Chapter 6

Processes Calculations and Their Applications

The preservation of foods is a basic mechanism for survival by most cultures because Earth's seasonal variations can prevent continuous supplies of particular crops in many regions, and food supplies that are constantly available either for consumption or trade have always been a critical aspect of day-to-day life. Historically, in more environmentally severe areas, it has been critical to store the results of a short growing season for use throughout the year as well as drying or preserving meat and fish that may have an undependable supply.

Traditional methods such as drying, pickling, fermenting, and salting are still used for many products, but with the advent of more modern processing methods in the early nineteenth century, as well as the subsequent understanding of the microbial processes involved, thermally processed foods heated in durable, hermetically sealed containers found an expanding market. Relatively inexpensive refrigeration systems and temperature-controlled supply chains that began with naturally occurring ice and expanded with the development of mechanical refrigeration allowed the development and distribution of frozen and chilled foods as well as wide distribution of fresh produce, which led, in turn, to the development of specialized crop-growing regions such as the vegetable farms and citrus groves of the American West.

Because of the simplicity, reliability, and ubiquity of thermal preservation either by heat or cold, so-called non-thermal preservation methods that use alternate forms of energy input to achieve industrial sterility have been under investigation for more than a century, but few have gained a significant market share. Nonetheless, they offer particular benefits for niche markets that may include flavor, texture, and nutrient retention.

Thermal Processing

Foods have been thermally processed since prehistory, with sun drying and cooking over open fires common wherever possible. Because of the long history of these terms (and their variability among various cuisines), food processing is often filled with simple and inelegant terminology. For the purposes of this book, thermal processing will refer to commercial processing, for the purposes of enzymatic or biological action reduction or inactivation in the product. Thermal processing in general can be broken down into a progression of general categories as follows:

Cooking

Cooking is traditionally considered to be the exposure of the food material to a temperature of at least that of the standard boiling point of water (100°C). It may include an entire cookbook's

Food and Package Engineering, First Edition. Scott A. Morris.

worth of terminology, depending on the method of heat application and the media in which the food product is cooked. For instance, heating food in boiling water may be stewing or poaching whereas heating the same food in hot oil might be sautéing or frying, and heating it in air might be baking.

Blanching

Blanching is done to reduce or destroy naturally occurring enzymes that can degrade food products, most often fresh fruits or vegetables or their juices. The process may be done with indirect heat, boiling water, or applied steam depending on the requirements of the food product and the scale of the operation. Blanching is almost always included as a step in the canning of fruits and vegetables to avoid color and quality degradation.

Pasteurization

Pasteurization is a process that will reduce the microbiological population of a food to a low level, and can incorporate blanching processes. While this can be done with several types of basic mechanisms, it usually involves an exposure of the product to a heating process. Pasteurization is typically used by itself for food products that are either consumed quickly or are kept in a temperature-controlled environment that further reduces the growth of microorganisms with off-odor and flavor development as an indicator of spoilage before significant levels of pathogen growth can occur. One of the most common processes is the thermal pasteurization of milk that is used to reduce or eliminate the incidence of *M. tuberculosis* and *C. burnetti*, which are responsible for tuberculosis and Q fever, respectively. Although the milk may be heated to reduce the number of pathogens to a minimal level, the continued refrigeration of the product is an essential part of maintaining an acceptably low level of contamination. Pasteurization may also be applied to products such as fruit juices and beer to remove spoilage organisms and reduce pathogen risk.

Sterilization

Sterilization is a process that will render all spoilage organisms – bacterial, yeast, or fungus – completely inactive. Because the energy input required for this is often either impractical or destructive to the product, the term *commercial sterility* is used to denote that no viable organisms can be detected with standard methods, or that the number of survivors is insignificant under usual conditions of canning and storage, or that the acidity (Ph), oxidation-reduction potential (Eh), or temperature will restrict growth [1].

Although the historical methods of food pasteurization and sterilization are most often thermally based, other types of energy input have been successfully exploited for nearly a century, ranging from simple electrical discharge to complex irradiation technologies and physical means such as ultrasound and ultra-high pressure.

Calculation of Thermal Process Times

The condition under which foods must be thermally processed to reduce or effectively eliminate the necessary number of microbes is a function of:

– The product type: pH, salt or sugar content, preservatives, water availability, fat, carbohydrates, and other composition factors.

– The microbe type, age, and number: resistance to heat, affinity for growth in the food product, and initial number of organisms present.
– The heat transfer characteristics of the package, the food product, and the heating source media.

Product Type

For extremely acidic products such as lemon juice (pH $\approx$ 2.3) or sauerkraut (pH $\approx$ 3.6), the processing requirements are very different than for a pH-neutral (pH $\approx$ 7.0) growth media such as beef broth. The primary reason for this is that *Clostridium botulinum* organisms, which are responsible for botulism poisoning and thus of great concern to processors, do not grow below a pH of slightly less than 4.6. For this reason, the dividing line between low-acid foods that require thermal treatment to achieve commercial sterility and inactivate spore forming bacteria and high-acid foods that generally only require pasteurization processing to inactivate molds and yeasts is set at a pH value of 4.6. Fermented, low-Ph foods such as sauerkraut are considered in the same category as high-acid foods. An additional category, *acidified foods*, includes low-acid foods that have had acids or acid foods added, and have a final, equilibrium pH of less than 4.6 and a water activity above 0.85. These require thermal processing to kill the vegetative cells of pathogens and spoilage organisms, but unlike low-acid foods, spore activation is considered to be controlled by pH. Low-water-activity ($A_w < 0.85$), high-acid foods such as jams and jellies are also not considered an acidified food provided criteria for acid, brix (sugar content), and other conditions are met.

Microbe Type, Age, and Number

Given the extraordinary number of bacteria, molds, and yeast that love to share our food with us, it would be difficult to discuss each of them in the space of this book, but concentrating on a few will give a general indication of the nature of the organisms and their growth preferences, as shown in Figure 6.1 [2].

Research beginning in the 1920s has indicated that the age of the particular colony may have some effect on the viability of some organisms, with thermal resistance at its lowest during the logarithmic growth phase and highest during the stationary phase, but the mechanism is not well understood and the organism should be treated as if it were at its most resistant period in order to provide a margin of safety [3].

Common sense would argue against subjecting the processing facilities and equipment to intentional contamination by a deadly pathogen such as *C. botulinum* for the purposes of testing whether or not thermal processes are adequate to the task of eliminating that particular organism. For this reason, a closely associated non-pathogenic organism such as *C. sporogenes* PA3679 is used because it is slightly less thermally susceptible (giving a safety margin in the thermal process) and can be easily assayed.

Thermal processing causes an exponential reduction in microbial activity that never exactly reaches zero even though it can attain infinitesimally small numbers, with a corresponding improbability of illness. For this reason, thermal processes are established on a probabilistic basis that seeks to produce an extraordinarily low probability of contamination. For example, for *C. Botulinum*, the standard end point for thermal processing is to provide a 10^{12} reduction in population, presuming an initial population of 1 spore per gram of product. This is often taken to be one (or fewer) surviving organism per 1,000,000,000,000 containers, neglecting

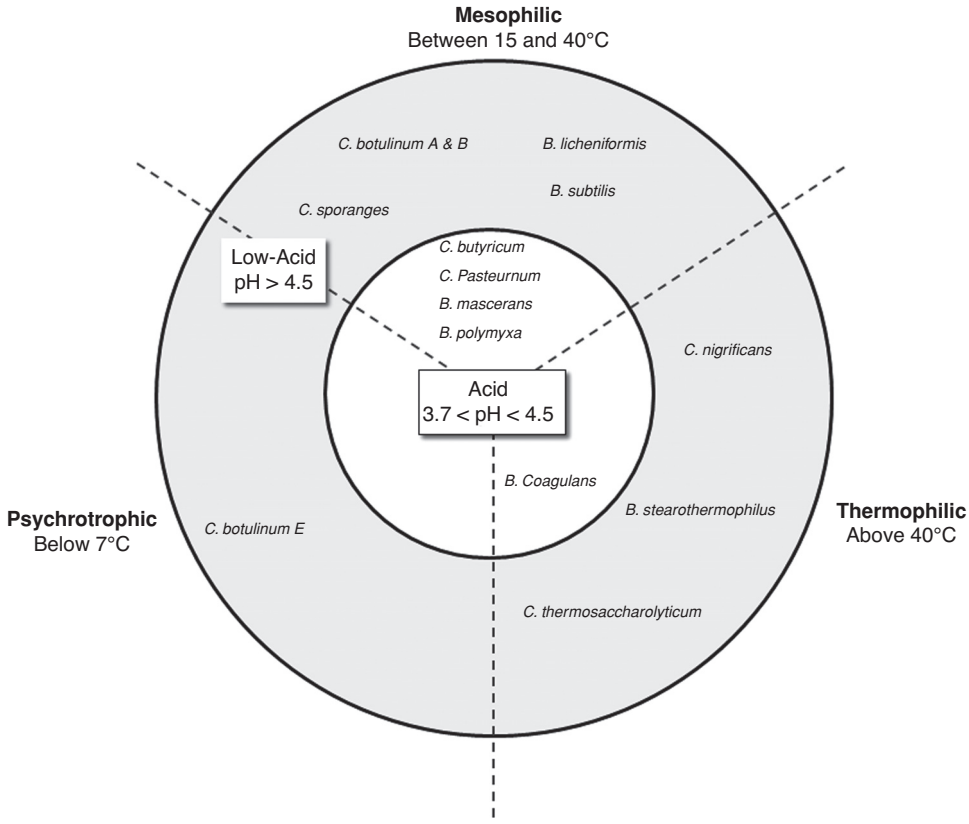

Figure 6.1. Thermal and pH Conditions for Spore-Forming Spoilage Bacteria

the size of the container in the calculation. Nevertheless, the processing industry practices rigorous controls, and outbreaks of botulism poisoning due to under-processing are extremely rare (estimated at four cases and two deaths between 1942 and 1980 after producing more than 30 billion cans of food) [4].

Heat Transfer Characteristics

Heat transfer, previously discussed in Chapter 2, is a critical part of thermal processing. For heat to kill organisms, it has to be applied to the media containing the organisms at sufficient level and for sufficient time to ensure commercial sterility. This can be a simple arrangement, such as in-home canning where the product is treated in boiling water for long periods, or a complex system such as in-flow microwave processing where microbial reduction occurs from heat being created in the product itself by an EMF field.

For simpler types of thermal processing, the basic type of processing (canning, in-line pasteurization, and the like) as well as the type of heat source (most often, steam) and heat transfer regime (conductive, convective, or a composite means) used will affect the processing variables such as time and temperature involved in achieving a particular level of pasteurization or commercial sterilization.

Microbial Destruction, Thermal Death Time, D, and z values

Thermal Death Time (TDT) is a measurement of the time a particular process requires to kill a given number of organisms at a specific set of operating conditions. These are usually temperature and operating pressure for traditional canning processes that hold containers for treatment or may be related to temperature and heating-section residence times in pasteurization and aseptic processing lines where the product flows continuously through the system. These requirements are usually determined by both calculation and test-operation using indicator organisms. It is critical to understand that although many examples and a great deal of literature is based on the assumption of a simple logarithmic death curve, the actual death time and death rate is more likely to be a statistically uncertain function and must always be checked against trials of the actual system to ensure adequate microbial reduction. Decimal Reduction Time, usually termed *D*-Value, and abbreviated as simply *D*, is the amount of time that a thermal treatment process, operating under a particular set of process parameters, requires to kill 90% of the pathogen population present at the start of the time interval. Processes are often specified in terms of the *D*-value and, as previously discussed, most commercial sterilization procedures require a *12-D* reduction in a particular organism (usually *C. botulinum*), producing the probability of contamination to be 1: 1.0×10^{-12} per gram of product. Note the effect of pH on the *D* value for an idealized product shown in Figure 6.2 [5].

The *z*-value is the temperature change required to shift the process microbial destruction curve by one log cycle, as shown in Figure 6.3. Thus, if a process has a 12-*D* value of 20 minutes,

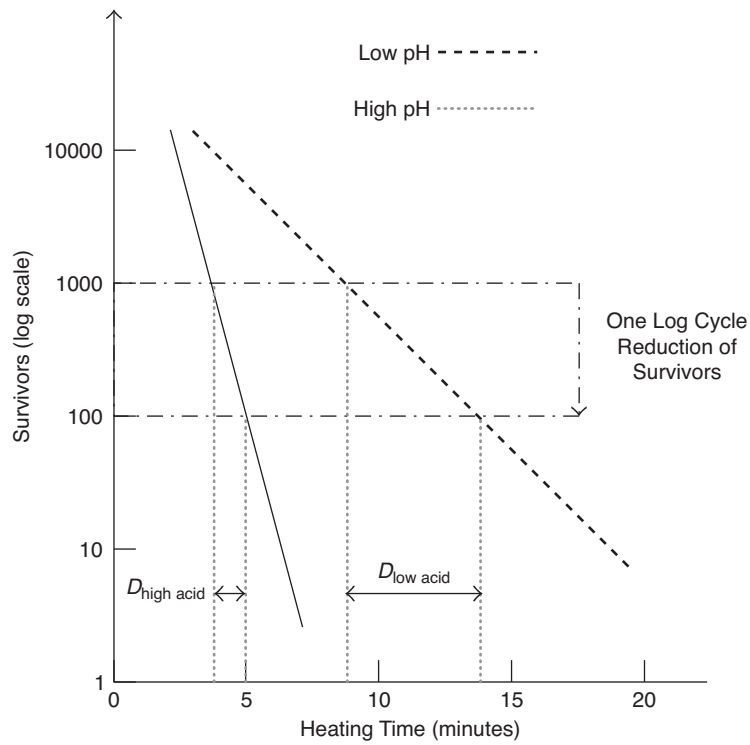

Figure 6.2. Calculation of *D* Values in Thermal Processing

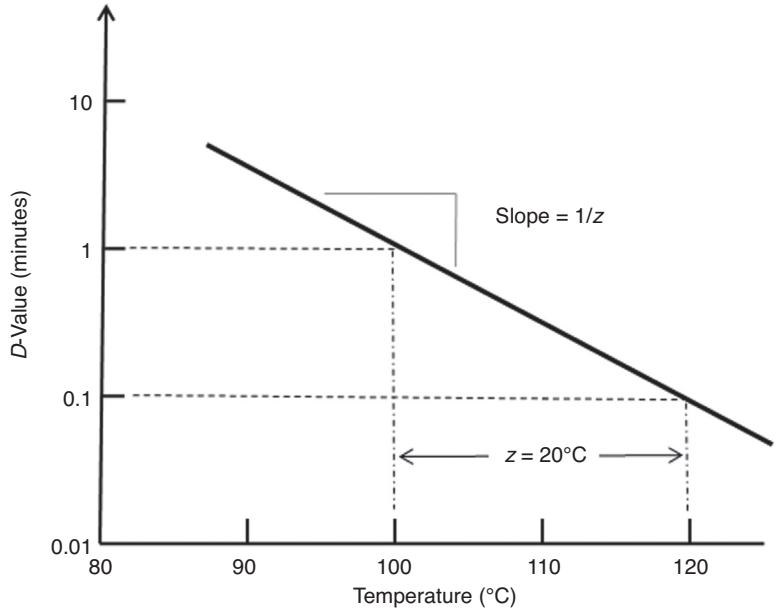

Figure 6.3. Calculation of *z* Values in Thermal Processing

and a *z*-value of 10°C, a 10-degree increase in temperature will result in the process time being shifted to 2.0 minutes, and a 10-degree decrease from the original starting temperature will result in the process time being shifted to 200 minutes. Of course, these figures have practical limits. *z*-values should be confined to known useful values for the microbial destruction curve – extrapolating the above figures by cooling the process by 50 degrees from the original starting temperature would extend the process time to 2,000,000 minutes – about 3.8 years!!

To evaluate and compare processes using a relatively standard reference point, the F-value represents the total time to achieve a desired *D*-value (E.g. 12-*D* for *C. botulinum*), and is frequently referenced at a standard temperature of 121°C (250°F), and thus is abbreviated as D_{121}. The simplest value for F is an F_0 value that assumes an instantaneous and homogenous temperature change throughout the product and will provide a calculation of the ideal-case for processing.

$$F_0 = D\,(\log N_\mathrm{i} - \log N_\mathrm{f}) \tag{6.1}$$

N_i: initial microbial load
N_f: final microbial load

Because of the realities of heat transfer and the economics and mechanics of real-world processing operations, neither the amount of thermal input will be instant, nor the temperature profile homogeneous. Thus, a number of methods have been devised for calculating the overall F value that integrates the thermal energy input and resultant lethality.

The usual notation for F values has the z value as the superscript and the temperature as the subscript, where both are expressed in the same units system. Thus, for a standard temperature of 121°C (250°F) and a z value of 15°C (27°F), the F value would be expressed as $F_{121}{}^{15}$ or $F_{250}{}^{27}$ in Fahrenheit units.

F-Values that represent the logarithmic-linear (first-order) reduction curve of the traditional literature must be modified with the realization that the assumption that microbial populations are homogeneous is as unlikely as the assumption that the human population is homogeneous. Research has shown that the thermal death kinetics of actual microbial populations are more often a higher-order function and may be described by several different mathematical functions.

For example, rather than a simple Arrhenius curve, the death curve may be a concave logarithmic function described either by

$$(Log\ N(t)/N_0) = -kt^p \tag{6.2}$$

$N(t)$: Population at time(t)
N_0: Population at time($t = 0$)
k: Population curve constant
t: Time, s
p: Process Constant

or by several other functions involving Weibull frequency distribution models that, although both literally and figuratively complex at times, can incorporate information about non-Arrhenius death curves and accurately describes nearly any real-world time-temperature death time correlations [6, 7]. Most of all, given that there is a likely degree of uncertainty about the exactness of the thermal death curves, it is important that validation studies are done to ensure adequate processing.

Thermal Process Calculations

Commercial Sterilization

Calculating accurate and useful operational parameters for thermal processing is the subject of a great deal of engineering effort and literature. Although there are formulas and tabular systems that are commonly used in determining process parameters, there is also a growing array of computational tools ranging from spreadsheets to complex neural network systems to provide accurate, safe process lethality parameters [8].

The General Method and its variants use the fact that within limits, time and temperature can be traded off to provide the desired lethality for a process. Thus, the sterilizing effect as the temperature profile changes in the food material is accommodated by factoring its equivalent value at a reference temperature (usually 250°F/121°C) and summing the values. The *lethal rate* can be calculated as shown in Equation 6.3.

$$\text{Lethal Rate} = 1/10^{(121^\circ C - T)/z} \tag{6.3}$$

Thus, for a z value of 20, the values for lethal rate are shown in Table 6.1.

These can be used to "accumulate" lethal rate value as the area under a lethal rate curve, as shown in Figure 6.4, which links temperature and time to lethality via translation through the lethal rate values (which, of course, can be automated in actual use).

This effect can accommodate both the heating and cooling profiles in the container, which are measured by placing a thermocouple or similar temperature recording device in the *cold spot* of the container and monitoring the temperature during the process operation. Summing the

Table 6.1. Lethal Rate Values for $z = 20$

Temperature	Minutes at 121°C	Temperature	Minutes at 121°C
100	0.089	119	0.794
101	0.100	120	0.891
102	0.112	121	1.000
103	0.126	122	1.122
104	0.141	123	1.259
105	0.158	124	1.413
106	0.178	125	1.585
107	0.200	126	1.778
108	0.224	127	1.995
109	0.251	128	2.239
110	0.282	129	2.512
111	0.316	130	2.818
112	0.355	131	3.162
113	0.398	132	3.548
114	0.447	133	3.981
115	0.501	134	4.467
116	0.562	135	5.012
117	0.631	136	5.623
118	0.708	137	6.310

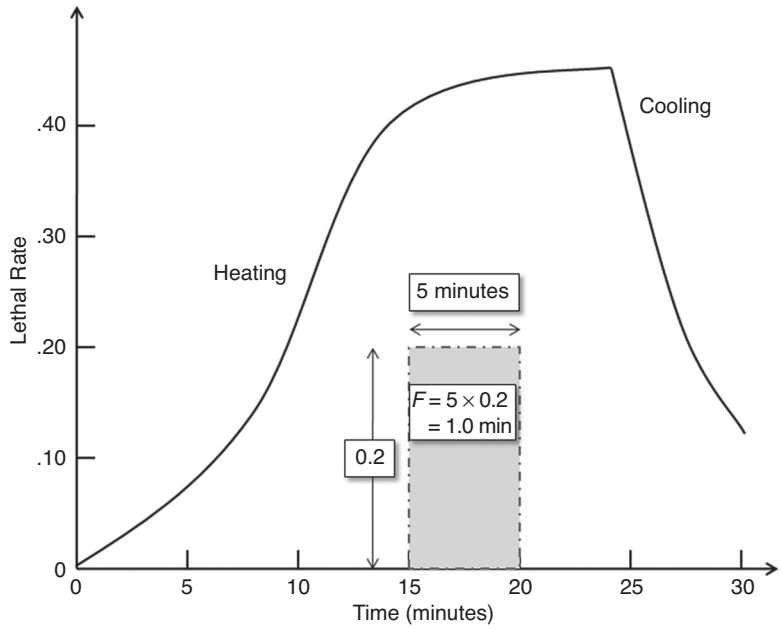

Figure 6.4. Lethal Rate Curve for Process Calculation

equivalent lethality values over the total process will give an equivalent lethality value ($F_{250/121}$) for the organism in question. This will have particular applications in calculating the lethality of pasteurization and aseptic processes where temperature lag is considered to be minimal. The general method and its variations have the limitation of only indirectly returning operating parameters such as retort shutoff time, and may require trial-and-error calculations to converge on a working answer.

The Formula Method, first published by Ball in 1923 [9] and subsequently refined [10], uses knowledge of the heating and cooling lag in the product as well as operating conditions in combination with tabular calculation coefficients to provide a good approximation of the operating time required by a particular process to achieve commercial sterilization. In its simplest variation, to calculate the value for the thermal process time, t_B, the general formula

$$t_B = f_h \log\left[\frac{j_h(T_M - T_i)}{g}\right] \tag{6.4}$$

f_h: the heating rate constant, the slope of the steady − state heating curve.

$$j_h = \frac{T_M - T_1}{T_M - T_0}$$

$$j_c = \frac{T_1 - T_M}{T_0 - T_M}$$

$$g = T_M - T_B$$

j_h: heating lag constant
j_c: cooling lag constant
T_M: The temperature of the heating media
T_i: The initial temperature of the product
T_B: The reference point (usually the "cold point") temperature in the container
g: The product temperature difference between the heating media and the reference point in the material at the end of the product heating

can be used where j_h, the heating lag constant, accounts for the nonlinear temperature change when heating begins and has not yet achieved a linear rate of increase, and j_c, the cooling lag constant, as defined by accounts for the nonlinear temperature change when cooling begins and has not yet achieved a linear rate of decrease.

It is most usual to use Ball's calculation method in conjunction with graphs (or their digital equivalent) of (f_h/U) versus log (g), where U is the Thermal Death Time for a particular z temperature value, which will allow rapid estimation of the thermal processing times.

The general and formula methods, as well as many variants that have been developed over the years, are rapidly being supplemented with computer-based heat transfer modeling that will allow not only a more accurate estimation of the heat penetration into the product and the resulting deaths of populations of microbes whose response to heat may be non-linear, but can help understand materials that undergo phase changes or other substantial thermodynamic effects in order to create the proper balance between product over-processing and inadequate sterilization. Additionally, these newer methods can be used to analyze less severe treatment methods such as pasteurization and sterilization using HTST/UHT processing. No matter which methods are used, however, it must be re-emphasized that process verification tests

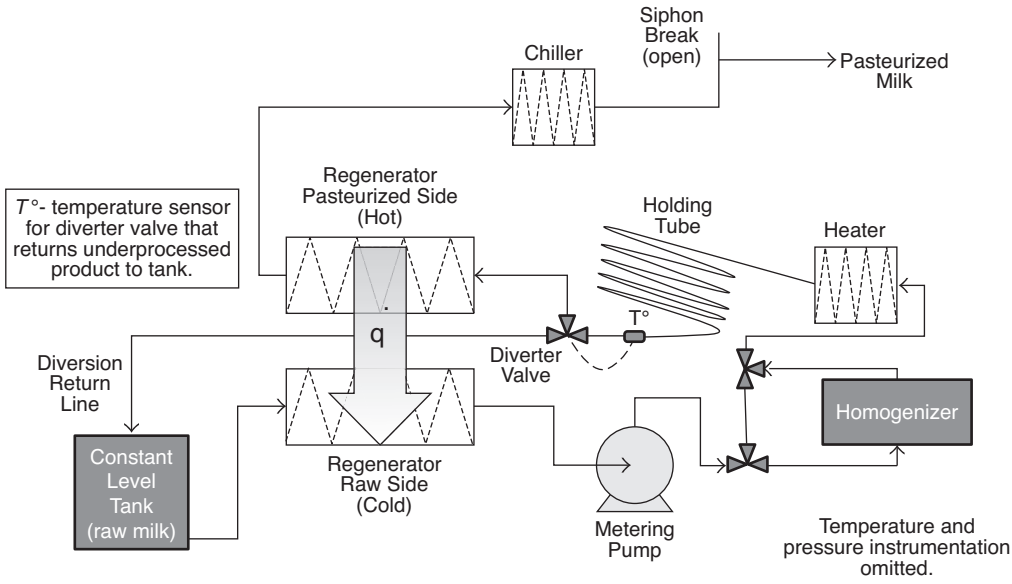

Figure 6.5. Basic Pasteurizer Flow Diagram

must be conducted to verify that calculations have resulted in the required degree of microbial destruction.

Pasteurization is a less severe form of heat treatment, generally applied to destroy particular organisms of interest in a particular food product. The most common form of pasteurization is applied to milk in order to destroy *Mycobacterium tuberculosis* and *Coxiella burnetii*. For milk products, the rate of destruction of phosphatase is used as the evaluation criteria because it is destroyed at a slightly higher temperature but approximately the same rate as the two organisms, and is easily assayed; presence of the enzyme is a simple indication of inadequate treatment.

The schematic for a pasteurizer is shown in Figure 6.5. Although there are a number of variations depending on manufacturer, all share several common safety features. In all designs, pressure gradients are incorporated to ensure that any leakage forces processed product back into the unprocessed stream (particularly through regenerator heat exchangers), thermal sensors return under-processed product to the process intake, and vacuum/siphon breaks are incorporated to prevent the system from drawing processed product back through the system.

UHT processing is an extension of this technology with higher temperatures and shorter residence times. Standard pasteurization schemes for milk can be any of several time and temperature combinations, as shown in Table 6.2 [11], with the resulting shelf life and intermediate processes such as ESL processes discussed in Chapter 7.

Although the HTST processing criteria presents the temptation to construct a *flash* process that heats milk to the boiling point for a fraction of a second, the realities of heat transfer and fluid flow usually provide a discouraging counterpoint in that it may be very difficult to create enough mixing and heat transfer to guarantee proper heating of every part of the product in such a short time without destroying product quality. As a result, the slower processes are more common in order to ensure both proper processing and a safety margin in the response capability of the pasteurization equipment.

Table 6.2. Pasteurization Times and Temperatures

Type of Pasteurization Process	Time	Temperature
Low Temperature, Low Time (LTLT)	63°C/145°F	30 minutes
High Temperature, Short Time (HTST)	72°C/161°F	15 seconds
	89°C/191°F	1.0 second
	90°C/194°F	0.5 second
	94°C/201°F	0.1 second
	100°C/212°F	0.01 second

Other types of products such as fruit juice are pasteurized both to extend shelf life under refrigerated storage and to reduce or eliminate the presence of certain microorganisms such as *E. coli* 0157:h7 that have been associated with unpasteurized cider [12]. Non-thermal methods such as ultraviolet pasteurization have been investigated for some of these applications in order to provide a low-cost means for small processors to meet standards. Other non-thermal and thermal-supplementing types of pasteurization such as ozone use or thermosonication have been investigated for some time and are discussed later in the chapter, but cost and dependability issues usually restrict widespread implementation [13]. Chemical additives can be used as an adjunct, but may interfere with flavor characteristics or prevent an "organic" label claim.

Hot Fill Processes

As the name implies, hot fill processing involves heating the product to a temperature below its boiling point, often 90–95°C, holding it at that temperature for up to 30 seconds, and then filling the container at a temperature up to approximately 85°C (185°F) when used with plastic containers, or possibly higher with glass and paper-based cartons. This offers the advantage of using the product itself to reduce or eliminate microbial activity on the packaging material, as well as providing a shelf-stable product for low-pH, high-acid foods and an extended shelf life for higher-pH products. This process is commonly used with juices, teas, and other drinks, and when combined with a properly stable or preserved product can provide ease of filling as well as thermal treatment to viscous food product such as jams and fillings. The packaging challenge has been to provide plastic containers that will withstand elevated filling temperatures, and PET, PBT, and its variants dominate this market for consumer applications, often with vacuum panel design features to disguise the vacuum collapse that occurs after cooling the sealed product. Hot filling for bulk ingredients may use any number of thermally stable containers including pails and aseptically lined intermediate bulk containers.

Degradation of Product During Thermal Processing

Many types of degradation may occur in products as a result of thermal treatment, even though the treatment may not be excessive. Thermolabile components may include enzymes, vitamins, starches, proteins, color, and flavor compounds all of which will have a thermal destruction profile similar in concept to those previously discussed for microorganisms (Table 6.3). In general, the balance that must be achieved trades degradation of the food product against microbial safety, and the impetus is to provide processing methods that reduce the former without compromising the latter. Because microorganisms are often much more thermally

Table 6.3. *z* and *D* Values for Bacteria and Food Components

	z-value (°C)	D_{121} (min)
Bacteria	5–10	1–5
Enzymes	30–40	1–5
Vitamins	20–25	150–200
Pigments	40–70	15–50

Source: University of Guelph. "Thermal Destruction of Microorganisms." http://www.foodsci.uoguelph.ca/dairyedu/TDT.html

susceptible than vitamins and pigments, the destruction rate per unit time may be much higher and the application of a short-term heat impulse can result in microbial destruction and enzymatic inactivation with minimal loss of other desirable components, as shown in Figure 6.6.

This difference in reaction time again leads to the development of Ultra High Temperature (UHT) processes that typically will hold the product for 1–3 seconds at temperatures well above boiling (typically, 135°C/275°F). This will retain many of the characteristics of the food product and lead to a shelf-stable product such as UHT milk and many soups, broths, and sauces currently on the market. The temperatures used may present other problems, such as the caramelization of sugars that gives UHT milk its distinctive taste, but many products previously sold in cans have shown a notable quality increase with the adoption of UHT processing combined with aseptic packaging operations.

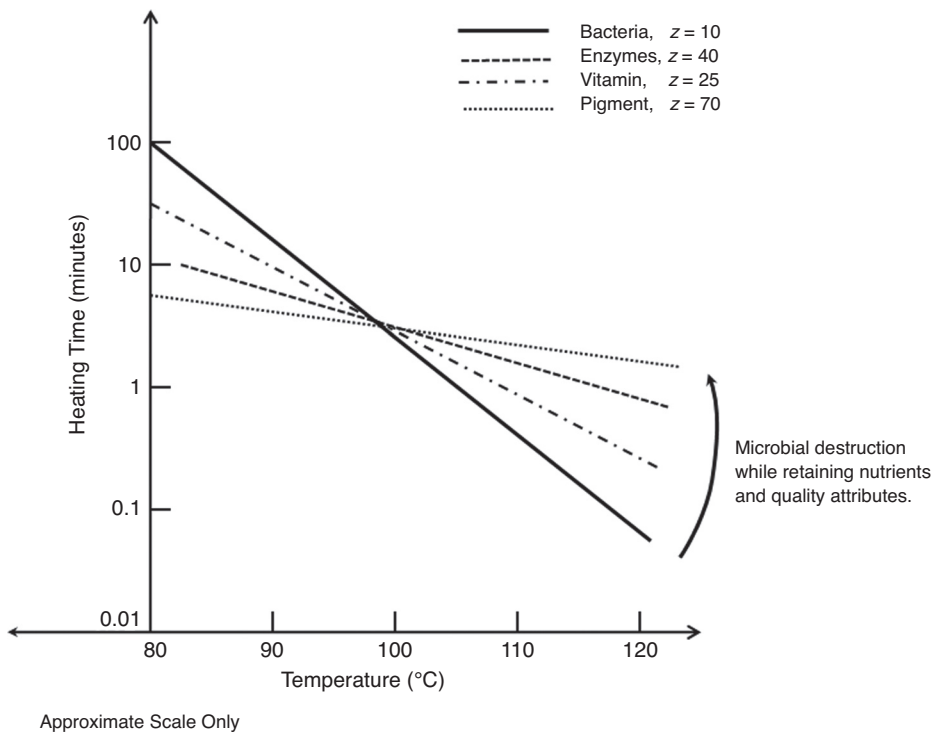

Figure 6.6. Relative *z* Values for Bacteria and Food Components

Aseptic Packaging

Aseptic packaging in and of itself is nothing new. Early canning experiments involved out-of-package sterilization and canning under steam or ultraviolet light, but the common current usage refers to a combination of UHT or HTST processing as previously described, hermetically sealed packaging to prevent recontamination of the product, and a fabrication and assembly system that operates under sterile conditions in order to fill and seal the package after processing the product such that further processing will not be necessary to provide an extended shelf life or commercial sterility.

The initial markets for early experimentation with aseptic processing and packaging systems were typically regional milk producers who needed to extend shelf lives of product, and various development efforts led to the production of the first tetrahedral milk packages formed from chemically sterilized barrier material in a modified TetraPak® machine in the early 1960s. Although the unusual original tetrahedral packages were never widely accepted by the American consumer who had ready access to quantities of refrigerated fresh milk, large quantities were successfully distributed to the military and to school lunch programs.

Subsequent production of more conventional rectangular and semi-rectangular packages followed in tandem with the growth of technical knowledge, improved barriers for packaging materials, and the inevitably slow regulatory approval, particularly for low-acid food products that are ideal bacterial growth media. Although the technology is much more energy and material efficient, as shown in Figure 6.7, the enormous infrastructure for the manufacture, distribution, and storage of canned, frozen, and refrigerated products in developed countries as well as regulatory concerns over process safety have slowed the adoption of many aseptic products on a widespread basis.

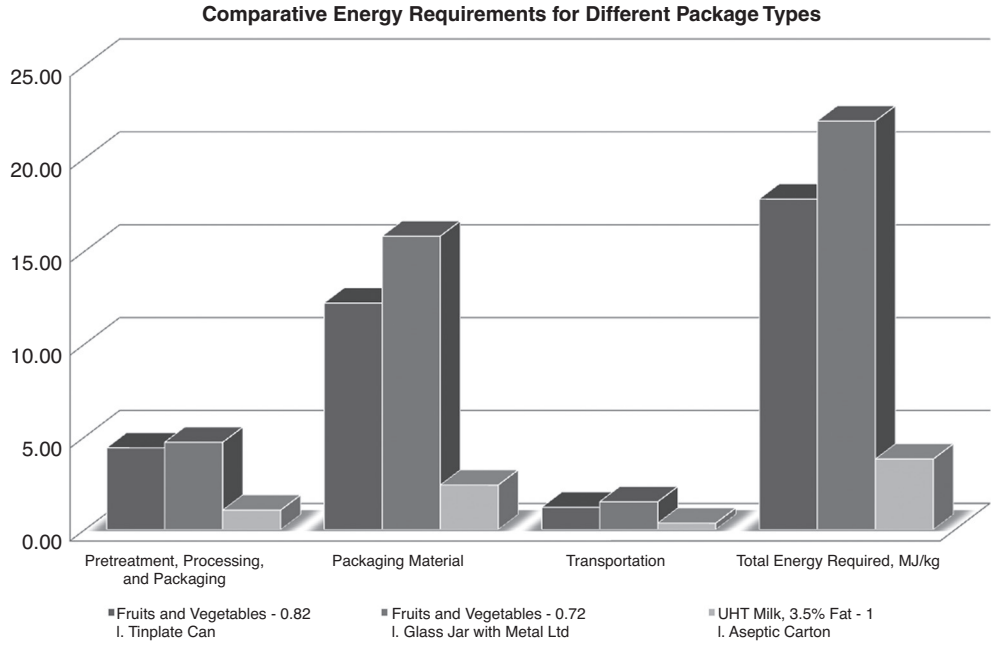

Figure 6.7. Energy Use in Aseptic and Traditional Processing Methods
Source: Derived from: Reuter, H. (1980) Verpackung von Lebensmitteln, 31, 132–6

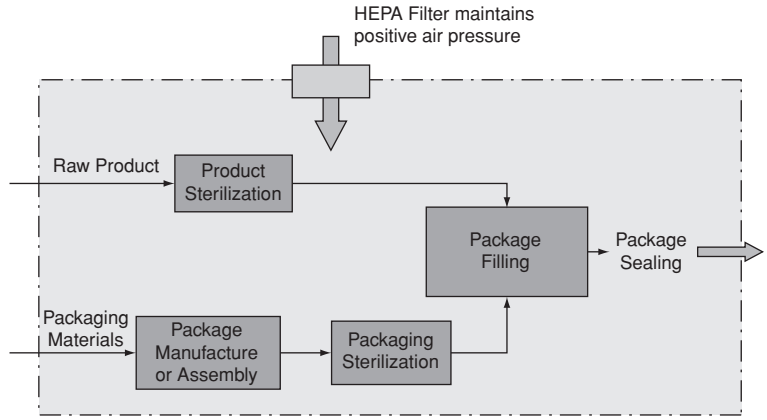

Figure 6.8. Aseptic Processing Line Diagram

Niche products such as soups, broths, cheese sauces, and the like have seen a high degree of success for particular applications by reducing empty container disposal costs, reducing injuries from cans, and often improving product quality for the consumer, and inroads are being made in traditional markets for processed whole tomato products, meat stews, and fish.

General Layout of Aseptic Processing Lines

As shown in Figure 6.8, the general layout of the aseptic process involves constructing and sterilizing the food package and treating the product, then combining them both under sterile conditions to provide a shelf-stable product. This is most often done in a sealed enclosure, under a positive air pressure environment provided by a HEPA (High Efficiency Particulate Air) filter and blower assembly. Given that there are a variety of package types, products, sterilants, and other variables, there are some general principles that are at work, but the larger process must be developed individually.

Product Sterilization

Product sterilization for non-particulate liquid products is a relatively straightforward process based on existing UHT technology, and the same simple heat exchangers may often be used. For products containing small particulates ($<$10 mm), the retardation of heat transmission to the center of the particles may require scraped-surface heat exchangers to ensure that proper heat transmission occurs, and may increase equipment costs and maintenance problems. Larger particulates ($>$10 mm) may require sterilization of the liquid and solid components in separate stages, then recombination in order to ensure adequate processing [14].

Packaging Material Sterilization

Material sterilization is an essential part of the aseptic packaging process and may be achieved by many means, although fewer are actually useful or effective because of material degradation, toxicity, workplace hazard, or cost. Many of the potential sterilizing methods and materials may be toxic, or may degrade or contaminate the packaging material or affect its ability to perform over long periods of time. Thus, ethylene oxide, hydrogen peroxide, heat, or steam may remain

the process of choice for some time. Electron beam or gamma radiation suggest themselves if the installation, operation, and safety concerns can be managed.

Equipment Concerns

Establishing and keeping a sterile environment within the operating machinery is one of the most difficult facets of making the transition to aseptic packaging operations. Training personnel to avoid accidental contamination is a critical facet of maintaining high production levels, because simply reaching inside to make an adjustment requires complete re-sterilization of the entire production line. Some newer installations are built with access glove boxes and tunnel-entry protective suits to allow workers "hands-on" access to equipment and operations. Additional considerations such as the maintenance of a sterile environment in the event of power fluctuation or HEPA airflow pressure variations are an additional concern.

Commercial Microwave Processing

"Commercial" microwave devices, whose principles of operation have been discussed in Chapter 2, have come to mean microwave ovens that are similar to home ovens but used in commercial facilities, most often restaurants and cafeterias, and these may use the 915 MHz frequency that was allocated (along with 2,450 MHz) for microwave use. Although various companies have worked with different types of microwave technology to produce competitors to current thermal methods of processing, the effectiveness of these against spore-forming bacteria have not been proven [15]. In addition, the cost and complexity of installed equipment, potential operator safety hazards, and the relatively low thermal efficiency relative to direct combustion or even resistance heating has always been a barrier for mainstream applications such as simple food processing, freeze drying, and grain drying, although microwave furnaces are used for critical applications in ceramics and composite materials processing. One paradoxical use that the food industry has found for microwave heating is for "tempering" frozen foods, particularly frozen meat carcasses and cuts – warming them to temperatures near the melting point so that additional operations such as slicing, grinding, and forming can be performed.

Alternate Processing Technologies

Other technologies being investigated for commercial use as sterilization or pasteurization techniques are often derived from technologies that have existed for some time but have been resurrected because of new developments such as advanced control systems or new refinements in methodology. As with many of the previously discussed methods, the refinements and developments for applications for food were originally developed for other industries and have been subsequently adapted to food processing uses. Some of these methods include light-based processing, pulsed electric field and electric discharge, ultrasound, ultra-high-pressure, and various *multiple-hurdle* processing methods that may combine several of these methods and include conventional thermal heating or chemical additives. Most of these new processing technologies are being tested on an experimental basis for use with fluid and semi-fluid food products [16].

Light-Based Processing

Light-based processing has existed for decades, with some of the original processes for pasteurization of milk dating back to the 1930s, but light offers a potentially good source of energy

transfer for the proper product. Ultraviolet light processing using equipment originally designed for UV curing of coatings and adhesives has been tested as a means for modest cider processors to achieve an approximately 5-log reduction in bacterial count and to reduce the risk from *e. coli 017:H7*. Although there is a risk of photolysis of flavor and nutritional compounds, the microbial contamination risk in these products is great enough that the small changes that might occur are considered acceptable. Research on very-high-intensity light, typically from a gas discharge tube or arc source, is still being done in an effort to reduce thermal degradation of flavor and odor during processing, particularly of UHT milk and similar products.

Because the transfer of energy to microbial contaminants is hampered by the opacity of a material or by the *shadowing* of organisms by particulates, light-based processing methods have limited usefulness in many products except for surface treatments and relatively transparent products. For this reason, most light and pulsed-light systems are systems for the pasteurization of clear or translucent fluids. Ultraviolet systems using short-wavelength, high-energy light typically comprise a flow cell and light source, typically of annular design that allows the product to flow in the space between an inner light tube and an outer shell. Pulsed-light systems are more often for "flash" treatment of both products and material surfaces, including packaging materials and medical devices and materials. Because pulsed-light systems operate intermittently by creating broad spectrum, high-energy discharges, they are typically constructed as treatment chambers, holding the product in static position temporarily allowing the light pulse to occur, although several in-flow systems have been licensed. The pulsed-light method has not proven to be a commercial success, however, and as of this writing is not being manufactured.

Pulsed and Oscillating EMF Treatments

Several variations of pulsed electric fields, oscillating or pulsed magnetic fields, and electric arc discharge have been used on an experimented basis, with only the pulsed electric field systems yielding a large body of promising research results. Arc discharge, which can produce any number of electrolytic breakdown compounds, works by producing both electrolysis compounds – particularly free radicals – and hydraulic shock phenomena that are similar to the ultrasonic methods discussed elsewhere. The breakdown compounds may cause problems with textures or flavors in food products, and have made the process one that is unlikely to be used in its current state for food products. Although the Electropure Process milk processing system dates back to the 1920s, current implementations claim that they can be more energy efficient than other electrically based systems. These can combine to lyse cells and produce pasteurization or sterilization effects, but current research is sparse and more work needs to be done for large-scale processing to be widely adopted.

High-intensity and oscillating magnetic field technology uses either an oscillating magnetic field where the polar orientation switches periodically or a static magnetic field where the polar orientation remains constant to attempt to reduce or eliminate microbial contamination. Results of these treatments are mixed, with different types of organisms exhibiting various reactions ranging from population reduction to increases in colony formation, depending on the type of organism. To further complicate the problem, generation of a high-intensity magnetic field typically requires a high-energy discharge from a capacitor bank, or other technology such as superconducting magnetic coils, which implies that large-scale processing would be complex and energy-intensive, although the magnetohydrodynamic induction effect on the ionic compounds implies that a properly constructed device could both treat and pump liquid materials simultaneously.

Pulsed electric field (PEF) technology has seen the most research focus, and various implementations have shown some promise for the pasteurization or sterilization of liquid materials. The chief mechanism for pathogen destruction appears to by electroporation or electrical breakdown of the cell wall. Both of these proposed mechanisms lead to the eventual destruction of the cell wall and loss of cell contents, killing the pathogen, although electroporation will allow the uptake of external material, and is a common laboratory technique for the introduction of external DNA into a cell culture. The effectiveness of PEF treatments is a multivariate effect of field intensity, number and duration of pulses, pulse waveform, temperature during treatment, and various product factors such as dielectric strength, pH, and composition, as well as the type and concentration of specific organism being treated. Current experimental methods involve using equipment that incorporates the discharge electrodes into a flow cell that will allow continuous flow of relatively low-viscosity material.

Ultrasonic Methods

Ultrasonic treatment of foods is a technology currently being studied both as a stand-alone technology and in combination with other treatments to provide a multiple-hurdle approach to microbial reduction. Ultrasonic treatment has been shown to act by disrupting the cell wall and allowing the cell contents to escape, and with the increased durability of piezoceramic transducers may provide an efficient method of energy transfer into microorganisms, unaffected by opacity or electrical properties. Because ultrasound is a periodic function, and is subject to loss factors in some types of materials where the energy is converted to heat and dissipated rather than being transmitted – a so-called *lossy* material – there would be some few limitations placed on materials that have a high percentage of entrained air such as foams. Microencapsulated materials might suffer product quality loss because of micellar rupture, essentially rupturing the microencapsulation system in a manner similar to that which provides destruction of the cellular membranes. Although ultrasound technologies have been shown to be moderately effective in reducing microbial populations, due largely to cavitation effects, the incorporation of ultrasound into multiple-hurdle technologies such as heat, reduced pressure, and the use of additional sterilants such as ozone and hydrogen peroxide and ultrasound shows promise for energy-efficient processing methods. Additionally, exploratory uses of multifrequency and multimode systems have been shown to increase the effectiveness of sterilization methods.

High-Pressure Processing

As previously discussed in Chapter 2, high-pressure processing (typically up to approximately 600 MPa) shows promise for the reduction or elimination of microbial populations of vegetative *E. Coli*, *Salmonella*, and *Listeria* in moist, air-free foods, and for the creation of extended shelf-life foods, although the resistance of spores to pressures in excess of 1 GPa has thus far limited applications to pasteurization [17]. These methods have shown great promise for the enhancement of traditional thermal processing methods but have seen the largest growth for use with products where the texture changes do not detract from perceived product quality, and the retention of flavor and shelf life justifies the high equipment costs.

Although many of these processes show promise to complement thermal processing, without the clear advantage of equivalent processing capabilities and safety at modest cost, their greatest

use may be to supplement or enhance existing process systems, although this will negate many of the low-temperature benefits. Improvements, standardization, and an understanding of the specific modes of action of these methods and others will be a critical factor in any potential replacement of thermal or radiation processing [18].

Refrigeration and Freezing of Foods

Foods have been frozen or preserved by low temperatures for as long as any kind of cold environment has been available. However, the modern variety of refrigerated and frozen foods has only come into its own since the World War II, assisted by home refrigeration and distribution and warehousing systems that allow relatively accurate temperature control throughout the product's pre-consumer existence.

Refrigerated distribution of commercial foods began with Swifts's early experiments with boxcars for carcasses bound from the Chicago slaughterhouses to eastern U.S. markets. These railcars were packed with ice in various configurations and early versions had problems either as a result of discoloring the carcasses or not adequately controlling the temperature. Later varieties solved these problems as well as load instability damage, although implementation by railroads was slow because of the threat to the railroads' existing live-animal handling infrastructure. Despite this, refrigerated carcasses steadily replaced live animal shipments throughout the 1880s, with the trend continuing until live-animal shipments became a rarity. When Chicago's rail lines were extended to nearby Milwaukee, Wisconsin, the beer manufacturers located there used the new technology to return-ship chilled, unpasteurized beer in the empty cars to Chicago's much larger markets.

Additionally, refrigerated and cooled railcars (*reefers*) allowed the development of the now-enormous national citrus and vegetable industries in areas of the American Southeast [19]. Iced reefers, initially dependent on regular loading with natural snow and ice, were in service until the 1970s but were all eventually replaced by cars with mechanically driven refrigeration, which allowed better temperature control and essentially unlimited use provided the compressor was refueled.

At the same time that reefers were being developed in the United States, mechanically refrigerated loads of mutton and beef were being shipped from Australia to Britain and Europe on freezer ships with compressors driven from the ships' steam engines, allowing both a needed supply of meat to those countries and a boost to the Australian economy. Japanese railroad systems were similarly reported to use cooled, ventilated, and occasionally iced shipments for the shorter-distance transport of seafood, fruit, and vegetables.

Current distribution methods include refrigerated truck trailers that typically have a diesel-driven compressor, as well as intermodal containers that can be run from a ship's central electric source so that unitized shipments can be maintained for very large distances.

Frozen foods were first commercialized in the United States by Clarence Birdseye who, while working as a naturalist to pay college expenses, observed that fish caught by the Inuit froze very quickly in arctic temperatures while maintaining their flavor and texture. Although foods were commercially frozen at higher temperatures, Birdseye's contribution was to flash-freeze foods so that ice crystals would have little time to grow and disrupt the cellular structure of the fruit, meats, and vegetables being processed, resulting in a higher-quality product. Birdseye's first continuous freezing system involved pressing waxed containers of food product between brine-chilled belts, which reduced air voids and increased heat transfer [20] – a method still in use in modified form using chilled plates.

Types of Freezing Systems

Freezing systems all seek to remove heat from the product as efficiently as possible. To this end, several varieties of refrigeration systems have been developed, which can be broadly grouped into three categories: non-contact, indirect contact, and direct-contact immersion/spray systems. All of these rely on large installations of refrigeration equipment that operate in a manner similar to the vapor compression refrigeration processes described in Chapter 5.

Non-contact *air blast* freezers rely on a high-velocity stream of refrigerated air to remove heat from the product, and are used for products that have shapes or configuration that do not allow contact-freezing to work well. Because of the insulating effects of packaging materials (if present) and the lower efficiency of heat removal using this method, the residence time in the freezer may be longer. Fluidized bed airflow freezers may allow quick freezing of granular or particulate materials in a manner similar to the fluidized bed dryers discussed later in this chapter, because the product will be suspended in the freezing air blast with good circulation around the product.

Indirect contact systems rely on conductive heat transfer away from the product, typically into chilled plates that may contain circulating coolant. Although conductive heat transfer may be more effective than other types, plate freezers may compress foods into unpalatable bricks, and single-surface plate freezers may not freeze foods rapidly enough. Liquid products are typically chilled to near-freezing in indirect heat exchangers before final freezing either in packages, as with fruit-juice concentrates, or in forms and molds as may be done with juice bars. Final freezing (*hardening*) can be done with an air-blast freezer.

Direct-contact freezing depends on the circulation of liquid or sprayed refrigerant on the product either before or after packaging. This may be even more effective than indirect-contact freezers, because the product can be sprayed or immersed directly in refrigerant, often carbon dioxide or nitrogen, as required. Brine-based immersion systems may be used as well, although the brine must be removed from the product after immersion, which may be difficult for unpackaged products.

Frozen Food Properties and Processes

Because the thermal properties of foods change as they change phase into a frozen state, the prediction of specific properties of frozen foods have been the subject of a good deal of research. A good example is simple water ice, which has a thermal conductivity approximately 3.8 times that of liquid water and a density of about 0.92. As one might expect, a freezing process will therefore be nonlinear as the *freezing front* – the boundary of the frozen region – progresses inward through the product. Similarly, the latent and sensible heat given up by the product as it freezes will both affect the freezing time and may produce profound effects in the product, because the extracted heat must be conducted outward through the already frozen material and may cause product degradation as it does so. Numerical analysis of freezing fronts have provided some insight into the nature of the thermal properties of food during freezing, but broad estimation and trial-and-error persist as the usual method of determining process time [21]. Degradation of frozen products is often termed *freezer burn* because of the browned, dehydrated state of the product. This has been shown to be a function not only of temperature but of the state of water in foods, with browning and other processes dependent on whether the food material is above or below the glass transition temperature, T_g covered in Chapter 7, which may be well below the observed *frozen solid* point for many foods [22]. The product

may be above its T_g point yet still appear to be thoroughly frozen, and with moisture more free to migrate through and out of the product, the shelf life will be severely reduced. Unfortunately, many consumer freezers are not capable of maintaining the extremely cold (−23°C [−10°F] to −32°C [−25°F]) conditions required to lower most foods below their T_g and achieve very stable storage conditions, so shelf life may be limited even with good protective packaging.

Refrigerated Foods

Refrigeration using natural ice, cold water, or mechanical means has been used to extend the shelf life of products throughout history. This typically occurs by reducing the growth rate of spoilage microorganisms for products such as meats and dairy, as well as reducing the growth and ripening rate of fresh fruit and vegetables. When combined with pasteurization to reduce the initial microbial load, products such as milk and lunch meats can be kept fresh for at least two weeks, allowing wide distribution and home consumption [23]. Most refrigerated products represent an early form of multiple-hurdle preservation in the sense that they are typically treated either with chemical preservatives, pasteurization, or other physical means to reduce the microbial count, and then the temperature is held as close as possible to freezing to reduce the microbial growth rate. This process has been extended to include multiple-technology extended shelf life products such as bagged salads, which use a chlorine-based wash to reduce microbial loads and then refrigeration and controlled-atmosphere packaging to stabilize the product for long periods of time. Fresh pasta products have seen similar success by using oxygen-absorbent packets, and dairy products such as cottage cheese are kept fresh by refrigeration and the addition of carbon dioxide [24].

One of the major concerns with refrigerated foods is *temperature abuse* in the supply chain – conditions that expose the product to destructive temperature rises and fluctuations. A malfunctioning refrigeration system can cause the temperature to rise and microbial growth to achieve an unacceptable rate without outward indication, but careless handling is usually the predominant cause of the problem. Many devices have been developed to track the temperature profile during storage, and most suffer from the lack of ability to record anything but surface temperatures, which can give a misleading impression of the internal temperature of a product. Newer devices that include sufficient data storage and computing power are doing a better job of estimating shelf life based on simple Arrhenius curves, but simple chemical and physical indicators will usually only give surface indications and may be error-prone for all but the most sensitive products [25]. The surface data collected by temperature loggers is useful for estimating product damage if the thermal properties of the product are known and *dummy* products (or genuine ones with embedded data collection) are occasionally used.

Temperature abuse beyond the supply chain is a major concern with regard to food safety – the inability of consumers to store foods in appropriate ways should never be doubted, and packaging should be clearly labeled if refrigeration or freezing is required, even if it seems obvious. Additionally, many consumers have poorly regulated refrigerators either because of mis-set thermostats, mechanical problems, or the refrigerator used in a house with children that can have the door opening and closing much of the day. Finally, it may not be possible for consumers to transport refrigerated or frozen foods any distance without special measures. Consumers in the American South and Southwest have been observed carrying frozen foods home from grocery stores in ice chests, even while travelling in air-conditioned cars, because of the extreme heat.

Drying of Foods

Another historical method of food preservation is drying, which effectively reduces moisture availability for spoilage and often provides a more compact means of food storage by eliminating excess water. The general process of adiabatic drying has been discussed in Chapter 2, but the practical application of drying processes can take many forms.

The earliest types of consumer food drying were simple air-circulation racks and drying trays that were used to dry fish, meats, and vegetables, some of which are still in use in remote areas both for crops such as dates and coffee as well as for locally consumed fish, meats, fruit, and vegetables. Because these are subject to both contamination by dirt, insects, and vermin as well as the vagaries of the weather, they are generally not accepted for many products that must meet an exact production schedule or strict cleanliness standards.

Because drying methods rely on the ability of moisture to be removed through the product cross-section, it is usually a common sense step to reduce this distance as much as possible to increase the speed and efficiency of drying and reduce the possibility of scorching or cracking the outer surface of the product. Thus, traditionally dried meats are cut into thin strips first, and in commercial operations fruit is typically cut in thin slices or chunks, while particulates, fluid films, and fluid drops are kept as small as practicable in most drying.

Generalized Drying Mechanisms

There are several distinct types of simple drying that occur, and these may occur sequentially during many types of drying processes. These have been segmented by mechanism into a constant rate and two falling rate curves that will occur when moisture content and drying rate are compared as shown in Figure 6.9.

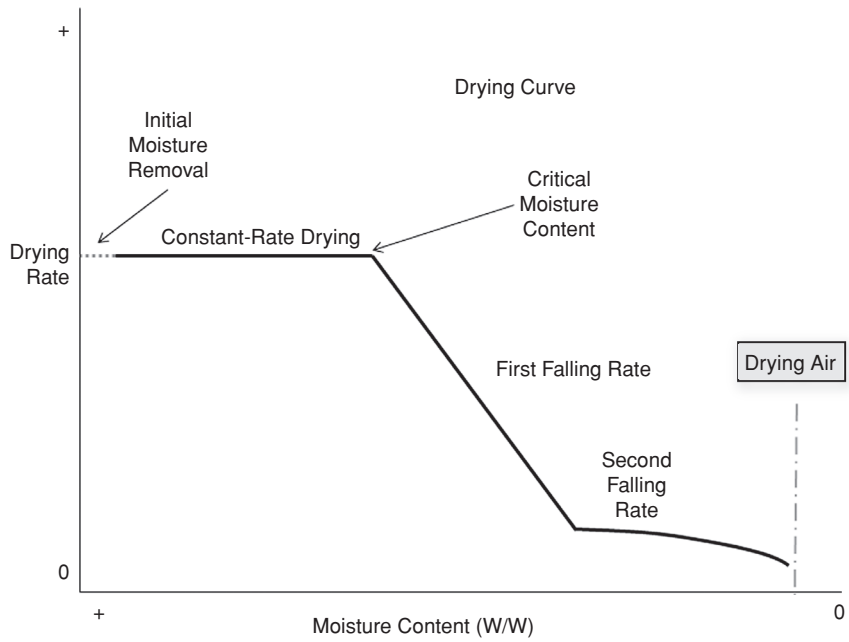

Figure 6.9. Generalized Drying Curve

Initial Moisture Removal (Warming Stage)

In this stage, surface moisture and moisture lying very near the surface will be removed by direct evaporation into the air stream for as long as there is moisture available, and the product may exhibit a temperature rise limited to that of the wet-bulb temperature of the air.

Constant Rate

Constant-rate drying usually occurs at a constant temperature, and is only limited by the heat-transfer rate into the product and moisture, and the mass-transfer rate into the air stream. When the easily removed moisture is completely removed – the so-called Critical Moisture Content – moisture removal will continue at a decreasing rate.

First Falling Rate

In the first falling rate, product drying is limited by the ability of moisture to diffuse from the center of the product to the surface for removal and is limited by heat transfer and the diffusion characteristics of water in the material.

Second Falling Rate

The second falling rate is the point at which the A_w of the product begins to decrease below 1.0 (the bound water capacity of the product), and the rate of moisture removal is limited by a complex set of factors such as thermal conductivity of the product because the moisture must be vaporized within the product before it can diffuse outward to the surface for removal. During this second rate, the moisture-binding mechanism may shift to a system that may appear as another falling rate, where the moisture is held in low-order molecular layers by capillary condensation* and is usually quite stable.

Termination

Termination of the drying process is usually determined by the desired moisture content of the product, because excess drying is costly, requiring both energy and time with diminishing returns near the termination point. Drying is usually terminated in the stable region described in the previous paragraph in order to provide a shelf-stable product, but if run indefinitely will produce a product that is in equilibrium with the drying airflow. For contact-drying (non-airflow) operations, more moisture may be driven out.

Prediction of Drying Times

Prediction of drying times may be taken from trial data and extrapolated graphically using a simple chart of time versus moisture content once the final drying rate has been established, or can be done algebraically using the slope of the experimentally determined drying curves, although this is usually unnecessary. In either case, the variability among product batches and drying machinery requires verification of drying times until the consistency of the process can be assured.

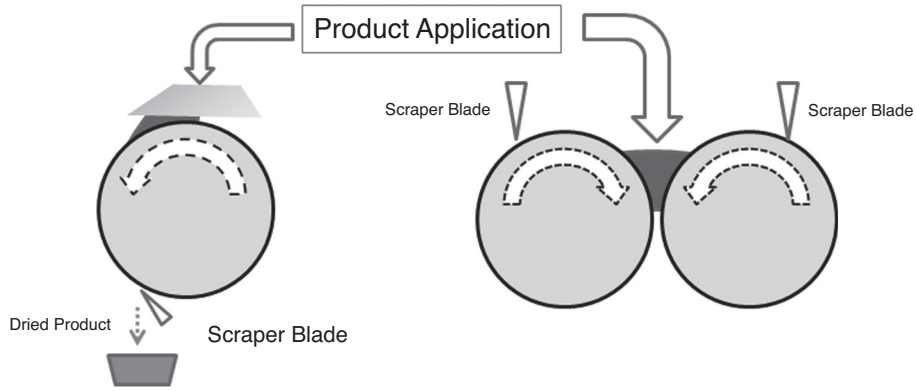

Figure 6.10. Drum Dryer Diagram

Drying Methods

Drum Drying

Drum drying is one of the few direct-heat-conduction-based drying methods, and is used to evaporate solvents from a thin film of material that is spread on the surface of a heated rotating drum as shown in Figure 6.10. Typical food products that are produced with drum drying include many powdered products such as cornstarch, dried yeasts, dried soups, milk and fruit pulps, and mashed potato flakes. Although the product is usually composed of solids contained in a liquid or slurry of some sort, it is possible to use a rolling hot drum to dry other products (as is done in the paper industry as described in Chapter 3). Cereals may be produced by heated rollers "ironing" blobs of cooked grain into dried flakes in a single process, and product feed is often very similar to the coating methods described in Chapter 5, with product either being fed directly to the heated steel drum, applied via a transfer roller or spray, or applied between two closely spaced drums. The surface finish of the drum may be important because application doctor blades may control the applied film thickness, and there may be scraper blades assisting in the removal of the dried product. Highly polished surfaces or nonstick plating may assist in removal of some products.

The rate of moisture removal is a function of the product's film thickness and thermal conductivity, the drum's heat differential between the drum surface, the product, and the outside air, the relative humidity of the air, the useful fraction of the drum that the product is in contact with, and the time that the film is in contact with the heated drum, which is a function of the product's feed rate and the drum rotation speed. Other factors such as the gelatinization region in starch feeds may also figure in some operations [26].

While it is possible to construct a general equation describing the heat transfer characteristics of particular machinery, these ballpark figures inevitably are used as a starting point for adjusting the process operations to a more optimal state. More importantly, recent developments in sensors and controls can allow a process operation to self-tune to match the product and any changes in the process while it is in operation.

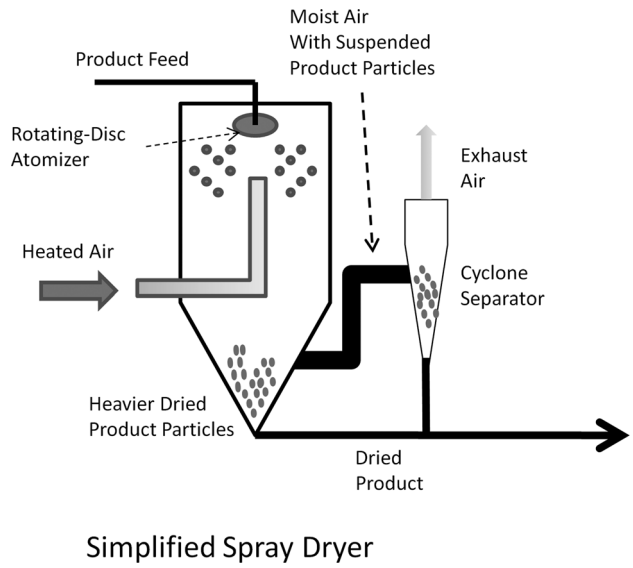

Figure 6.11. Spray Dryer Diagram

Spray Drying

Spray drying is used to dry liquid materials such as instant beverages and other food components. It relies on a stream of liquid particles produced by a spinning disc, which are descending into an upward-flowing stream of heated air (Figure 6.11). As the liquid particles lose moisture and collapse into granular particles, they fall to the bottom of the dryer and are collected. Because of the small particle size, drying is accomplished quickly and the product is somewhat protected by the evaporative cooling that occurs in the dryer. Product spray must be kept in suspension long enough so that it will dry to particles that do not have enough moisture to clump or cake when collected, typically below 5%. There is also a limitation on the types of materials that may be processed in a spray dryer because, for example, extremely high sugar contents will cause the droplets to spin outward as long threads, effectively converting the dryer into a cotton-candy machine. Because of problems with particle adhesion either because of inadequate drying or static charge, cleaning devices such as sonic horns may be fitted to dryer cavities to force accumulations loose.

Spray drying calculations are often based on mass-transfer principles derived from idealized droplet sizes, airflow, and operating conditions, and are largely derived from coefficients based on drying curve data and psychrometric data for the airflow. Many of the calculations do not account for the changing nature of the droplets both in terms of physical dimension and thermodynamic properties, nor do they account for humidification of the airflow while in the dryer. Because of this, manufacturer's operating information and experience are critical in optimizing practical performance of spray dryers.

Fluidized Bed Drying

Fluidized bed drying suspends a moving horizontal flow of particulate material in an upward-flowing stream of air. This allows air to forcibly circulate around the product and, if designed

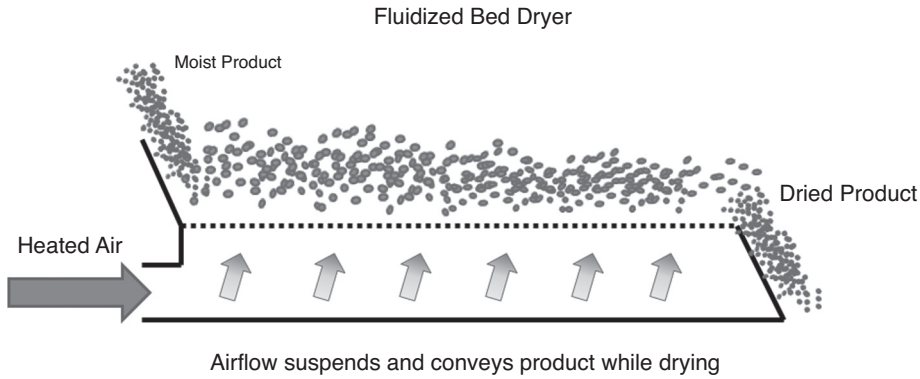

Figure 6.12. Fluidized Bed Dryer

properly, will cause the product pieces to rotate and tumble somewhat (preferably without being ejected), leading to more uniform drying, as shown in Figure 6.12. Proper design and adjustment of the dryer is important because the product will lose mass as it moves to the end of the process, so there is a risk of the product lifting out of the dryer stream as it progresses. Design parameters for fluidized bed drying are similar to spray drying in that moisture loss is governed by similar mechanisms, and a force balance between the downward force of gravity and the lifting force of the aerodynamic drag on the particles must be considered (similar to the force balance considerations for the Stokes Equation shown in Figure 6.18). Lighter products are more readily accommodated by this method because high velocities of airflow will be needed for heavy materials, but for very specific applications, such as fluidized-bed combustion systems in furnaces used to efficiently burn fuels such as coal and municipal waste, it is possible to develop an appropriate heavy-materials process.

Tray, Belt, and Tunnel Dryers

Tray and tunnel dryers, and their continuous-feed counterpart, belt dryers, all work on the principle of simple airflow around a product that is mechanically suspended in the drying airstream, usually on mesh belts or ventilated sheet metal trays. For some types of dryers, drying is conducted under reduced pressure in order to facilitate moisture removal at lower temperatures to preserve flavor, although the operating costs of this type of process can be higher. Larger tunnel systems may accommodate stacked cars of trays that travel through the airflow in the drying tunnel.

Belt and tunnel systems may be operated in either a concurrent or countercurrent mode with the movement of the product either opposing or coinciding with the airflow, depending on product quality requirements. Countercurrent operation offers the advantage of having the wettest product exposed to the hottest and driest airflow with an attendant efficiency increase (as with heat exchangers, discussed in Chapter 2), but at a risk of damaging the product from overheating resulting in surface cracking, caramelization, or an undesirable texture change.

Infrared, Geothermal, Solar, and Microwave-Assisted Dryers

Radiant heat may be used for drying, as it always has been for earlier civilizations, but the undependability of weather and security of power networks makes a strong argument for

exposure to artificial drying energy, even if the initial source of the power is from a solar installation. Some processing plants are intentionally located near sources of geothermal heat or in areas where there is a predominantly sunny climate or the climate is dominated by hot, low humidity air to increase the efficiency and reduce energy consumption in the processing plant. Infrared drying may be done with electric or fueled burners built behind radiant plates.

Microwave-assisted drying has been an experimental subject for many years, but the inefficiency of energy conversion and expense and hazards associated with high-energy microwave generation have restricted practical implementation. The singular advantage of microwave heating has – efficient energy penetration – makes it useful for very specific types of drying such as pharmaceutical products, and it has been incorporated to a certain extent in freeze-drying operations.

Freeze-Drying

Freeze-drying, first used as an atmospheric-pressure sublimation process by Incas in the cold, dry Peruvian Andes mountains, was industrially developed as an extension of the lyophilization process developed in the early part of the twentieth century to dry chemicals in the laboratory. Although freeze-drying is most usually applied in the pharmaceutical industry and comprises a relatively small portion of the entire food-drying industry, it can offer many advantages for specific applications such as light weight, long shelf life, and high product quality.

The general process path of freeze-drying exploits the direct sublimation of solid ice to vapor under a moderate vacuum. Although the process is fairly simple, there are some limitations inherent in the heat and mass-transfer that can cause problems with a high quality product.

General Freeze-Drying Procedure

The general process path for freeze-drying is shown in Figure 6.13 and usually consists of:

1. Loading of frozen foods into vacuum chamber: The foods are typically frozen to a temperature below the eutectic point (the lowest temperature point where liquid and solid coexist, and in the case of water the triple point). Materials with indistinct phases may use a *critical point* similar to the eutectic.
2. Reduction of pressure to below the eutectic: This guarantees that water will sublimate directly to vapor rather than going through a liquid phase.
3. Heating Stages: After this point is reached, heat is supplied under vacuum to the material in order to drive the water into the vapor where it is removed by vacuum pumps and condensed or adsorbed out of the system. There may be several heating stages to liberate not only free water but adsorbed water and possibly other volatiles. The ability of the system to remove water is often practically limited by the ability of the material to conduct heat from the heated surfaces. Because the material is desiccated at the region nearest the heat source first as moisture is driven out, the thermal conductivity of that layer of product decreases rapidly as the drying process occurs – it becomes self-insulating in a sense – and can cause insufficient drying in layers of product that are too thick. Similarly, moisture that is sublimated away from the heating surface can remain in the surface regions of the product, causing quality and texture problems as shown in Figure 6.14.

   Experiments have been conducted with microwave heating of food during freeze-drying [27], but although this method overcomes many of the heat conduction problems exhibited by tray-type heaters in freeze-drying, practical implementation has been slow.

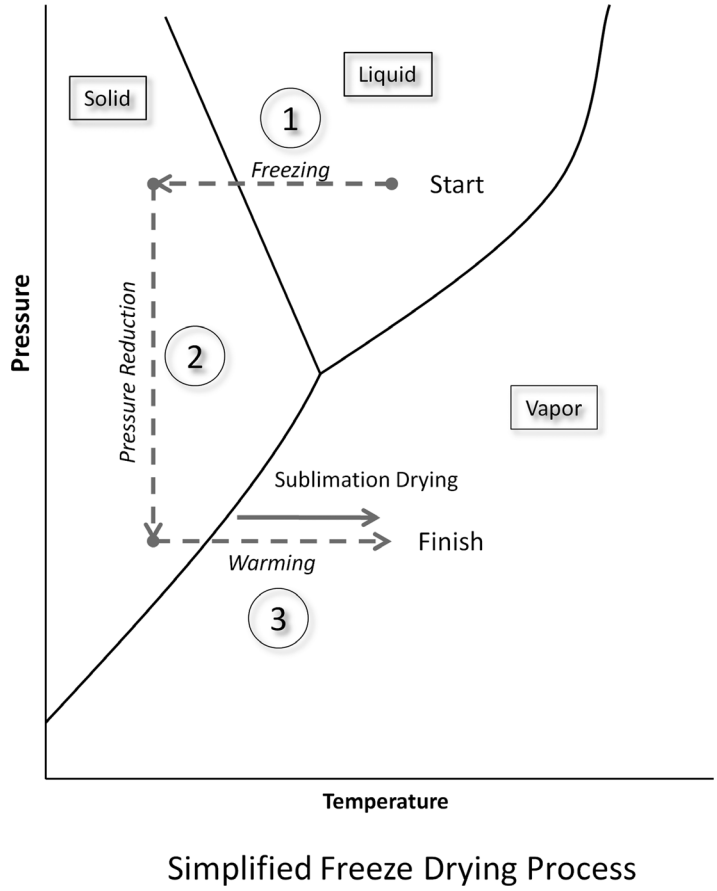

Figure 6.13. Freeze-Drying Process Path

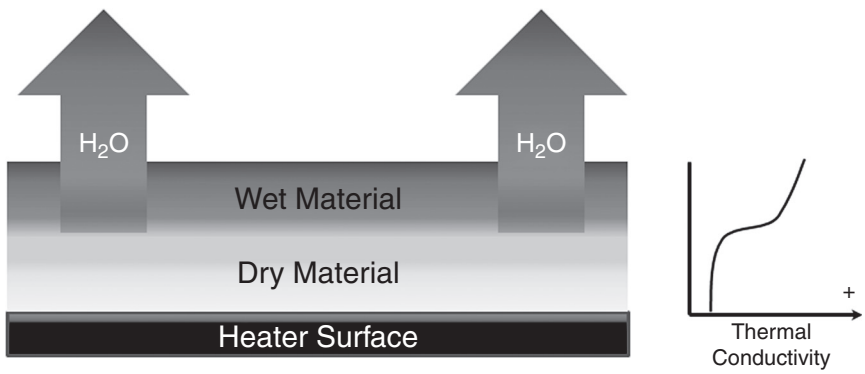

Figure 6.14. Insulation and Mass-Transfer Effects During Freeze Drying

4. The process is ended by returning the system back to atmospheric pressure either with a simple venting or by backflushing with inert gas to protect the product, although the latter is more common with freeze-drying in the pharmaceutical industry.

One of the product limitations inherent in freeze-drying is that the region nearest the heat will sublimate moisture first, which has two effects. The first of these is that the product will usually become porous as ice is removed, which reduces its thermal conductivity enormously – in effect creating its own "foam" insulating layer and disrupting the ability of the product to be heated further. The second of these effects occurs because the moisture must travel through the remaining product as it vaporizes, which may over-humidify the rest of the product. The result of too-fast drying may be scorched or collapsed product that is unappealing, or a *stalled* process that has a moist surface that cannot be easily removed. Because of the reduced pressure, convective heat transfer may be difficult, so radiant and/or conductive heat with carefully controlled operational parameters is most often used. Microwave heating is an appealing addition to this because it is not dependent on conduction and can provide a uniform heat profile in the product, but the expense and hazards associated with this have restricted its large-scale implementation in bulk food processing [28].

Drying time estimation can be expressed as [29]:

$$t_d = \frac{L^2 \rho (m_i - m_f) \Delta H_s}{8 k_d (T_s - T_i)} \tag{6.5}$$

t_d: drying time, s
L: slab thickness, m
ρ: density, kg/m^3
$(m_i - m_f)$: moisture content difference at dry/frozen interface, kg/kg
H_s: latent heat of sublimation, kj/kg
k_d: thermal conductivity of dry layer, $\frac{\text{W}}{\text{m} \cdot {}^\circ\text{K}}$
T_s: surface temperature, $^\circ$K
T_i: internal temperature at dry/frozen interface, $^\circ$K

In general, freeze-dried and lyophilized products are processed in thin cross-sections of solid or liquid product to minimize the heat transfer and moisture diffusion problems. Often, a great deal of trial and error is involved in this, particularly with oily or high-sugar-content products, to avoid the collapse of the structure and loss of organoleptic properties while efficiently transferring heat into and moisture out of the product.

Irradiation

Irradiation using radioactive substances or charged particles is one of the newer types of food preservation techniques, and is an outgrowth of radiation research that began in earnest before World War II, although the concept of preservation with radiation was considered in 1905 with the idea of embedding food with thorium in order to render it sterile (fortunately, the Pure Food and Drug Act of 1906 intervened)[30]. The National Food Irradiation Program that spanned the period from 1953 to 1980 provided the largest impetus for developing food irradiation processes

and techniques, with that function being assumed by the USDA in 1980. Since then, irradiation has been approved for use with pork, spices, vegetables, poultry, shell eggs, and various meat products. All of these approvals are at very specific dosage ranges and are intended to extend shelf life and to control foodborne pathogens [31]. Additionally many other items from medical supplies to cosmetics may be irradiated at high levels for the purposes of sterilization.

Irradiation Background

When accelerated electrons, X-rays from scattered electron beams, or charged particles (typically gamma rays) interact with both food and packaging materials, radical species are formed that damage the DNA and metabolic structure of microorganisms, causing both death and the inability to reproduce, as well as potentially causing radiolysis products and chain interactions in the polymers of packaging materials.

Three types of irradiation are typically used: electron beam, X-ray, and gamma radiation. The first two are based on electron accelerators and require substantial electric supplies but do not contain any permanently radioactive materials – turning off the power removes any charged particles. Both of these rely on the acceleration of electrons; however, the electron beam (*E-beam*) systems produce a stream of electrons that have a limited ability to penetrate targets (typically to a depth of approximately 3–6 cm.), and requires only modest shielding. X-ray systems generate more energetic X-rays by directing the electron beam at a highly electron-dense plate (often of gold, tungsten, or tantalum) where the impinging electrons knock orbital electrons out of the plate material's inner orbits, cascading replacement electrons from higher-energy orbits to fill the orbital shell and causing the emission of their excess energy as X-rays. This latter process is very inefficient and requires heavy shielding, but offers the advantage of much greater penetration depth (in excess of 120 cm).

The last method, gamma irradiation, relies on an isotope (most commonly, ^{60}Co, although ^{137}Cs may be used in some countries) that emit high-energy photons but do not emit neutrons, which effectively eliminates the formation of radioactive species in the target. These installations require an extensive infrastructure both for handling materials to be irradiated and for the storage of isotopes and protection of personnel, but offer the advantage of very good penetration and very-high-energy irradiation.

Levels of total absorbed irradiation are regulated in kilograys (kGy) and are typically used at the following levels given in (Table 6.4), although not all are approved for commercial food processing use in the United States (Table 6.5) [32–34].

Effects on Packaging Materials

One of the great advantages of food irradiation is that it can penetrate a finished and sealed package, so the effects of radiation on packaging materials are of concern both from the standpoint of public perception and of safety regulations. A list of approved materials was originally produced during the initial period of intense irradiation research in the early 1960s, but as the preservation method went out of favor, new approvals were not sought because there was insufficient demand to justify the time and expense of getting full approval. With the resurgence of interest in irradiation, materials manufacturers and regulators were caught off-guard by the demand for approval of materials that had been developed in the interim. Fortunately, there are currently Threshold of Regulation (TOR) exemptions in the United States for low-dosage packaging systems using approved food contact polymers and adjuvants that

Table 6.4. General Irradiation Levels for Foods and Other Applications

Levels of Absorbed Radiation for Various Applications*	
1 kGy = 100 kilorad, where the kilorad (krad) is an older unit of measure. Both are the equivalent of 1 kilojoule of energy per kilogram of material.	
Dose Level	Application
"Low" doses (up to 1 kGy)	
0.15–0.5 kGy	Control insects in grains
0.05–0.15	Inhibit sprouting in white potatoes, onions, garlic, ginger, etc.
0.15–0.5	Control trichinae in pork, insect infestation in dried fruits and dried fish.
0.15–1.0	Inhibit ripening and decay and control insects in fruits and vegetables.
"Medium" doses (1–10 kGy)	
1.0–3.0	Control *Salmonella*, *Shigella*, *Campylobacter, Yersinia*, and *E. Coli* in meat, poultry, and fish; delay mold growth on strawberries and other fruits.
1.0–7.0	Eliminate spoilage of fresh, frozen, and pathogenic seafood, and of raw or frozen poultry and meat.
2.0–7.0	Improve technological grapes (increasing properties of food juice yield), dehydrated vegetables (reduced cooking time), etc.
"High" doses (greater than 10 kGy)	
10–30	Kill microorganisms and insects in spices, enzyme food additives, ingredients preparations, etc.
30–50	Commercially sterilized foods. The only US approval for this dosage level is for food products for immune-compromised patients.
25–40	Complete sterilization of implants, sutures, drapes, syringes, and neurosurgery devices.

*Generalized figures – not all of these are approved by all regulatory agencies in the US or other countries.

operate at doses of 3 kGy or less and either under an oxygen-free environment or under vacuum while frozen. Current U.S. Approval Levels for irradiation of packaging material are given in Table 6.6.

Polymeric packaging materials will exhibit simultaneous radiation-induced cross-linking and chain-scission, with the final effect being the result of the dosage and conversion efficiency of each process. Secondary factors such as oxygen availability, secondary compounds, type, dose, and dose rate, among others, will affect the final results of the materials treatment. E-beam and X-ray treatments have been shown to produce fewer volatiles such as aldehydes, ketones, and carboxylic acids than gamma irradiation in polymeric packaging materials [35]. Mechanical changes will follow with the scission/cross-linking competition, with an increase in cross-linking increasing the modulus of the materials and beyond a certain level causing embrittlement and failure. Paper, which is composed of cellulose chains, will show radiolytic reactions similar to synthetic polymers, with scission of the cellulose chains, darkening of the paper structure, and an increased susceptibility to acid hydrolysis and alkali solubility [36]. Metals and glass are sufficiently stable for irradiation, but secondary compounds associated with them, such as coatings, linings, and sealants, may show degradation.

Food products for retail to consumers that have been irradiated must display the radura emblem on the packaging, as well as a label statement indicating processing by irradiation,

Table 6.5. U.S. Food Irradiation Levels

Current US Approval Levels for Irradiation of Food Products[i]		
Food Type	Purpose	Dose Level
Fresh, non-heated processed pork	Control of *Trichinella spiralis*	0.3 kGy min. to 1 kGy max.
Fresh foods	Growth and maturation inhibition	1 kGy max.
All foods	Arthropod disinfestation	1 kGy max.
Dry or dehydrated enzyme preparations	Microbial disinfection	10 kGy max.
Dry or dehydrated spices/seasonings	Microbial disinfection	30 kGy max.
Fresh or frozen uncooked poultry products	Pathogen control	3 kGy max.
Frozen packaged meats (solely NASA)	Sterilization	44 kGy min.
Refrigerated uncooked meat products	Pathogen control	4.5 kGy max.
Frozen uncooked meat products	Pathogen control	7 kGy max.
Fresh shell eggs	Control of *Salmonella*	3.0 kGy max.
Seeds for sprouting	Control of microbial pathogens	8.0 kGy max.
Fresh or frozen molluscan shellfish	Control of *Vibrio* species and other food-borne pathogens	5.5 kGy max.
Fresh Fruit	Delay Maturation	1.0 kGy

[i]"US FDA/CFSAN–REGULATORY REPORT–Irradiation of Food Packaging Materials." http://www.cfsan.fda.gov/~dms/irradrpt.html.

Table 6.6. U.S. Approved Packaging Materials for Irradiation

Packaging Materials Listed in 21 CFR 179.45 for Use During Irradiation of Prepackaged Foods.		
Section	Material	Max. Dose (kGy)
179.45(b)	Nitrocellulose-coated cellophane	10
	Glassine paper	10
	Wax-coated paperboard	10
	Polyolefin film	10
	Kraft paper	0.5
	Polyethylene terephthalate film	10
	Polystyrene film	10
	Rubber hydrochloride film	10
	Vinylidene chloride-vinyl chloride copolymer film	10
	Nylon 11 [polyamide-11]	10
179.45(c)		
	Ethylene-vinyl acetate copolymer	30
179.45(d)		
	Vegetable parchment	60
	Polyethylene film	60
	Polyethylene terephthalate film	60
	Nylon 6 [polyamide-6]	60
	Vinyl chloride–vinyl acetate copolymer film	60

Note: Additional packaging materials for use during irradiation of prepackaged food are listed in the Threshold of Regulation Exemptions or Inventory of Effective Food Contact Substances.

Figure 6.15. FDA Radura Symbols for Irradiated Foods

both to inform the consumer and to prevent re-irradiation during subsequent use or processing. Many other irradiated products such as ingredients in multicomponent foods and foods for food service operations do not require that the final product be labeled as irradiated. There has been some effort to reduce these labeling requirements because of resistance by consumers to irradiated products. Current regulations require a radura emblem (Figure 6.15) and a statement regarding irradiation for whole foods that have been irradiated. Foods containing irradiated ingredients such as spices are not required to be labeled. Public perception of irradiated foods has been affected by both the historical association with military testing and irradiation research and by concerns with both radioactivity of the foods and secondary species of compounds and pathogens that might be created by the process. While the process has been shown to be safe when used carefully and sparingly, it is a developing technology that offers both the promise of improved safety and some risk of unintended consequences.

Food Safety, Quality, and Irradiation

Irradiation has continued to be a contentious issue, although many medical products and spices have been irradiated for years. Concern about the development of novel radiolytic species, or accelerating the rate of mutation of pathogens and insects as well as providing an uncontested growth environment for organisms such as aflatoxin-producing molds all have been raised as potential objections. Studies have also shown that although for the most part, spoilage organisms survive irradiation better than pathogens (causing food to spoil before becoming toxic), the survival of spore-forming organisms such as *C. Botulinum* type E under low radiation doses demands careful post-process handling [37]. While some of these issues remain a matter of debate, concerns expressed about using radiation in place of good sanitation practices are worth considering when irradiation is suggested as a final safety treatment for products, because the temptation to rely on the single, final treatment may create poor practice in other parts of the operation.

Food quality may be affected by extreme doses of radiation, to the point where early experiments on meat products at doses in excess of 70 kGy would reduce them to badly oxidized and

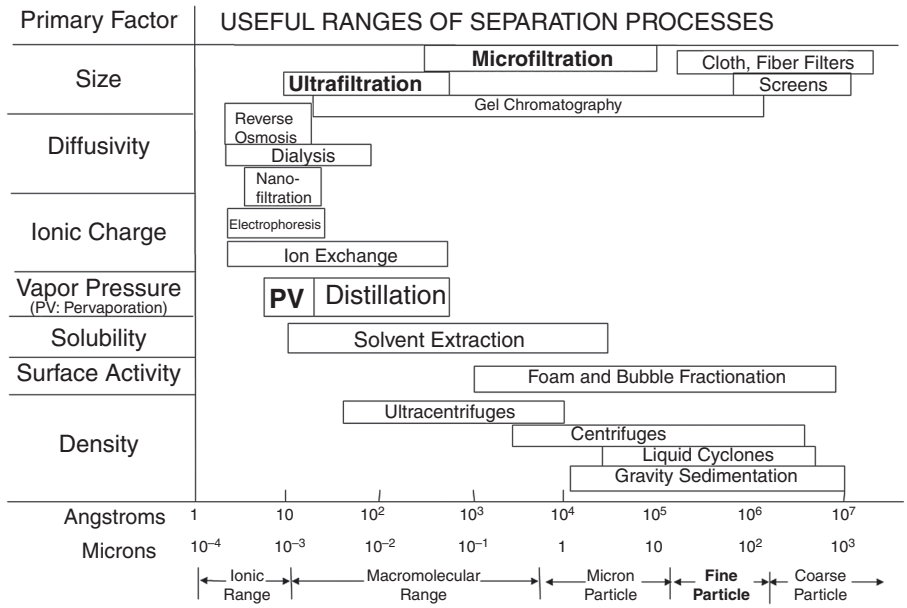

Figure 6.16. Range of Separation Processes
Source: M. Cheryan, (1989) "Ultrafiltration Handbook". Used with permission of the author.

inedible waste. At lower, approved doses, effects on food quality are similar to those produced by thermal processing, with water-soluble vitamins being oxidized most readily. Fats and oils may begin oxidizing more rapidly because of the direct energy input initiating oxidation reactions, and so control of oxygen availability may be an important step in preserving product quality, and oily foods such as fresh avocados that have been reported as generating off flavors with as little as 0.15 kGy may have to be minimally processed or given an alternative treatment.

Concentration and Separation of Food Products

Many processing systems rely on changing the concentration of particular components in a material. For food materials, there is a plethora of separation methods based on differentiating characteristics that are well described by Figure 6.16.

Most separation and concentration technologies may look intimidating in an industrial application because of the scale and secondary complexity. Despite this, nearly all of them are scaled-up versions of very simple ideas, typically using a particular (and preferably unique) differential in some physical property to separate materials.

Separation and Concentration by Size Differential

Size separation depends on both a useful size distinction and a method for passing (or not passing) materials of a particular size through a process. The oldest method of this type is simple sifting through a screen or grid that will separate materials or objects by size, allowing smaller units to pass and retaining larger ones. This has long been standardized using the gap in a wire mesh to allow both single and progressive sifting of materials under mechanical agitation. The

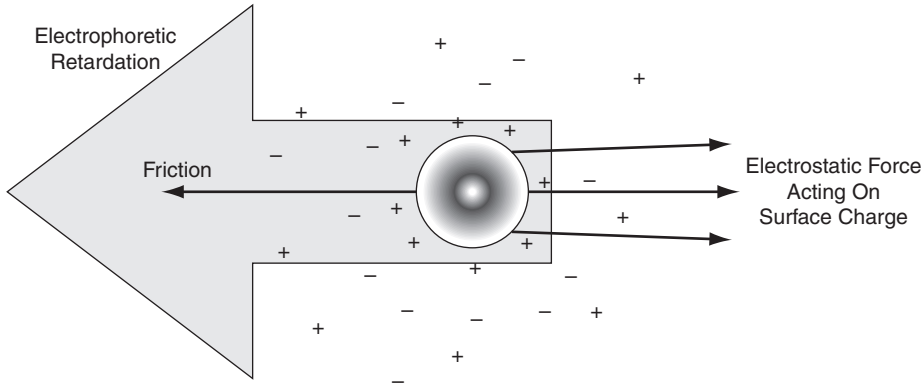

Figure 6.17. Eletrophoresis Forces on a Particle

agitation serves to reorient materials that may be lying across the screen in an effort to pass any material that is smaller in any of its dimensions than the opening in the mesh. As particulate components get smaller, the separation method becomes more reliant on a statistical average of pore sizes in a filter structure because these are often produced of fibrous mats or perforated sheets of solid material, and the exclusion size may be a function of both pore size and the orientation of the material in the filtrate. A good example of this would be a long, thin structure such as a protein strand that will pass end-on through an opening but is retained when it lies across the pore structure. Because of this, many filtration methods are very dependent on the particulars of the media, particulates, and the filter membrane itself, and will require evaluation for suitability before large-scale use is undertaken. Additionally, at the micro and ultra-filtration levels, it is possible to have surface chemistry interactions between the filtrate and the filter structure, which can affect the accuracy of the filtration. Nanofiltration exploits this in the separation of dairy by-products by allowing non-valent ions, water, and low-molecular-weight solids through and rejecting higher-molecular-weight solids and divalent and multivalent ions [38, 39].

Electrophoresis (Figure 6.17) takes this to the molecular level by driving the constituents through a fluid material via a uniformly applied electrical field. In this case, the materials are driven by electrostatic force and finally separated by the friction they exert, as well as their electrophoretic retardation force (a countervailing force that occurs due to a diffuse layer of opposite-charge ions about the material particle), as they move through the viscous fluids.

Separation and Concentration by Density Differential

Simple separation by density differential has also been done since prehistory with the separation of fatty components in dairy products and oil-water mixtures during the secondary olive pressings that use hot water to liberate oil from the ground olive paste. Simple settling of particulate matter in fluids is usually accomplished by giving the material a quiescent place (a settling tank or storage bin) to settle, but demands for higher-speed processing of materials that may have smaller density differences (and therefore longer settling times) can demand that the settling and separation rate be artificially enhanced either by agitation to accelerate the rate of particulate stratification, or by cyclone or centrifuge separation that provides artificial acceleration to the particles, in turn increasing the force of separation between the disparate masses. Additionally,

it is often useful to add material of a particular density to assist in separation, or to induce a flocculation process (described under surface activity separation).

Separation time in a simplified settling scenario that assumes a roughly spherical fluid particulate, as shown in Figure 6.18, in a two-part fluid system can be calculated using the Stokes equation, which is valid for small Reynolds number values (Re < 1.0), although attention must be paid to the direction of travel, as dictated by the density difference between the fluid and particle:

$$v_p = \frac{D^2 a(\rho_p - \rho_f)}{18\mu} \tag{6.6}$$

v_p: velocity of particle or droplet, m/s

D: diameter of spherical particle or droplet, m

a: applied acceleration, m/s^2

This is usually (g) gravitational force, 9.81 m/s^2

ρ_p: density of particle, kg/m^3

ρ_f: density of fluid, kg/m^3

μ: viscosity, $\frac{kg}{m \cdot s}$ or $Pa \cdot s$

Thus, for the fat and non-fat components in whole milk (which is an oil-in-water emulsion) under the existing force of gravity, the separation time can be calculated as

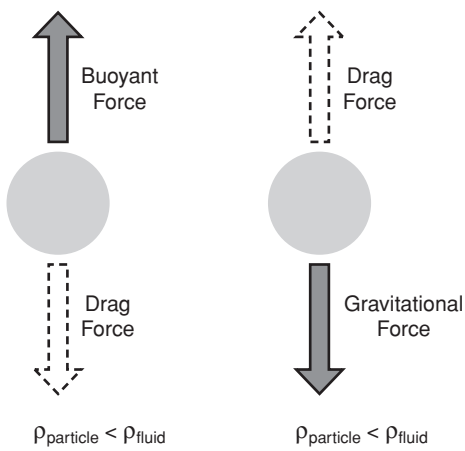

Figure 6.18. Stokes Equation Force Balances

From the extraordinary amount of time required for the fat components to separate in milk, one can see why early European estates had milk settling rooms where shallow pans of milk would be put out to reduce the distance that the fat component had to travel before being poured off.

A much simpler (and more sanitary) solution was eventually devised – a milk separator, which is effectively one of the first continuous-flow centrifuges, as shown in Figure 6.19. Centrifuges are mechanical devices that induce a high outward force by mechanical rotation of materials, one of which is usually a fluid. This creates an artificially high value for g, and proportionally reduces the separation time and overcomes the effects of Brownian motion on very small (< 0.1 μm) spherical particles:

$$a_c = r(2\pi N_r)^2$$

$$F_{\mathrm{rc}} = \frac{r(2\pi N_{\mathrm{r}})^2}{g} = \frac{a_{\mathrm{c}}}{g} \tag{6.7}$$

a_{c}: centrifugal acceleration, $\mathrm{m/s^2}$

F_{rc}: relative centrifugal force, expressed in "g's" or multiples of the force of gravity (g)

r: rotational radius, m

N_{r}: number of rotations, $\dfrac{\text{revolutions}}{\text{second}} = \dfrac{\text{RPM}}{60}$

g: gravitational acceleration, $9.81\ \dfrac{\mathrm{m}}{\mathrm{s^2}}$

Thus, from the Stokes equation

$$v_{\mathrm{p}} = \frac{D^2 a(\rho_{\mathrm{p}} - \rho_{\mathrm{f}})}{18\mu}$$

v_p: particle velocity, $\dfrac{\mathrm{m}}{\mathrm{s}}$

under the acceleration generated by a centrifuge

$$a = a_c = r(2\pi N_r)^2$$

$$v_p = \frac{D^2 a_c(\rho_p - \rho_f)}{18\mu}$$

$$= \frac{D^2(r(2\pi N_r)^2)(\rho_p - \rho_f)}{18\mu}$$

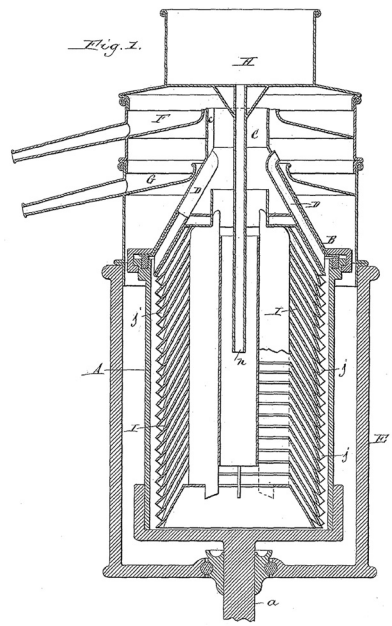

Figure 6.19. Cream Separator Patent Drawing
Source: US Patent 463794

Laboratory centrifuges are capable of creating millions of **g** in specially constructed static tubes that are mechanically rotated around a central driveshaft. Milk separators and continuous-flow extractors of this type operate similarly except that they depend on a series of closely spaced plates to reduce the "rising" distance, and to both rotate the components and to channel them either to the center or the radial edge of the rotor depending on density. Because the net *g* force is outward (roughly corresponding to "down"), the lighter fraction will be extracted up through the central axis ("*F*") while the heavier fraction is expelled along the edge ("*G*").

The time for a fat globule to travel to the "top" of the fluid is reduced significantly by increasing the effective force of gravity in a centrifuge. By closely spacing the plates, and reducing the distance, the "rising" time can be reduced to seconds rather than many hours, ensuring a useful throughput. Because this is a continuous flow device, the separation time is usually considered to be a *residence time* – the time that the fluid remains within the extractor – rather than a more intuitive *settling time*. Also, because of some small degree of mixing and other factors, the separation will not be complete in the sense that a careful centrifugation in a laboratory might produce, but will produce an acceptable product.

Homogenization

The Stokes equation also describes the necessary parameters for many kinds of homogenization, which is the converse of accelerated separation processes. Most often, a homogenizer will be designed to reduce globule or particle sizes below the point where they can aggregate or separate. For milk homogenization, the whole milk, which has fat globules that are both large (mean diameter ca. 2 μm) and buoyant enough ($\rho^{20^\circ C} = 915$ kg/m^3) to cluster and separate readily over a few hours, is forced under high pressure (ca. 15 MPa) through a series of valves that exert a great deal of fluid shear and force the mean particle size to approximately 0.5 μm and nearly eliminates clustering.

For other types of homogenization, such as that applied to peanut butter to prevent oil separation, an additional component – higher-melting-point saturated fat – is added to bind the oil droplets in place at room temperatures.

Cyclone and Hydrocyclone Separation

Cyclones are a type of extractor that rely on fluid flow to create rotation in the fluid column and to extract the heavier fraction or particulates from ports on the outer radius or a settling area at the bottom, while allowing the lighter component to escape through the central axis, as shown in Figure 6.20. Cyclones are limited by the kinetic energy of the fluid flow and can suffer from the inability to cleanly separate materials with very similar densities, but offer the advantages of simplicity and durability. Nearly every industrial process that creates a great deal of dust, shavings, or other waste will have an air cyclone in the air exhaust from its dust and particulate extraction systems, and consumer appliances such as vacuum cleaners have been designed to operate on the same principle.

Cyclones for liquids, called *hydrocyclones*, are used to separate liquids or liquid/solid combinations in the same manner, although the different density will require much stronger construction. Hydrocyclones can be manufactured to create radial forces thousands of times that of gravity, allowing separation of oil and water, agricultural processing components such as starch, removal of waste material in mining waterflow, and the removal of contaminants such as staples and sand in recycled paper pulping operations. Additionally, *air-sparging*

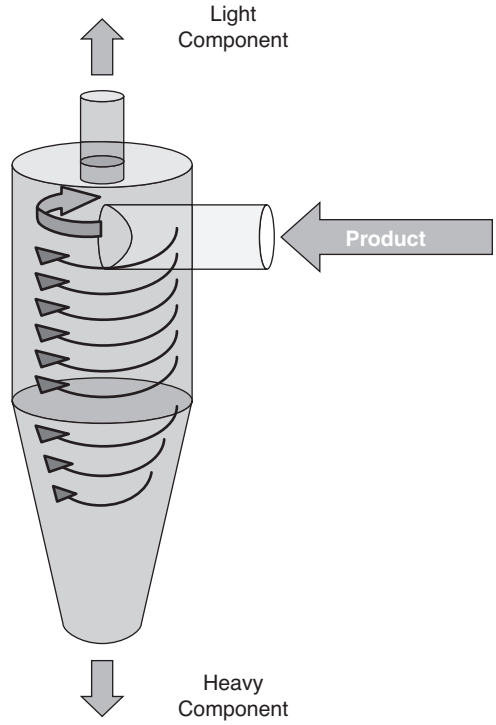

Figure 6.20. Cyclone Separator Diagram

hydrocyclones inject fine air bubbles into the fluid system, which attach to a particular component as a flocculent and aid in completeness of separation by creating a distinct density boundary layer between fluid and extracted material.

Separation and Concentration by Vapor Pressure

Separation of fluids from solids and oils by simply letting water evaporate is a traditional method that was augmented by adding heat to boil away water. This is a basic recovery method for sugar and salt, and has been enhanced by using multiple-effect evaporators that recover the heat of condensation in successive stages to improve energy efficiency in a series of staged evaporator pans.

When applied to water-alcohol mixtures, it was observed that a flammable vapor was produced at relatively low temperatures, which is possibly the source of the original Arabic term *al-ġawl* (roughly, "spirit"), although the intoxicating effects may also be the source of the name. This process was first isolated and extensively studied by the chemist Razis (Muhammad ibn Zakariya al-Razi) in the ninth century. Distillation is simply the heating of a mixture of components and then causing the condensation of the vapors of the lower boiling point fraction using chilled surfaces (often cold water circulating in pipes) to recover the low vapor pressure fraction. Although the process is simple, the engineering of optimized distillation and separation systems can be quite involved.

Distillation systems are typically constructed to allow multiple layers of vapor and increasingly concentrated distillate to interact via *plates* or *trays* in a manner similar to the

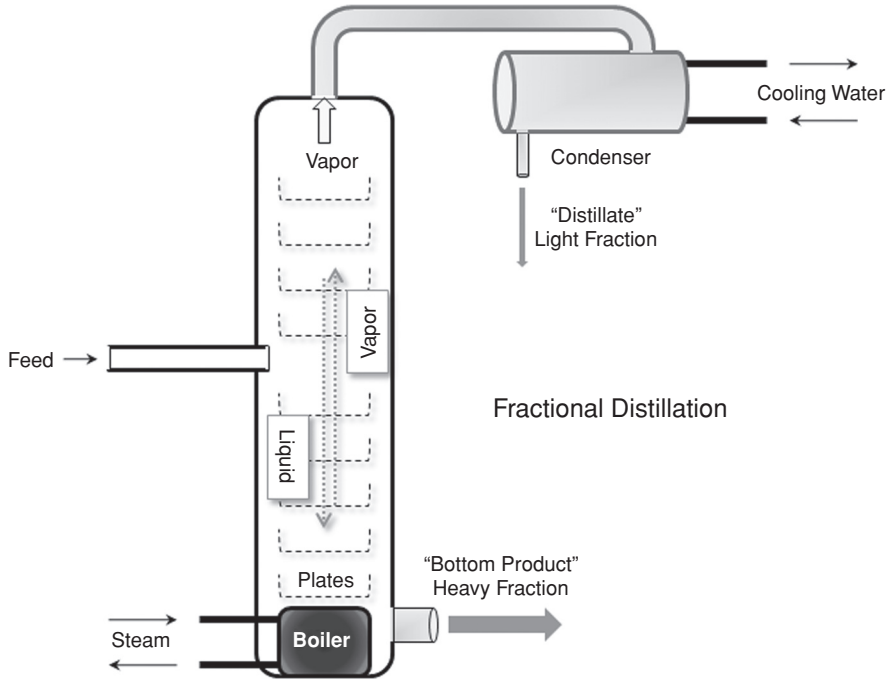

Figure 6.21. Fractional Distillation Diagram

multiple-effect evaporators, as shown in Figure 6.21, so that the efficiency of the separation process is maximized. As the heated vapors of the light fraction rise, they are forced upward through a downward-cascading flow of liquid heavy fraction, enabling simultaneous mass and heat transfer that enriches the vapor with more light fraction contained in the liquid and the heavy fraction with any residual heavy components in the vapor.

The result of a well-run distillation operation will be a purified *Top Product* and a *Bottom Product* made of the concentrated feedstock materials in the process. For desalination and water purification, the installation is quite simple, and newer installations have been devised that exploit the vapor pressure of water in a near-vacuum and use waste heat for domestic water treatment.

Figure 6.22 shows a McCabe-Thiele diagram of the operating curves of an ethanol-water distillation process. This provides a useful way of visualizing the exchange of top and bottom products as the vapors rise and liquids settle, although the number of *theoretical plates* must be translated into actual design data by correlating with actual plate or tray efficiencies [40]. From this diagram one can gather that there are limitations on the process that restrict the purity of the final products. The equilibrium curve in the example suggests that there is a so-called *pinch-point* where the extraction process is no longer practically feasible. This is the case because ethanol-water systems form lowered-boiling-point azeotropes at concentrations of 96% ethanol, and any further processing will actually start enriching the azeotropic mix with water.

Steam Distillation

Steam distillation, or steam stripping, relies on the principle that an immiscible mixture of two compounds will boil at a temperature that is often well below that of the high-boiling-point

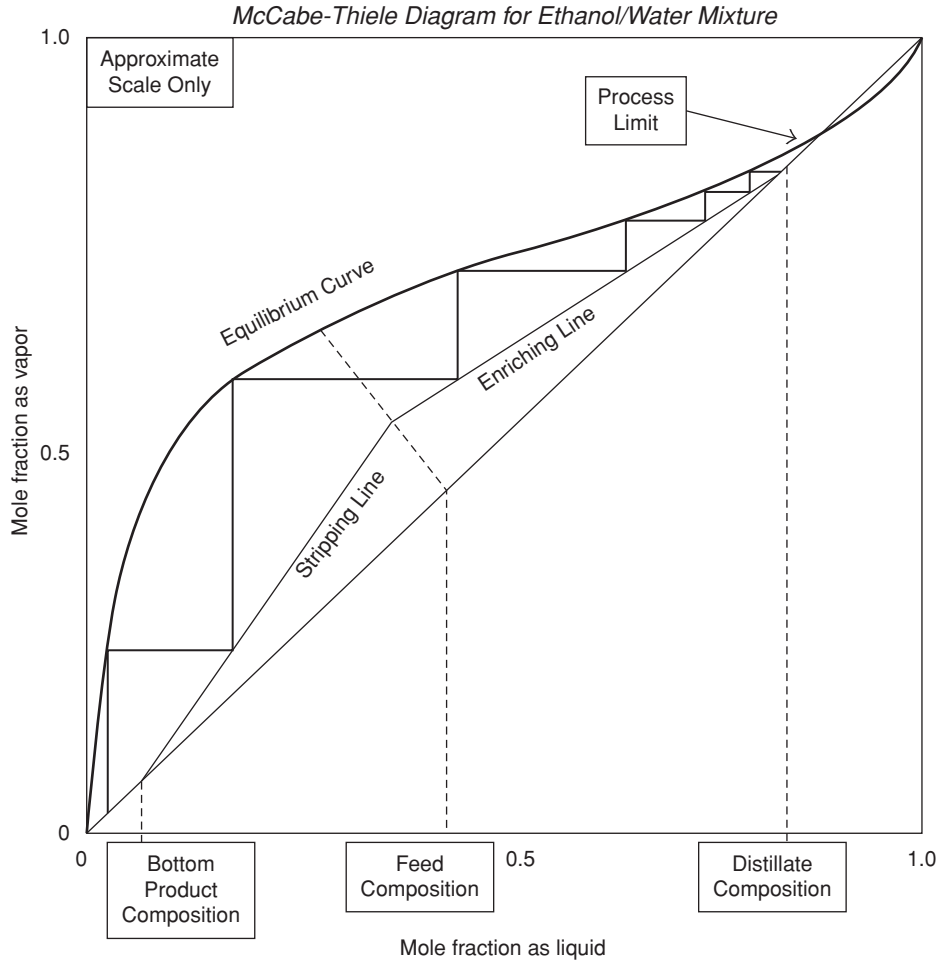

Figure 6.22. McCabe-Thiele Process Diagram

compound. This process is often used for the purification of essential oils that may be susceptible to decomposition from heat. It relies on the principle that immiscible compounds will each make a partial contribution to the total vapor pressure in the system as if the other compounds were not present, with the final effect being that the mixture will vaporize at a temperature that is lower than the highest-boiling-point fraction. Additional inducement to vaporize the desired compounds may be introduced by lowering the system operating pressure, amplifying the effect by creating a *vacuum distillation* system that can operate at reduced temperatures either alone or in conjunction with other distillation systems.

Separation by Crystallization

Extraction by crystallization is closely related to evaporative techniques in many parts of the world, very often those involved in sugar production and salt harvesting. Although these are most often simple evaporation processes that are allowed to run until the solvent is completely

removed, it is also possible to operate them on a continuous basis by forcing the solvent to become supersaturated and then harvesting the crystals as they appear. Another version of this is freeze concentration, which allows a highly concentrated solution to cross into supersaturation by reducing the temperature.

Freeze concentration stems from the observation that quiescent freezing will allow one fraction of a compound to preferentially crystallize while the other remains liquid. The most common example of this is in the inadvertent freezing of soft drinks in home refrigerators, which results in the syrup remaining liquid while the water component crystallizes.

This may be a preferred method of separation and concentration in operations that are severely restricted in temperature range because of the delicacy of the components, or in geographic regions where freezing product is a relatively simple process. The costs of installing and operating mechanical refrigeration systems makes the economics of this type of separation questionable where simpler or less expensive alternatives might be used.

Separation and Concentration by Solubility Differential

Solubility differential separation and concentration is another old technology that began with the simple removal of flavor components, minerals and sugar by water, and has been steadily refined and improved to use solvents to extract any manner of flavor and aroma compounds as well as specific constituents in the food manufacturing process. Water and ethanol are obvious solvent choices for many products, yet the use of more complex organic solvents is often more efficient. This in turn creates the problem of residual solvents in the final product, which have become less and less acceptable as the health risks associated with many formerly common solvents such as benzene used in decaffeination have come to light. Even if still-accepted solvents such as ethyl acetate are used, the product must often be heated or steamed for a long period to remove the residual solvents.

Phase-based solubility differential extraction may be used, utilizing steam as an extraction agent as previously described in order to remove particular components. An extension of this exploits very high pressures to create a solvent out of an otherwise innocuous material such as carbon dioxide.

Supercritical Fluid Extraction

Supercritical Fluid Extraction (SFE) exploits the differential solubility of organic compounds in liquefied gasses. These can be nearly any kind of gas, but is most often CO_2 that is above the *critical point*, as shown in Figure 6.23, where it can only exist as a liquid [41].

Because this requires very high pressures (in excess of 7.38 MPa/1070 psig) at temperatures above 31°C, the equipment for large-scale handling may be cumbersome and expensive, but the advantages of an organic-solvent-free product that is modified without heat can provide an enormous advantage for particular processes such as the decaffeination of coffee.

Because of CO_2 having a limited degree of polarity, it may be modified with the addition of ethanol or methanol in order to broaden the range of materials that can be extracted. Once the extraction process has been completed, the solvent is moved to a reduced-pressure system that exploits the pressure dependency of the solvents' dissolving power and precipitates the extracted materials. The gas can then be recycled or vented off depending on the type and size of the process operation.

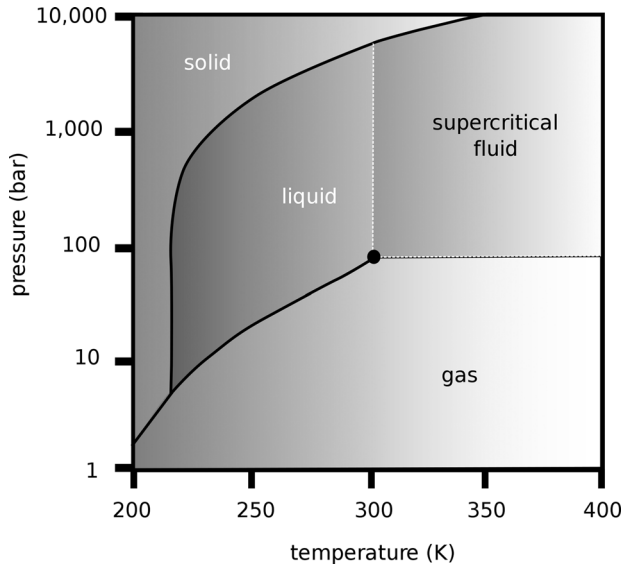

Figure 6.23. CO_2 Pressure – Temperature Chart

Separation and Concentration by Diffusivity

Diffusivity of a material through another material – an essential component of permeation that has been discussed in the context of packaging – can be exploited as a separation methodology. Osmosis, a naturally occurring type of diffusion, moves solvent from a region of low solute concentration to a region of high solute concentration across a *semi-permeable* membrane, as shown in Figure 6.24. These membranes are capable of passing solvent, but restrict solute movement and can range in size from cell walls in living organisms to large-scale desalination systems used for drinking and process water.

Osmotic pressure can be approximated with the Morse Equation:

$$\Pi = iMRT \tag{6.8}$$

Π: osmotic pressure
i: van't Hoff factor
M: molarity of solute, moles/liter
R: ideal gas constant
T: absolute temperature

Some examples of the van't Hoff factor are given in Table 6.7.

Because the object of most extraction processes is to remove pure solvent rather than add it – something that could usually be done more directly – reversing these processes becomes of more interest than their "forward" operation. Reverse osmosis depends on the ability of externally applied hydrostatic pressure to reverse the movement of solvent that produces osmotic pressure.

Because the osmotic pressure created can be quite large, the largest cost of desalination is usually due to the high pumping costs associated with pressurizing the osmotic membrane

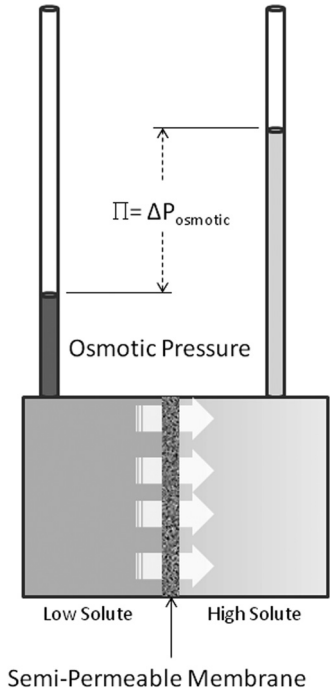

Figure 6.24. Osmotic Pressure Diagram

cartridges. Reverse osmosis can also be used to drive other solvents and moisture out of products that are being concentrated using similar methods, as shown in Table 6.8.

Pervaporation concentration is a similar process that is used for gas purification and extraction, and is based on reversing the Fickian diffusion exhibited through membranes in a manner similar to those used with fluids in reverse osmosis. The hydrostatic pressure applied to the feed gas overcomes the diffusion potential of the membrane and causes concentration of a purified gas on the *high concentration* side of the membrane, where it is extracted and either refrigerated or compressed.

Electrodialysis achieves an inverted version of osmosis in that it transports salt ions or other polar species through a membrane structure using an applied electric field in a manner similar to electrophoresis, and can be further utilized to concentrate or even create organic acids using selective membranes coupled with the appropriate electric fields.

Table 6.7. Table of van't Hoff Factors

Compound	Disassociates To	van't Hoff factor
Non-Electrolyte		1
Weak Electrolyte		1–2
NaCl	Na^+, Cl^-	2
$CaCl_2$	Ca^+, $2Cl^-$	3
$AlCl_3$	Al^+, $3Cl^-$	4
Na_2SO_4	$2Na^+$, $SO4^-$	3 (polyionic)

Table 6.8. Osmotic Pressures of Common Solutes

	Osmotic Pressure		Concentration
Solute	g/liter	moles/liter	PSI
NaCl*	35	6.0E-01	398.0
NaCl	1	1.7E-02	11.4
$MgSO_4$	1	8.3E-03	3.6
$MgCl_2$	1	1.1E-02	9.7
$CaCl_2$	1	9.0E-03	8.3
Sucrose	1	2.9E-03	1.1
Dextrose	1	5.6E-03	2.0

*Approximate Value for Seawater.

Separation and Concentration by Surface Activity

Foam and bubble separation uses an injected gas, usually compressed air, to create a froth of surface-active materials that adhere to the liquid-foam interface and are then carried out of the process along with entrained surface liquid [42]. This process can be helped by adding flocculating materials such as casein to help solids adhere to the rising bubbles and to be transported along with the foam. The foamate is then collapsed to give a resulting liquid that has a higher level of the surface-active material and any attached solids. Many types of industrial waste-water processing operations and protein concentration systems operate on this principle in order to concentrate solids and proteinaceous materials. An optimum velocity for gas-to-liquid mixes in foams can be constructed using Equation 6.9, though the reader is recommended to Stevenson (2007) [43] for a more thorough treatment of the derivation.

$$j_g^* = \frac{\rho g r_b^2}{\mu} mn \left(\frac{2}{n+1}\right)^2 \left(\frac{n-1}{n+1}\right)^{n-1} \tag{6.9}$$

j_g^*: maximum superficial velocity for a stable foam, m/s
ρ: interstitial liquid density, kg/m^3
g: acceleration of gravity, m/s^2
r_b: harmonic mean bubble radius, m
μ: interstitial liquid dynamic viscosity, $Pa \cdot s$
j_d: superficial drainage velocity, m/s
m, n: surfactant type and concentration factors such that
$m\varepsilon^n = \frac{\mu j_d}{\rho g r_b^2}$: Stokes number

Endnote

*Capillary condensation is a process where adsorption of a vapor-phase material into a porous medium continues until the pores become filled with condensed material, which may occur well below the saturation vapor pressure due to the large number of Van derWaals interactions in the minute spaces of the porous medium.

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