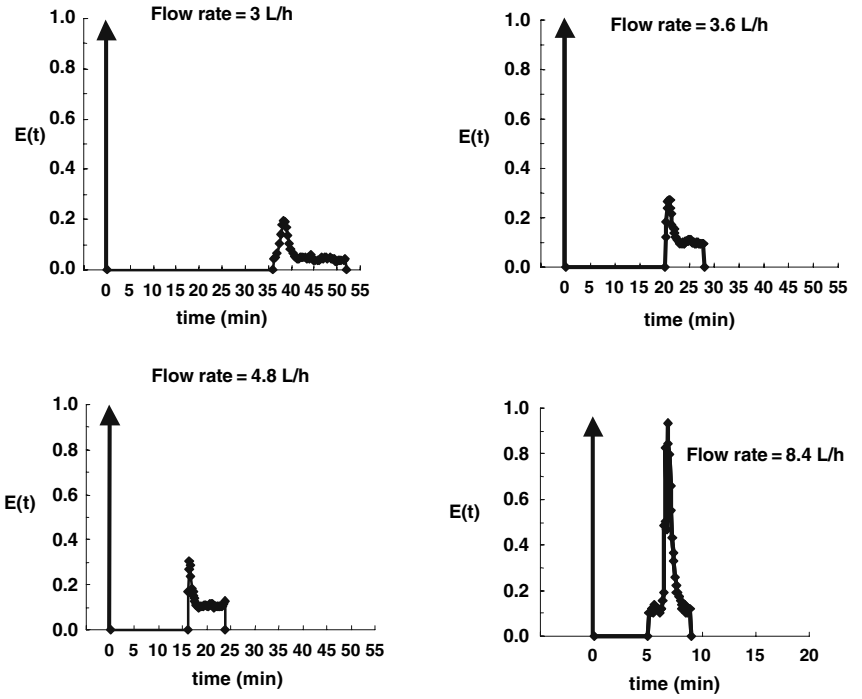

Figure A15.1. Residence Time Distribution (RTD) of fruit juice flowing through the system and detected at the exit of the system (the arrow represents tracer injection).

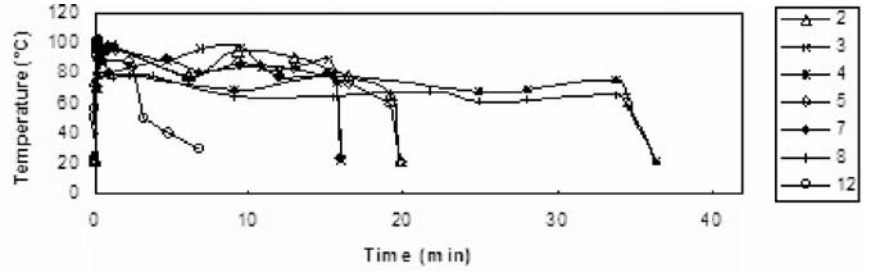

Figure A15.2. Time–temperature history.

Table A15.1A. Calibration line

	concentration [g/l]	peak area
Dilution 1	0.0050	0.161177
Dilution 2	0.0100	0.357121
Dilution 3	0.0200	0.742209
Dilution 4	0.0399	1.546308
Dilution 5	0.0599	2.515498
Dilution 6	0.0799	3.317897

Table A15.2. Experimental Data

Run #	Max temp. reached(°C)	Flow rate ($l\ h^{-1}$)	Dwell time in the holding tube [1]	Peak area	$\left(\frac{C}{C_0}\right)_{exp}$
2	98	3.6		0.0070	
3	98	4.8		0.0068	
4	80	3.0		0.0065	
5	90	3.6		0.0078	
7	85	4.8		0.0073	
8	70	3.0		0.0074	
12	90	8.4		0.0088	

[1] Approximate calculation according to Equation 15.10.

Table A 15.3. Results from pasteurization

	Flow rate ($l\ h^{-1}$)	Dwell time in the holding tube [1]	Peak area	Concentration ($g\ l^{-1}$)
Sample 11	3		0.0088	
Sample 12	3		0.0900	
Sample 21	8.4		0.0149	
Sample 22	8.4		0.0145	

[1] Approximate calculation according to Equation 15.10.

ANNEX 16. HEAT PROCESSING – II. STERILIZATION IN CANS

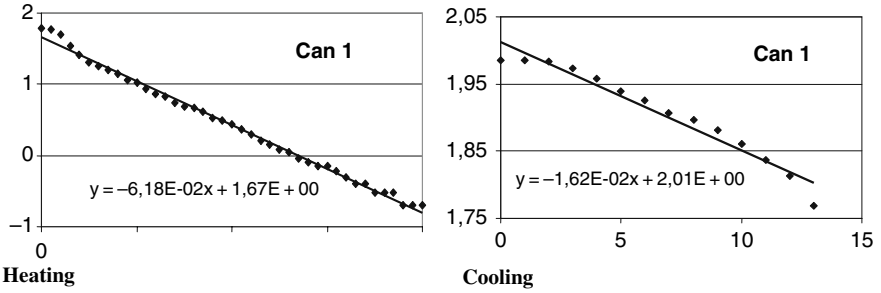

Figure A16.1. Plots of Decimal logarithm of the difference between the plateau and the can temperature as a function of time for heating and cooling.

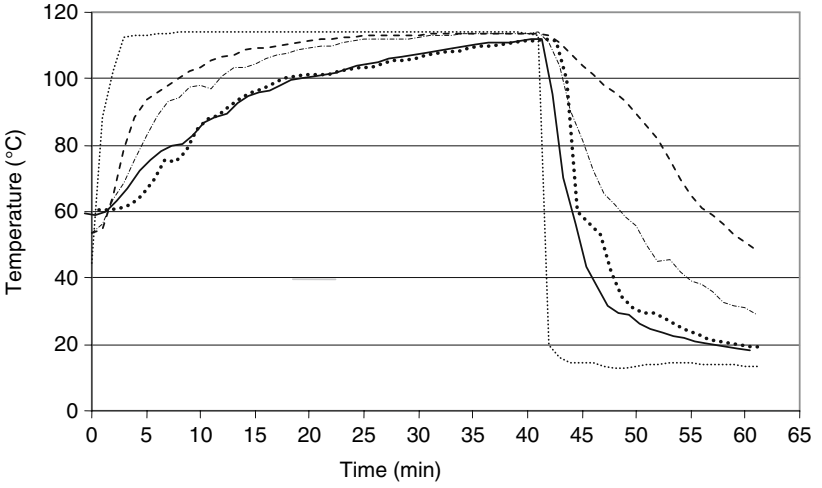

Figure A16.2. Time-temperature profile of the 4 cans during sterilization of tomato sauce. ●●●●●Retort temperature,——, Can 1.,●—● , Can 2●●●●●● Can3 — Can4.

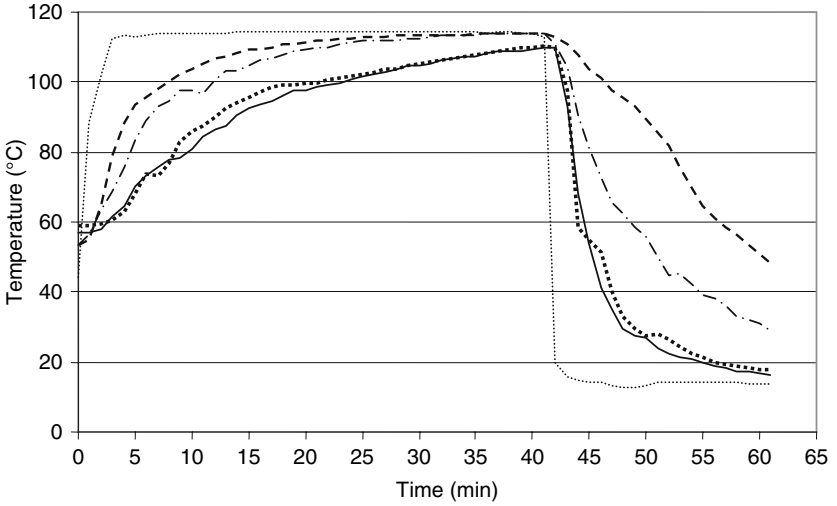

Figure A16.3. Time-temperature profile of the 4 cans during sterilization of tomato sauce with pasta. . ●●●●●Retort temperature,——, Can 1.,●—● , Can 2●●●●●● Can 3 and — Can 4.

ANNEX 17- INGREDIENTS IN INFANT FOODS – PREGELATINIZED AMARANTH FLOUR USING A DRUM DRYER

Table A17.1. Results of the drum drying trials

PARAMETER									
G kg h^{-1}	Z S	$_b$ %	$_e$ %	R m	I m	φ °	R kJ/kg	C_P kJ kg^{-1}	t_b°C
19,5	3600	88,3	2,2	0,25	0,5	300	2260	16981	21,3

ANNEX 18 WHEAT CRISPS – EXTRUSION COOKING TECHNOLOGY

Table A 18.1. Diameter and weight (in 5 l) of 10 measurements

Sample	diameter (mm)	weight (g)
1	4,37	745
2	3,92	796
3	4,75	756
4	3,97	768
5	4,44	799
6	3,97	784
7	4,05	793
8	3,78	775
9	4,49	771
10	4,23	782

ANNEX 19 SEMOLINA – MILLING AND SIEVING

Table A19.1. Semolina production No: 1

Wheat reference:		Melissa
Moisture content before tempering:		11.06%
Moisture before milling:		16.5%
Quantity milled:		531 g
Tempering time:		20 h
	g	%
MILLING		
Break		
B1.Overtails of sieve no 25	102.8	19.4
B2.Coarse semolina	426.5	80.3
Reduction		
R1.Medium coarse semolina	349.0	65.7
R2.Fine semolina	47.5	8.9
R3.Flour	24.0	4.5
Sieving		
S1.Coarse semolina	66.0	12.4
S2. Medium	66.5	12.5
S3. Fine semolina	120	22.6
Semolina Extraction		
R3 + S1 + S2 + S3	276,5	52

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